



# Removal of perfluoroalkyl substances (PFAS) from different water types by techniques based on anion exchange (AIX), powdered activated carbon (PAC), iron(III) chloride and nanofiltration (NF) membrane – A systematic comparison

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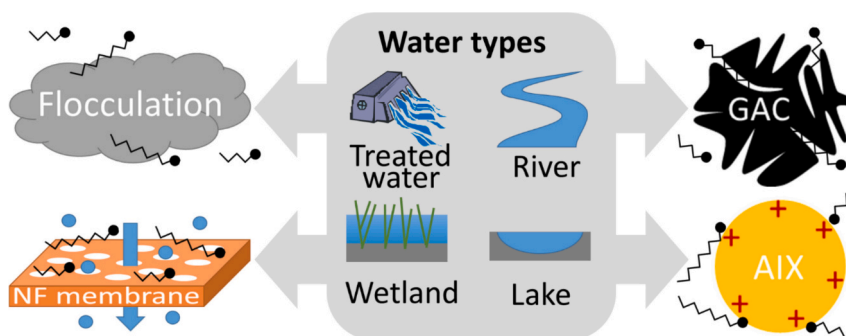
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## HIGHLIGHTS

- Systematic comparison of AIX, PAC, FeCl<sub>3</sub>, and NF membrane for different water types
- PFAS removal using NF membrane was low for PFAS <400 Da and higher for PFAS >400 Da.
- PFAS removal depended on CF-chain length and functional group.
- $\sum$ PFAS removal efficiency significantly increased with increasing PAC dose.
- Individual PFAS were significantly correlated with DOC and DOC-related parameters.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Presence of *per*- and polyfluoroalkyl substances (PFAS) in groundwater and surface water used for drinking water production is a major concern, due to possible adverse effects of PFAS on human health. Stricter guidelines on PFAS levels in drinking water currently being implemented on global scale typically require use of advanced techniques for water treatment. The aim of this study was to systematically compare four different treatment techniques for removal of PFAS and to evaluate the impact of water type on the removal efficiency. We hypothesized that the water type has a significant influence on the removal efficiency for the tested treatment techniques. The four different treatment techniques included i) anion exchange (AIX) MIEX®, ii) powdered activated carbon (PAC), iii) coagulation with ferric chloride (FeCl<sub>3</sub>), and iv) nanofiltration (NF) membrane. Mean  $\sum$ PFAS removal was found to be highest for NF membrane ( $48 \pm 7.6$  %), followed by AIX ( $30 \pm 7.7$  %), PAC ( $18 \pm 3.7$  %) and FeCl<sub>3</sub> ( $8.8 \pm 8.9$  %). For NF membrane, observed removal efficiency of PFAS was best described by a sigmoid curve centred around 400 Da, with low removal (25–35 %) of low-molecular-weight PFAS (<400 Da) and higher removal (47–75 %) of PFAS with greater molecular weight (>400 Da). For AIX and PAC, PFAS removal depended on perfluorocarbon chain length and functional group, e.g. mean  $\sum$ PFAS removal efficiency

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significantly increased ( $p < 0.05$ ) from 12 % using a PAC dose of 20 mg L<sup>-1</sup> to 46 % using a PAC dose of 100 mg L<sup>-1</sup>. Significant correlations were observed between removal of individual PFAS and dissolved organic carbon (DOC) removal and DOC characterisation parameters (specific ultra-violet absorbance (SUVA), humification index (HIX), freshness index (FI), absorbance at 254 nm (UV254)). This illustrates the importance of considering DOC characteristics and their seasonal variations when choosing PFAS removal technique and indicates potential of these parameters as predictors of PFAS removal efficiency.

## 1. Introduction

Per- and polyfluoroalkyl substances (PFAS) have attracted attention in recent decades due to their environmental persistence, bio-accumulation, toxicity potential and ubiquitous distribution in the environment (Ahrens and Bundschuh, 2014). Because of their unique physicochemical properties (they are both hydrophilic and hydrophobic), PFAS are used in various industrial and consumer products, such as water-repellent textiles, paints, waxes and aqueous film-forming foam (AFFF) (Buck et al., 2011). PFAS can be released into the environment via diffuse sources such as atmospheric deposition (Söregård et al., 2022) and water run-off (Skaar et al., 2019) or from point sources such as wastewater treatment plants (WWTPs) (Comber et al., 2021), landfills (Knutsen et al., 2019) and firefighter training facilities (Ahrens et al., 2015). PFAS are very persistent and, once released, can be translocated between different compartments in the environment (Chen et al., 2019).

Presence of PFAS in groundwater and surface waters used for drinking water production is a major concern (Gyllenhammar et al., 2019), due to possible adverse effects of PFAS on human health including alterations in the development and endocrine system, immunotoxicity, cancerogenicity, hepatotoxicity and reprotoxicity (Sunderland et al., 2019). To combat this, stricter drinking water guidelines are being implemented worldwide (Gobelius et al., 2018). For example, the new Drinking Water Directive introduced by the European Parliament in December 2020 sets a limit of 100 ng L<sup>-1</sup> for  $\sum_{20}$ PFAS and 500 ng L<sup>-1</sup> for total organic fluorine as a PFAS sum parameter (EU, 2020). Some countries within Europe, such as Sweden and Denmark, are planning to apply even stricter drinking water limits, such as 4 ng L<sup>-1</sup> and 2 ng L<sup>-1</sup>, respectively, for the sum of four PFAS ( $\sum_4$ PFAS) (perfluorooctanoate (PFOA), perfluorononanoate (PFNA), perfluorohexanoate (PFHxS), perfluorooctanoate (PFOS)) (EU, 2020). Thus, there is increasing regulatory and consumer pressure on water companies to reduce PFAS levels in drinking water. However, conventional drinking water treatment techniques such as flocculation, sedimentation, sand filtration, and chlorine disinfection, which are intended to remove particles, dissolved organic carbon (DOC), and harmful bacteria, are generally not efficient at removing PFAS (Rahman et al., 2014). However, some conventional treatment methods such as coagulation with ferric chloride (FeCl<sub>3</sub>) has shown high removal efficiencies for longer chain PFAS (Bao et al., 2014), but more research is needed with respect to different types of PFAS.

A wide variety of treatment techniques for removal of PFAS from drinking water have been investigated (Mastropietro et al., 2021). The most commonly tested treatment option is the use of granulated activated carbon (GAC), but the removal efficiency of GAC decreases over time due to saturation and is generally low for shorter-chain PFAS (McCleaf et al., 2017). Furthermore, powdered activated carbon (PAC) has been used for removal of PFAS showing a general higher removal of PFAS for PAC with a larger surface area and pore size (Lei et al., 2023). Anion exchange (AIX) resin generally achieves better removal efficiency for shorter-chain PFAS, but the removal efficiency decreases over time, and better methods for regenerating the resin are needed (Boyer et al., 2021). Additionally, DOC can influence the PFAS removal efficiency of sorbent filters using AIX or GAC during drinking water treatment (Kothawala et al., 2017), however, there is a lack of research of the influence of DOC on PFAS removal for different types of treatment techniques. Reverse osmosis (RO) and nanofiltration (NF) membranes

can achieve good PFAS removal efficiency but often require pretreatment due to the risk of membrane fouling and clogging (Mastropietro et al., 2021), and they are also more costly than GAC- and AIX-based methods. Many other treatment technologies have also been tested for PFAS removal, but mainly at laboratory scale (Ross et al., 2018). Moreover, there has been limited systematic comparison of existing treatment techniques for PFAS removal, and there is a lack of understanding of the impact of water quality on PFAS removal efficiency.

The overall aim of this study was to systematically compare four different treatment techniques for removal of PFAS and to evaluate the impact of water type on the removal efficiency. The treatment techniques were: AIX, PAC, coagulation with FeCl<sub>3</sub> and hollow-fibre NF membrane, all of which are commonly used in water treatment. Each treatment was applied to samples of six different types of water containing DOC with varying characteristics, spiked with 14 PFAS of different perfluorocarbon chain length and three different functional groups. Consideration of different DOC characteristics is important, as drinking water treatment plants (DWTPs) that use surface water typically encounter seasonal and event-driven changes in the characteristics of DOC of their raw water. However, very little is known about how DOC characteristics affect PFAS removal. Therefore, correlations between PFAS removal efficiency for the six different water types and their DOC content and DOC characterisation parameters were evaluated.

## 2. Material and methods

### 2.1. Chemicals and material

The 14 PFAS used to spike water samples were: C<sub>3</sub>-C<sub>11</sub>, C<sub>13</sub> perfluoroalkyl carboxylates (PFCAs; i.e., PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUnDA, PFDoDA, PFTeDA), C<sub>4</sub>, C<sub>6</sub>, C<sub>8</sub> perfluoroalkyl sulfonates (PFSAs; i.e., PFBS, PFHxS, PFOS), and perfluorooctanesulfonamide (FOSA) (purchased from Sigma-Aldrich). In addition, 10 mass-labelled internal standards (IS) (<sup>13</sup>C<sub>4</sub> PFBA, <sup>13</sup>C<sub>2</sub> PFHxA, <sup>13</sup>C<sub>4</sub> PFOA, <sup>13</sup>C<sub>5</sub> PFNA, <sup>13</sup>C<sub>2</sub> PFDA, <sup>13</sup>C<sub>2</sub> PFUnDA, <sup>13</sup>C<sub>2</sub> PFDoDA, <sup>18</sup>O<sub>2</sub> PFHxS, <sup>13</sup>C<sub>4</sub> PFOS, M<sub>8</sub>FOSA) and one injection standard (InjS) (<sup>13</sup>C<sub>8</sub> PFOA) (purchased from Wellington Laboratories, Ontario, Canada) were included in the analysis. For further details, see Table S1 in Supporting Information (SI).

### 2.2. Water samples

The six different water types used for the experiments were taken from four different locations in Sweden (Tables 1 and S2 in SI) to obtain a range of DOC characteristics and assess the effect on PFAS removal efficiency (Kothawala et al., 2017). The sampling locations were a wetland in the Krycklan catchment (sample C4), a large humic lake Bolmen (sample BO), a river in Tostarp (sample TO) and the Kungsängsverket wastewater treatment plant in Uppsala, Sweden (sample EB). Sample C4 was divided into two fractions, one of which was exposed to ultraviolet-C radiation (sample UV). The last water sample has been cultivated using algae in MilliQ water in the laboratory (sample SA).

The four original water samples (i.e. C4, UV, BO and TO) contained terrestrial DOC primarily originating from decomposed plant material, known as allochthonous origin (i.e. generally hydrophobic and high molecular weight) (Fabris et al., 2008). The two remaining water

samples (i.e. EB and SA) also contained organic matter produced by bacteria and algae respectively, known as autochthonous origin (i.e. generally hydrophilic and lower molecular weight) (Fabris et al., 2008). The water samples were characterised based on classical water parameters, and DOC was characterised based on specific ultraviolet absorbance (SUVA) (i.e. DOC quality as indicator of carbon aromaticity), humification index (HIX) (i.e. degree of humification) (Ohno, 2002), freshness index (FI) (i.e. DOC source from microbial (higher FI) or terrestrial (smaller FI) origin) and absorbance at 254 nm (UV254) (Fabris et al., 2008; Parlanti et al., 2000). All water samples were diluted to give a DOC range of 6.1 to 9.6 mg L<sup>-1</sup>, in order to approximate the observed DOC variation in typical raw water used for drinking water production and to exclude any strong effect of DOC concentration on removal of PFAS (Kothawala et al., 2017). The water samples used in the PFAS removal experiments were spiked with 100 µL of a mixture of the 14 PFAS (c = 480 µg mL<sup>-1</sup>) in 9.6 L of water (c = 5 µg L<sup>-1</sup> for individual PFAS) to enable quantification of PFAS before and after treatment. This concentration has been selected to make it analytical possible to quantify PFAS before and after treatment even if a substance was nearly removed completely. These concentrations were in the upper range of values observed previously at PFAS-contaminated sites in Sweden (Gobelius et al., 2018) and global-scale (Kurwadkar et al., 2022), but generally lower than the values used in most previous laboratory studies on PFAS removal treatments (Boyer et al., 2021).

## 2.3. Water treatment techniques

All experiments were performed under controlled conditions in the laboratory at a constant temperature of 20 °C. For all PFAS removal treatments except NF membrane, a standard chemical reactor (flocculator, KEMIRA) with 1 L glass bottles was used, to make it possible to adjust stirring rate and time (Fig. S1 in SI). For the NF membrane experiments, the membrane module was connected to an external plastic container, which was kept at 20 °C using a water bath (7 L).

### 2.3.1. Anion exchange (AIX)

For the AIX experiments, magnetic ion-exchange resin (MIEX®) (Gold resin, IXOM, 0.1–0.28 g mL<sup>-1</sup> density, 180–250 µm particle size) was added to 1000 mL of water sample. The amount of MIEX®, which was primarily developed for DOC removal from drinking water, was scaled depending on the DOC concentration of the different water types tested (Table S2 in SI). The doses used were: 5 mL L<sup>-1</sup> MIEX® for C4, UV, BO and SA; 3.5 mL L<sup>-1</sup> MIEX® for TO; and 4 mL L<sup>-1</sup> MIEX® for EB, according to the supplier's recommendations (personal communication). The experiments were performed with a stirring rate of 250 rpm for 15 min, and then the MIEX® resin was allowed to settle for 5 min before taking samples.

### 2.3.2. Coagulation with FeCl<sub>3</sub>

For the experiments using iron(III) chloride (FeCl<sub>3</sub>) (KEMIRA PIX-111; iron content 35–45 %), a dose of 41 µL L<sup>-1</sup> was added to 700 mL of water sample for C4, UV, BO and, SA (FeCl<sub>3</sub> dose of 8 mg L<sup>-1</sup>) and 28

µL L<sup>-1</sup> for TO and EB (FeCl<sub>3</sub> dose of 5.4 mg L<sup>-1</sup>), as typically used during drinking water treatment (Siélichi et al., 2008). To optimise the performance of FeCl<sub>3</sub>, the pH was kept within the known optimal range (4.9–5.2) using sodium hydroxide (NaOH) or hydrochloric acid (HCl). The samples were stirred for 30 s at a speed of 350 rpm, then stirred slowly for 20 min at 40 rpm, followed by a settling time of 60 min before taking samples.

### 2.3.3. Powdered activated carbon (PAC)

For the PAC experiments, PAC (Norit W90, total surface area 725 m<sup>2</sup> g<sup>-1</sup>; particle size 17 µm) was added to 700 mL of water sample in an amount scaled depending on the DOC concentration in the water type (Table S2 in SI). The doses used were: 20 mg L<sup>-1</sup> for C4, UV, BO and SA; 13.6 mg L<sup>-1</sup> for TO; and 13.5 mg L<sup>-1</sup> for EB. For sample C4, two additional PAC doses were tested (50 mg L<sup>-1</sup> and 100 mg L<sup>-1</sup>) to investigate the removal efficiency of PFAS at higher doses. The experiments were performed with a stirring rate of 250 rpm for 15 min, then the PAC was allowed to settle for 5 min before taking samples.

### 2.3.4. NF membrane

NF membrane separation was performed using a X-Flow T/RX-300 module fitted with a Pentair HFW1000 membrane (hydrophilic membrane, negatively charged surface, 120 fibres, diameter 0.8 mm; dimensions 200 mm × 1538 mm, total membrane area 40 m<sup>2</sup>; 0.5 m s<sup>-1</sup> cross flow; 1 bar transmembrane pressure). This hollow-fibre membrane has a nominal molecular weight cut-off (MWCO) of 1000 Da (Pentair, 2023). Initially, the sample was circulated across the membrane for 45 min, with both the reject and permeate recirculated back to the feed water container to equilibrate the system. Then the first sample was collected from the permeate valve, followed by two more samples after 25 % (~1.5 h) and 50 % (~3 h) of the feed volume had been filtered. The flow rate was monitored and ranged between 0.76 and 1.0 L h<sup>-1</sup> (10–14 L m<sup>-2</sup> h<sup>-1</sup>), which is a typical flux for this kind of membrane.

## 2.4. PFAS analysis

Prior to extraction, the water samples were filtered using a glass microfibre filter (GF/C, Whatman, 1.2 µm). Extraction was performed as solid-phase extraction (SPE) using Oasis Wax cartridges (Waters, 6 cc, 150 mg, 30 µm) as previously described (Ahrens et al., 2009). In brief, each water sample was spiked with 100 µL of PFAS IS mixture (20 pg µL<sup>-1</sup> per compound) prior to SPE. The SPE cartridges were preconditioned with 4 mL 0.1 % ammonium hydroxide in methanol, followed by 4 mL of methanol and 4 mL Millipore water. The cartridges were then loaded with ~80 mL water sample at a rate of one drop per second and washed with 4 mL 25 mM ammonium acetate buffer in Millipore water. Elution was carried out with 4 mL methanol and 4 mL 0.1 % ammonium hydroxide into 15 mL PP-tubes. The samples were concentrated to 1 mL using nitrogen evaporation (N-EVAPTM112, Organomation Associates, USA). Finally, 10 µL InjS (concentration 200 pg µL<sup>-1</sup>) were added to each sample and they were analysed for PFAS using liquid chromatography-mass spectrometry (HPLC-MS/MS; Agilent

**Table 1**

Characteristics of the six different waters used for this study including type, pH, conductivity, dissolved organic carbon (DOC) concentration, SUVA, HIX, Freshness Index (FI), and UV254 (for details see Table S2 in SI).<sup>a</sup>

Water	Type	pH	Conductivity (S m <sup>-1</sup> )	DOC (mg L <sup>-1</sup> )	SUVA	HIX	FI	UV254
C4	Wetland	7.6	18	9.0	4.7	0.94	0.36	0.42
UV	Wetland, UVC radiation of C4	7.5	14	7.0	4.6	0.96	0.27	0.32
BO	Coniferous	7.4	26	9.1	4.0	0.93	0.51	0.36
TO	Deciduous	6.7	26	6.1	3.9	0.95	0.45	0.24
EB	WWTP after biofilter	7.3	300	7.0	2.0	0.87	0.90	0.14
SA	algae	8.1	34	9.6	1.3	0.93	0.45	0.12

<sup>a</sup> WWTP = wastewater treatment plant.

Technologies LC 1200 series coupled to 6460 Triple Quad system).

### 2.5. Quality control

As part of the quality control process, blanks, method detection limits (MDLs), recovery, duplicate samples, and positive controls were evaluated (Tables S3 and S4 in SI). Blank concentrations ( $n = 9$ ) were in the low nanograms per litre range, except for some PFAS during the NF membrane experiments due to insufficient cleaning between the experiments (Table S3 in SI). The MDLs were calculated based on mean blank concentration plus three standard deviations. If no PFAS was detected in the blanks, the lowest calibration point was used for MDL. Mean recovery for individual IS in the samples ranged between 68 % and 98 % ( $n = 93$ ; Table S4 in SI). Mean standard deviation of duplicate samples ranged between 4.8 % and 20 % ( $n = 8$ ). Positive untreated controls (i.e. with no sorbent (AIX/FeCl<sub>3</sub>/PAC) or NF membrane used) ( $n = 3$ ) were included in the treatments involving flocculation and NF membrane on sample C4. Loss of individual PFAS was generally below 7 % for the flocculation treatments and below 30 % (except for PFDODA (42 %) and PFTeDA (45 %)) for the NF membrane treatment, and thus no correction of the results based on the positive controls was necessary. The loss of PFAS in the positives controls were generally comparable or lower in comparison to previous studies (Campos-Pereira et al., 2020).

The residual PFAS concentrations obtained for the different treatment techniques were corrected by the average loss in positive control samples to compensate for the sorption of PFAS to the walls of beakers, tubing or the flocculator. Statistical analysis was performed using Pearson correlation, student's *t*-test and principal component analysis (PCA) ( $\alpha = 0.05$ ) to check for correlations between the removal of PFAS and DOC content or characterisation parameters (i.e. SUVA, HIX, FI, UV254).

## 3. Results and discussion

### 3.1. Removal of PFAS using AIX

Mean  $\sum$ PFAS removal efficiency using AIX showed wide variation (range 17–39 %) for all six water types (Fig. 1A). In general, mean removal efficiency increased with PFCA perfluorocarbon chain length, from 6.1 % for C<sub>3</sub> PFBA to 39 % for C<sub>8</sub> PFNA, and then levelled off to 36 % for C<sub>11</sub> PFDODA and decreased to 18 % for C<sub>13</sub> PFTeDA. Mean removal efficiency also increased with PFSA perfluorocarbon chain length, from 41 % for C<sub>4</sub> PFBS to 68 % for C<sub>8</sub> PFOS. In addition, removal efficiency depended on the functional group, with the highest removal efficiency for C<sub>8</sub> PFOS (68 %), followed by C<sub>8</sub> FOSA (46 %) and C<sub>8</sub> PFNA (39 %). Similarly, C<sub>4</sub> PFBS showed higher removal efficiency (41 %) than C<sub>4</sub> PFPeA (12 %) and C<sub>6</sub> PFHxS showed higher removal efficiency (63 %) than C<sub>6</sub> PFHpA (15 %).

Similar trends have been observed previously for AIX in batch tests (Boyer et al., 2021) and column tests on PFAS-contaminated groundwater with a bed volume (BV) of 62,920 (McCleaf et al., 2017), 48,000 BV (Franke et al., 2021) and up to 160,000 BV (Zaggia et al., 2016). However, AIX MIEX® has been used primarily for DOC removal and has not been tested extensively for removal of PFAS (Tamanna et al., 2023). Overall, PFAS removal efficiency by AIX was generally lower in this study than reported previously, which could be due to non-equilibrium conditions (Park et al., 2020), high DOC content (6.1–9.1 mg L<sup>-1</sup>) (Park et al., 2020) and competitive sorption behaviour of PFAS (Wang et al., 2019) in this study. A previous study found that the negative charge of PFAS was better correlated with the equilibrium constant for sorption to AIX than hydrophobic interactions, indicating that the electrostatic charge interaction mechanism plays an important role in PFAS removal rate (Park et al., 2020). Removal of short-chain PFAS is mainly due to electrostatic charge interaction, whereas long-chain PFAS are mainly removed by hydrophobic interactions (Du et al., 2014), which explains

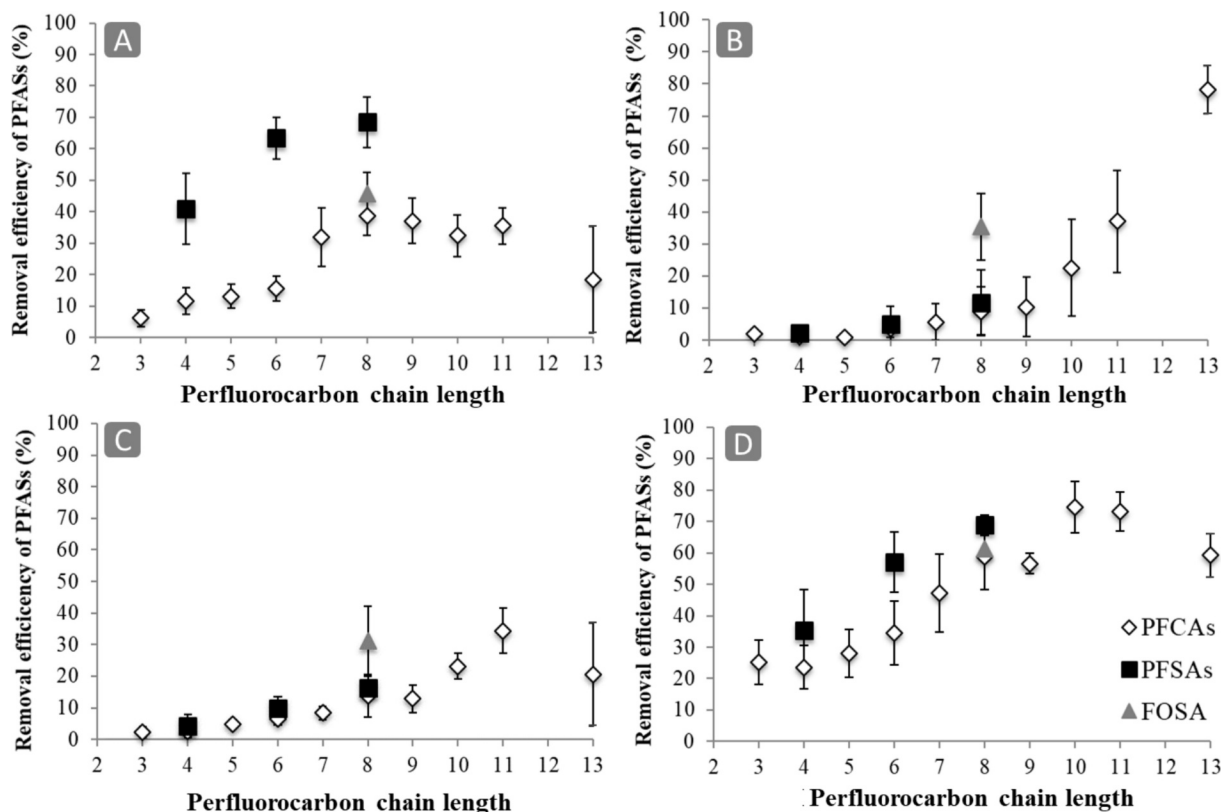


Fig. 1. Perfluoroalkyl substance (PFAS) removal efficiency (mean value for all six water types tested) by the different treatment techniques: A) AIX MIEX®, B) FeCl<sub>3</sub>, C) PAC (dose of 20 mg L<sup>-1</sup>), and D) NF membrane.



the better removal of short-chain PFAS reported for AIX compared with GAC (McCleaf et al., 2017). AIX is available as single-use resins (Park et al., 2020), but regeneration of resins is complicated by irreversible PFAS uptake, although use of salt together with organic solvents has been shown to achieve successful regeneration (Chularueangksorn et al., 2013). However, for drinking water treatment applications, the choice of regeneration chemical is limited to inorganic solutes or salts, which are known to be less efficient than mixes of salts and/or organic compounds and solvents (Liu and Sun, 2021).

### 3.2. Removal of PFAS using $\text{FeCl}_3$

Mean  $\sum$ PFAS removal efficiency for  $\sum$ PFAS using  $\text{FeCl}_3$  showed wide variation (0–25 %) for all water types (Fig. 1B). In general, mean removal efficiency increased with perfluorocarbon chain length, from ~1 % for  $\text{C}_3$ – $\text{C}_5$  PFCAs to 78 % for  $\text{C}_{13}$  PFTeDA and from 2.3 % for  $\text{C}_4$  PFBS to 12 % for  $\text{C}_8$  PFOS. The removal efficiency also depended on the functional group, with the highest removal efficiency for  $\text{C}_8$  FOSA (35 %), followed by  $\text{C}_8$  PFOS (12 %) and  $\text{C}_8$  PFNA (9.0 %). However, only small differences were observed for  $\text{C}_4$  PFPeA and  $\text{C}_4$  PFBS (1.2–2.3 %) and for  $\text{C}_6$  PFHpA and  $\text{C}_6$  PFHxS (3.5–4.9 %).

The generally low removal efficiency of  $\text{FeCl}_3$  is in agreement with previous reports of low removal for  $\text{FeCl}_3$  as coagulant (Bao et al., 2014). Removal efficiency generally increases with increasing dose of the coagulant (Xiao et al., 2013) and depends on the size of the flocs (Bao et al., 2014). Higher removal efficiencies for PFOS and PFOA can be achieved at lower pH, as lower pH results in positively charged flocs that induce electrostatic charge interactions with the ionisable PFAS (Bao et al., 2014). However, for drinking water purposes, that is not a valid option due to economic considerations and challenges with corrosion of concrete tanks at low pH.

### 3.3. Removal of PFAS using PAC

Mean  $\sum$ PFAS removal efficiency using PAC (dose of  $20 \text{ mg L}^{-1}$ ) ranged from 6.0 % to 15 % for all water types (Fig. 1C). In general, mean removal efficiency increased with perfluorocarbon chain length, from 2.2 % for  $\text{C}_3$  PFBA to 34 % for  $\text{C}_{11}$  PFDoDA, and levelled off to 21 % for  $\text{C}_{13}$  PFTeDA and from 4.2 % for  $\text{C}_4$  PFBS to 16 % for  $\text{C}_8$  PFOS. In addition, removal efficiency was influenced by the functional group, with the highest removal efficiency for  $\text{C}_8$  FOSA (31 %), followed by  $\text{C}_8$  PFOS (16 %).

%, and  $\text{C}_8$  PFNA (14 %). However, only small differences were observed for  $\text{C}_4$  PFPeA and  $\text{C}_4$  PFBS (2.5–4.2 %) and  $\text{C}_6$  PFHpA and  $\text{C}_6$  PFHxS (6.6–10 %). Previous studies have found slightly higher removal efficiency (typically >60 %) using similar PAC doses to the lowest tested in this study, i.e.  $25 \text{ mg L}^{-1}$  (Hansen et al., 2010), and  $30 \text{ mg L}^{-1}$  (Yu et al., 2014). The reason could be non-equilibrium conditions (Hansen et al., 2010; Yu et al., 2014), high DOC content ( $6.1$ – $9.1 \text{ mg L}^{-1}$ ) (Franke et al., 2021) and competitive sorption behaviour of PFAS (McCleaf et al., 2017) in this study.

Mean  $\sum$ PFAS removal efficiency increased significantly ( $p < 0.05$ ) with PAC dose, from 12 % with  $20 \text{ mg PAC L}^{-1}$  to 46 % with  $100 \text{ mg PAC L}^{-1}$  for water type C4 (Fig. 2). However, removal efficiency for shorter-chain  $\text{C}_3$ – $\text{C}_7$  PFCAs (12–41 % using a PAC dose of  $100 \text{ mg L}^{-1}$ ) and PFBS (45 %) was still <50 %, whereas removal efficiency for longer-chain PFAS was >70 % for  $\text{C}_8$ – $\text{C}_{13}$  PFCAs and > 65 % for  $\text{C}_6$  and  $\text{C}_8$  PFSAs (PAC dose  $100 \text{ mg L}^{-1}$ ). However, continuous dosing with  $\geq 50 \text{ mg PAC L}^{-1}$  is not a cost-efficient and practical solution for full-scale DWTPs, however, dosing of PAC can be used as a chemical barrier in response to incidence (e.g. AFFF-spill). A similar trend has been observed previously in studies using PAC doses of 30, 60 and  $100 \text{ mg L}^{-1}$  (Sun et al., 2016) and 30, 80 and  $100 \text{ mg L}^{-1}$  (Yu et al., 2014), indicating that PFAS removal depends on the PAC dose. Previous studies have also shown that PFAS removal by PAC depends on the particle size (Lei et al., 2023), which is correlated to the surface area and pore size, with greater number of binding sites available and thus generally higher removal by PAC compared to, for example, GAC (Hansen et al., 2010). Furthermore, PFAS sorption depends on the surface chemistry of AC characterised by hydrophobic, electron donor–acceptor, and electrostatic interactions, as well as negative charge-assisted hydrogen bond formation (Kim et al., 2024).

### 3.4. Removal of PFAS using NF membrane

Mean  $\sum$ PFAS removal efficiency using NF membrane ranged from 37 % to 59 % (on average,  $48 \pm 7.6$  %) for all water types (Fig. 1D). In general, mean removal efficiency increased with perfluorocarbon chain length, from 25 % for  $\text{C}_3$  PFBA and  $\text{C}_4$  PFPeA to 75 % for  $\text{C}_{10}$  PFUnDA and  $\text{C}_{11}$  PFDoDA, and levelled off to 59 % for  $\text{C}_{13}$  PFTeDA and from 35 % for  $\text{C}_4$  PFBS to 69 % for  $\text{C}_8$  PFOS. There were relatively small differences due to functional group, with the highest removal efficiency for  $\text{C}_8$  PFOS (69 %), followed by  $\text{C}_8$  FOSA (61 %) and  $\text{C}_8$  PFNA (59 %). Greater

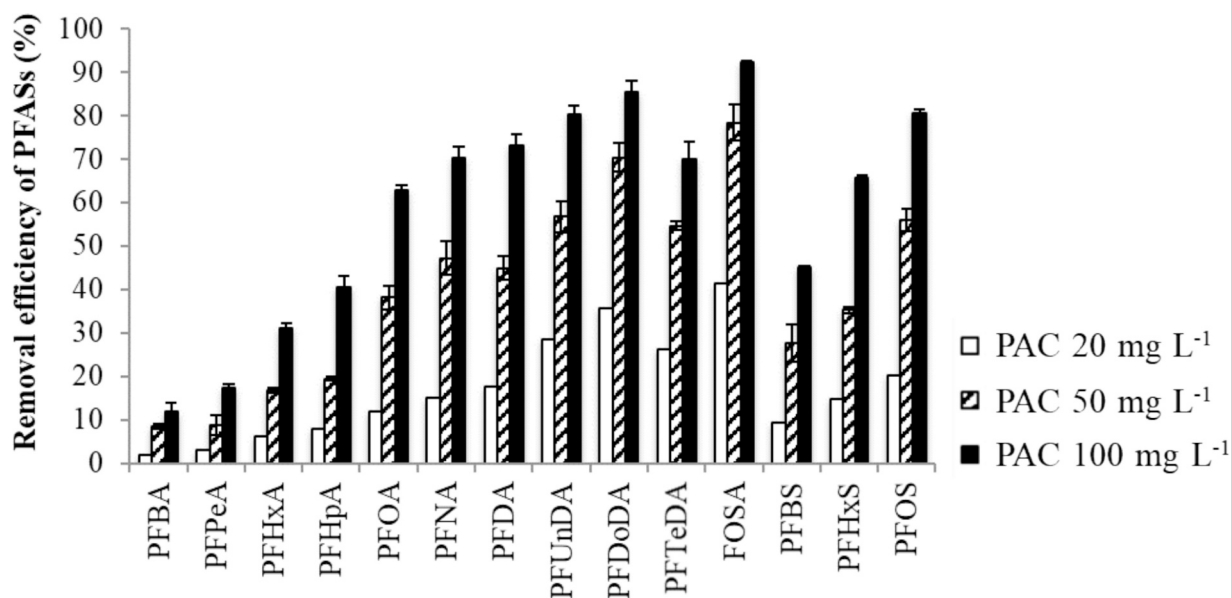


Fig. 2. Perfluoroalkyl substance (PFAS) removal efficiency from water type C4 (wetland in Krycklan catchment) using powdered activated carbon (PAC) at concentrations of  $20 \text{ mg L}^{-1}$ ,  $50 \text{ mg L}^{-1}$  and  $100 \text{ mg L}^{-1}$ .

differences in removal efficiency were observed for C<sub>4</sub> PFPeA and C<sub>4</sub> PFBS (24 vs 35 %) and C<sub>6</sub> PFHpA and C<sub>6</sub> PFHxS (28 vs 57 %).

The PFAS removal efficiency of the NF membrane was, as expected, mainly influenced by molecular weight (Fig. 3), which in turn is highly dependent on perfluorocarbon chain length. The observed PFAS removal efficiency was best described by a sigmoidal curve centred around 400 Da. Low-molecular-weight PFAS (<400 Da) had removal efficiencies ranging from ~25 % (PFBA, PFPeA) to 35 % (PFBS, PFHpA), whereas PFAS with higher molecular weight (>400 Da) had removal efficiencies ranging from 47 % (PFOA) to 75 % (PFUnDA). Thus, PFAS at lower molecular weight were removed by the NF membrane, for which the MWCO was 1000 Da. Very similar behaviour has been observed previously for humic substances, with removal below ~450 Da using a similar type of NF membrane (MWCO = 1000 Da) (Köhler et al., 2016). Removal of compounds below 400 Da could be due to sorption, while observed permeation above 400 Da could be due to the linear structure of PFAS allowing them to permeate more easily than larger branched humic compounds. The negative charge on the membrane itself would tend to exclude molecules with higher specific charge. The results of this study show that not only the MWCO is important to consider when selecting NF membranes for PFAS removal but also the surface charge of the membrane material (Mastropietro et al., 2021).

Overall, removal of PFAS by NF membrane was generally lower than reported previously, e.g. >93 % removal efficiency of nine target PFAS by NF membrane from deionised water and artificial groundwater (Appleman et al., 2013) or 99 % removal efficiency by NF membrane of PFAS from PFAS-contaminated groundwater used for drinking water production (Franke et al., 2019). However, the MWCO provided by the membrane supplier in those studies was only 270 Da (Franke et al., 2019; Appleman et al., 2013), which was much lower than that used in this study (MWCO = 1000 Da). Furthermore, the PFAS removal by NF membrane depended on molecular weight of PFAS present, DOC, salts (Lee et al., 2022) as well as electrostatic repulsion and charge on the membrane surface (Mastropietro et al., 2021). Ultimately, hollow-fibre membranes have a number of advantages over spiral wound membranes with regard to hardness removal, ease of cleaning, and energy consumption, and have the potential to efficiently remove PFAS to below the stricter drinking water guideline values recently implemented in Sweden and Denmark (4 ng L<sup>-1</sup> and 2 ng L<sup>-1</sup>, respectively, for  $\Sigma_4$ PFAS).

### 3.5. Comparison of the different treatment techniques in removal of PFAS and impact of DOC

The removal efficiency of individual PFAS by the different treatment techniques showed wide variation (Fig. 4). The highest mean removal

rate for  $\Sigma$ PFAS was observed for NF membrane (48 ± 7.6 %), followed by AIX (30 ± 7.7 %), PAC (18 ± 3.7 %) and FeCl<sub>3</sub> (8.8 ± 8.9 %). The four treatment techniques used here for removal of PFAS from different water types have not been compared systematically in previous studies, which typically focus only on one type of treatment method such as PFAS removal using different types of sorbents (Söregård et al., 2020) or membranes (Tang et al., 2007). Removal efficiencies for all 14 spiked PFAS could be calculated for all four treatment techniques to investigate trends regarding perfluorocarbon chain length and functional group. The lowest removal efficiency was generally observed for shorter-chain PFAS (i.e. C<sub>3</sub>–C<sub>6</sub> PFCAs, PFBS), for which NF membrane showed the best average performance (29 ± 5.5 %), followed by AIX (16 ± 12 %), PAC (5.4 ± 3.4) and FeCl<sub>3</sub> (~0 %, i.e. practically no removal of short-chain PFAS). The longer-chained PFAS (i.e. C<sub>7</sub>–C<sub>13</sub> PFCAs, C<sub>6</sub>, C<sub>8</sub> PFSA, FOSA) showed higher removal efficiency, with NF membrane again showing the best average performance (62 ± 8.85 %), followed by AIX (39 ± 16 %), FeCl<sub>3</sub> (24 ± 25 %) and PAC (23 ± 11 %). Overall, the removal efficiencies obtained for NF membrane, AIX and PAC were partly lower than described in the literature, which could be due to non-equilibrium conditions (Park et al., 2020; Kothawala et al., 2017), high DOC content (6.1–9.1 mg L<sup>-1</sup>) (Lei et al., 2023) and competitive sorption behaviour of PFAS (Wang et al., 2019) in this study.

Removal of DOC varied between the treatment techniques and water types. It was highest for NF membrane (76 ± 12 %), followed by FeCl<sub>3</sub> (60 ± 17 %), AIX (44 ± 30 %) and PAC (1.3 ± 1.3 %) (Figs. S2–S4 in SI, Fig. 5). There was a significant positive correlation ( $p < 0.05$ ) between DOC removal and removal of individual short-chain C<sub>3</sub>–C<sub>6</sub> PFCAs for AIX ( $p < 0.05$ ) and between DOC removal and removal of short-chain C<sub>3</sub>–C<sub>8</sub> PFCAs and C<sub>4</sub> and C<sub>6</sub> PFSA for NF membrane. It has been shown previously that DOC can have a positive impact on removal of PFAS using AIX (Kothawala et al., 2017; Franke et al., 2019; Franke et al., 2021) and NF membranes (Mastropietro et al., 2021), but the overall impact of DOC is typically negative since it drastically reduces the lifetime of AIX and other sorbents (Gagliano et al., 2020) and membranes (Verliefde et al., 2007). On the other hand, there was a significant negative correlation ( $p < 0.05$ ) between DOC removal and removal of C<sub>3</sub>–C<sub>4</sub>, C<sub>7</sub>–C<sub>10</sub> PFCAs, C<sub>4</sub>, C<sub>6</sub>, C<sub>8</sub> PFSA and FOSA for FeCl<sub>3</sub>. Inconsistent findings on the impact of natural organic matter (NOM) on PFAS removal have been reported previously, e.g. decreasing PFOS and PFOA removal with increasing NOM (Bao et al., 2014) or increasing sorption of PFOS with increasing NOM, probably because of co-removal of PFOS with NOM during coagulation (Xiao et al., 2013). For PAC, DOC removal efficiency was too low (1.3 ± 1.3 %) to allow any significant trend to be discerned.

Significant correlations ( $p < 0.05$ ) were also observed between PFAS removal and DOC characterisation parameters (i.e. SUVA, HIX, FI, UV254) (Table S5 in SI). In general, removal of shorter-chain C<sub>3</sub>–C<sub>8</sub>

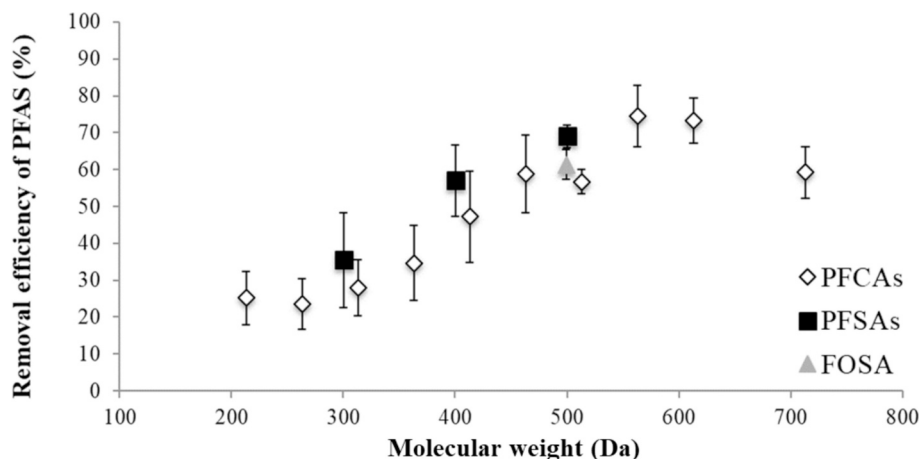
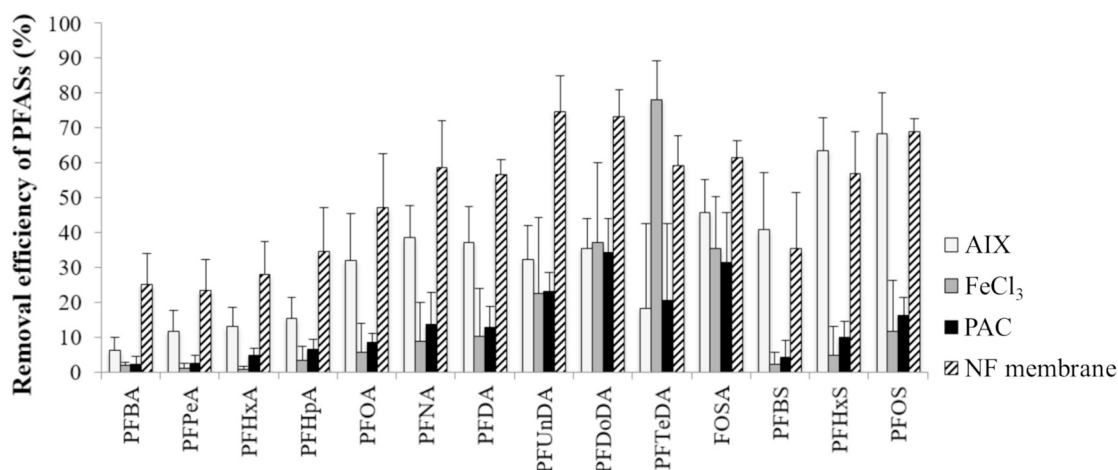
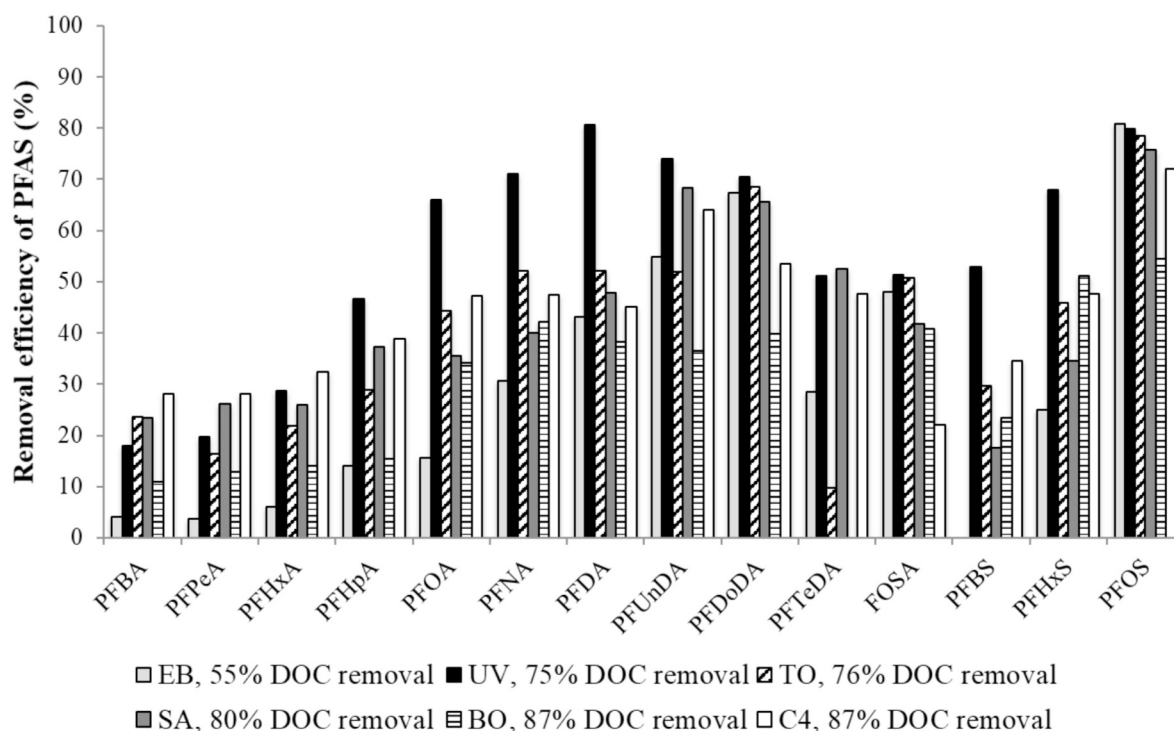


Fig. 3. Perfluoroalkyl substance (PFAS) removal efficiency by nanofiltration (NF) membrane as a function of molecular weight (Dalton, Da).



**Fig. 4.** Perfluoroalkyl substance (PFAS) removal efficiency, presented as an average for all six used water types, for AIX MIEX®, FeCl<sub>3</sub>, PAC (dose of 20 mg L<sup>-1</sup>) and NF membrane.



**Fig. 5.** Removal efficiency for PFAS and DOC for the different water types using NF membrane.

PFCAs, C<sub>4</sub> and C<sub>6</sub> PFSAs was positively correlated with SUVA, HIX and UV254 for AIX and NF membranes, whereas the trend for other PFAS was inconsistent. For FeCl<sub>3</sub>, several PFAS were negatively correlated with SUVA, HIX and UV254, which was similar to the correlation with DOC content. For PAC, there was no consistent correlation between the removal of individual PFAS and the different DOC characterisation parameters. The most consistent finding was a significant negative correlation between FI and removal of individual PFAS by AIX, FeCl<sub>3</sub> and NF membrane. A previous study suggested that FI can be a valuable indicator of water treatment efficiency (Köhler et al., 2016), but more research is needed on the use of DOC characterisation parameters to predict removal of PFAS in water treatment processes. Biplot analysis on the impact of water type on removal of individual PFAS by the different treatment techniques revealed that high removal of individual PFAS by

AIX and NF membrane was mainly associated with presence of terrestrial DOC (i.e. samples UV and C4) (Fig. 5 and Figs. S5–S8 in SI). In contrast, water containing organic matter produced by bacteria (i.e. sample EB) was negatively associated with removal of PFAS by AIX and NF membrane. For FeCl<sub>3</sub> treatment, water containing organic matter produced by algae (i.e. sample SA) was mainly associated with high removal of individual PFAS. For PAC treatment, the different water types had no clear impact on removal of individual PFAS. Previous studies have shown that the DOC varies seasonally (Groeneveld et al., 2023), which has not been investigated in this study. Thus, it is important to consider variations in DOC characteristics when choosing a PFAS removal technique.

#### 4. Conclusions

This study compared the removal efficiency of 14 individual PFAS using four different water treatment techniques commonly used in water treatment. The mean removal efficiency of  $\sum$ PFAS was in the following order: NF membrane ( $48 \pm 7.6\%$ ) > AIX ( $30 \pm 7.7\%$ ) > PAC ( $18 \pm 3.7\%$ ) >  $\text{FeCl}_3$  ( $8.8 \pm 8.9\%$ ). The removal efficiency was strongly related to the perfluoroalkyl chain length, with lowest removal efficiency for shorter-chain PFAS (i.e.  $\text{C}_3\text{-C}_6$  PFCAs, PFBS) and highest for longer-chained PFAS (i.e.  $\text{C}_7\text{-C}_{13}$  PFCAs,  $\text{C}_6$ ,  $\text{C}_8$  PFSA, FOSA). There was a significant positive correlation between DOC removal and removal of individual short-chain  $\text{C}_3\text{-C}_6$  PFCAs for AIX and short-chain  $\text{C}_3\text{-C}_8$  PFCAs and  $\text{C}_4$  and  $\text{C}_6$  PFSA for NF membrane ( $p < 0.05$ ). In contrast, there was a significant negative correlation between DOC removal and removal of  $\text{C}_3\text{-C}_4$ ,  $\text{C}_7\text{-C}_{10}$  PFCAs,  $\text{C}_4$ ,  $\text{C}_6$ ,  $\text{C}_8$  PFSA and FOSA for  $\text{FeCl}_3$  ( $p < 0.05$ ). For PAC, DOC removal efficiency was too low ( $1.3 \pm 1.3\%$ ) to allow any significant trend to be discerned. In general, removal of shorter-chain  $\text{C}_3\text{-C}_8$  PFCAs,  $\text{C}_4$  and  $\text{C}_6$  PFSA was positively correlated with SUVA, HIX and UV254 for AIX and NF membranes. For  $\text{FeCl}_3$ , several PFAS were negatively correlated with SUVA, HIX and UV254, which was similar to the correlation with DOC content. For PAC, there was no consistent correlation between removal of DOC parameters and individual PFAS. The most consistent finding was a significant negative correlation between FI and removal of individual PFAS by AIX,  $\text{FeCl}_3$  and NF membrane, which indicate that FI might be a valuable indicator for water treatment.

In pilot- or full-scale drinking water treatment using the different techniques, there are other important factors to consider apart from PFAS removal efficiency, such as cost-efficiency (Franke et al., 2021), life cycle assessment (Ellis et al., 2023), regeneration or disposal of sorbents (Gagliano et al., 2020), sorption selectivity (in particular for shorter-chain PFAS), and impacts of DOC (McCleaf et al., 2017). Additionally, handling of waste fractions such as reject water from the NF membrane (Franke et al., 2019; McCleaf et al., 2023) and upscaling to full-scale treatment (Belkouteb et al., 2020) are crucial considerations. It should also be noted that treatment train solutions can be more efficient for PFAS removal than single-treatment solutions (Lu et al., 2020; Franke et al., 2019), so further research on these is needed.

#### CRedit authorship contribution statement

**Lutz Ahrens:** Writing – original draft, Visualization, Validation, Project administration, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Sandra Lundgren:** Writing – review & editing, Methodology, Investigation, Formal analysis, Data curation. **Philip McCleaf:** Writing – review & editing, Methodology, Investigation, Conceptualization. **Stephan Köhler:** Writing – review & editing, Methodology, Investigation, Conceptualization.

#### Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Lutz Ahrens reports financial support was provided by Swedish Research Council Formas. If there are other authors, they 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.scitotenv.2025.179004>.

#### Data availability

Data will be made available on request.

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