



# Drinking water nanofiltration with concentrate foam fractionation—A novel approach for removal of per- and polyfluoroalkyl substances (PFAS)

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## ABSTRACT

Per- and polyfluoroalkyl substances (PFAS) are recognized as persistent pollutants that have been found in drinking water sources on a global scale. Semi-permeable membrane treatment processes such as reverse osmosis and nanofiltration (NF) have been shown effective at removing PFAS, however, disposal of PFAS laden concentrate is problematic. Without treatment of the concentrate, PFAS is released into the environment. The present work examined a novel PFAS removal scheme for drinking water using NF filtration with treatment of the resulting NF concentrate via foam fractionation (FF) with and without co-surfactants. The NF-pilot removed 98% of PFAS from AFFF contaminated groundwater producing permeate with 1.4 ng L<sup>-1</sup> total PFAS. Using FF resulted in  $\sum$ PFAS removal efficiency of 90% from the NF concentrate and with improved removal of 94% with addition of cationic co-surfactant. The resulting foamate composed approximately 2% of the NF feedwater volume and contained greater than 3000 ng L<sup>-1</sup> PFAS or 41 times greater than the NF feedwater. Addition of the cationic co-surfactant to the FF process resulted in increased removal efficiency of the shorter chain PFAS, specifically 37% for PFPeA, 9% for PFHxA, and 34% for PFBS thus attaining 59%, 99% and 96% removal efficiency, respectively. PFOA, PFPeS, PFHxS, PFOS each attained 99% FF removal with or without co-surfactant addition.

## 1. Introduction

Per and polyfluoroalkyl substances (PFAS) have been found in drinking water sources throughout the world (Ericson et al., 2009; Post et al., 2012; Appleman et al., 2014; Rahman et al., 2014; Gyllenhammar et al., 2015) and due to health-based effects, lower acceptable limits for PFAS in drinking water have been implemented or proposed in Europe and the U.S. (USEPA, 2016a; USEPA, 2016b; EU, 2020; Federal Register, 2022). For example, in Sweden (SLV, 2022), the proposed action limit is 4 ng L<sup>-1</sup> for the sum of four PFAS ( $\sum$ PFAS<sub>4</sub>), namely, PFNA, PFOA, PFHxS, and PFOS and a limit of 100 ng L<sup>-1</sup> for the sum of twenty-one PFAS ( $\sum$ PFAS<sub>21</sub>) (Table S1 lists these twenty-one). Even more challenging is Denmark's regulation of 2 ng L<sup>-1</sup> for  $\sum$ PFAS<sub>4</sub> (DMOE, 2021).

Fortunately, reverse osmosis (RO) and nanofiltration (NF) have been shown effective at reducing PFAS concentrations by 90–99% (Appleman et al., 2013; Wang et al., 2018; Franke et al., 2019; Soriano et al., 2020, 2021; Lee et al., 2022) thereby providing a method to lower PFAS concentrations to the required limits. Unfortunately, for PFAS contaminated feedwater RO and NF produce a PFAS laden concentrate with typical concentrate volumes being 10–20% of the feedwater with 5–10

times greater PFAS concentration which must be treated before discharge or managed so as not to contaminate the environment (Tow et al., 2021; Franke et al., 2021; USEPA, 2022; HVMFS, 2019).

The present work examines using NF treatment to meet lower PFAS limits in drinking water while utilizing foam fractionation (FF) for removal of PFAS from NF concentrate thereby allowing safe concentrate discharge into the environment. No reports of FF treatment for PFAS removal from NF concentrate have been published to the authors knowledge, however, FF has been shown to be effective at removing PFAS from landfill leachate, wastewater, and contaminated groundwater (Meng et al., 2018; Dai et al., 2019; Robey et al., 2020; McCleaf et al., 2021; Smith et al., 2022). Whereas NF can reliably remove PFAS from even relatively dilute PFAS contaminated source waters to low levels in the permeate, FF is not typically efficient unless applied on waters with higher concentrations of PFAS and thus may be more effectively used for treatment of NF concentrate than source water. Another advantage of using FF on the NF concentrate as opposed to directly on the source water is that smaller volumes would need to be treated, thereby reducing the size and complexity of the FF process installation. One of the present work's key research questions is to

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determine if the PFAS level in NF concentrate is high enough to make FF a good removal process for treating NF concentrate.

FF utilizes the surface-active molecules found in the liquid which attach to the gas-liquid interface of the bubbles and form a foam on the liquid surface (Burghoff, 2012a, Burghoff, 2012b). The process can be run in batch or continuous mode and foam once removed from the retentate and collapsed is referred to as foamate while the treated liquid as retentate. Process parameters such as pH, gas flow rate, temperature, bubble size, and additives have been investigated (Merz et al., 2011; Chai et al., 1998; Fanaie et al., 2020). FF has the added benefit that due to the resulting low volume, highly concentrated PFAS foamate, destructive techniques can be more efficiently applied such as advanced chemical oxidation, plasma-based treatment, chemical reduction, electrochemical oxidation, supercritical water oxidation, sonochemical methods or incineration (Lu et al., 2020; Tow et al., 2021; Banks et al., 2020; Li et al., 2019; Mahinroosta et al., 2020). Laboratory and bench-scale application of FF for PFAS removal has been reported with average removal efficiencies ranging from 20% to 100% for spiked lab-scale studies and for contaminated landfill leachate and groundwater (Robey et al., 2020; McCleaf et al., 2021; Smith et al., 2022; Buckley et al., 2021; Burns et al., 2021, 2022). A common finding is removal efficiency of over 90% for individual long-chain PFAS but with lower removal efficiency for short-chain PFAS. In lab studies the addition of co-surfactants has been shown enhance PFAS removal from wastewater, landfill leachate and contaminated groundwater (Meng et al., 2018; Taseidifar, 2020; Buckley et al., 2022). Yueh-Feng et al. (2021) reported enhanced PFOA removal efficiency from wastewater using cationic surfactants and postulated that cationic surfactants with a linear alkyl chain with the same number of carbons in the chain are the best flotation aids for PFOA. The addition of a co-surfactant has been reported to decrease the critical micelle concentration (CMC) and create a more active surface especially for surfactants with the same carbon chain length (Tian et al., 2017; Regev et al., 1996). Vo et al. (2023) found in lab-scale that cationic and zwitterionic co-surfactants improved removal of short-chain PFAS from landfill leachate due to charge interaction with oppositely charged PFAS functional groups.

The present study is unique in that removal of PFAS from contaminated municipal groundwater was investigated for the novel combination of NF for drinking water treatment coupled with FF for treatment of the resulting NF concentrate. The removal efficiency of a NF pilot using full-scale membranes was monitored for almost 6 months using feedwater from PFAS contaminated wellfields. The concentrate collected from this NF pilot was used to investigate the efficiency of FF for treating NF concentrate in two trials. Trial 1 consisted of batch lab-scale testing of FF with and without the addition of a co-surfactant aid. Four co-surfactants (cationic, two anionic, non-ionic) were tested with three different dosages. Trial 2 consisted of determining PFAS removal efficiency of continuous pilot-scale FF with and without addition of the co-surfactant determined as most effective in Trial 1. Three different surfactant dosages were tested each in triplicate. The efficiency and selectivity of FF for removal of the eight PFAS compounds found in the concentrate were determined along with improved efficiency afforded by the co-surfactant. The work addressed the specific conditions encountered for designing a future full-scale NF facility at Uppsala Water and Waste Ltd.'s Bäcklösa drinking water treatment plant to meet the drinking water goal of  $\sum \text{PFAS}_4 < 4 \text{ ng L}^{-1}$  (SLV, 2022) while preventing the discharge of PFAS laden concentrate to the Lake Malaren, Sweden's largest source of raw drinking water.

## 2. Materials and methods

The treatment setup investigated in the work shown is shown in Fig. S1 with water quality sampling points. Eight PFAS of the thirty-four PFAS analyzed (Table S1) were found above limit of detection (LOD) concentrations in the raw water fed to the NF pilot, specifically PFPeA, PFHxA, PFHpA, PFOA, PFBS, PFPeS, PFHxS, and PFOS.

### 2.1. Nanofiltration (NF) pilot

Raw water from the municipal wellfield was diverted to the NF membrane pilot after the aeration stage at the Bäcklösa drinking water treatment plant in Uppsala, Sweden. The NF pilot, as shown in Fig. S1, consisted of a constant pumped flow of approximately  $10 \text{ m}^3 \text{ h}^{-1}$  taken from after the Bäcklösa drinking water treatment plant's aeration stage to a biofilter for pretreatment (for general water chemistry data see Table S2). The biofilter consisted of a 1.5 m diameter, 3 m high down-flow filter containing a 1.5 m bed of Norit GAC1240W and had been in operation for approximately 6 months at the time of this work. From the biofilter, water was stored in a stainless steel feed tank ( $10 \text{ m}^3$ ), before feedwater pumping into the 2-stage NF pilot. The two-stage membrane process utilized six membranes in stage one and three membranes in stage 2 (NF90-400; Dow Filmtech™ Membranes). A constant feed water flowrate  $8 \text{ m}^3 \text{ h}^{-1}$  was pumped through a  $5 \mu\text{m}$  pre-filter consisting of seven elements (GE Infrastructure Water and Process Technology Pur-trex 5–30 filter) to remove any solids in the raw water before entering the NF unit. Concentrate internal recirculation to the feedwater inlet was provided at  $6 \text{ m}^3 \text{ h}^{-1}$ . The biofilter pretreatment was intended to prevent manganese fouling of the NF membranes which had been a problem in previous work (Franke et al., 2019). The NF process goal was PFAS removal and reduction of uranium-238, DOC, and hardness from the raw water while providing concentrate water for the FF batch and continuous tests.

The NF pilot results reported are for 175 days of operation with 80% recovery and transmembrane pressure which increased from 4.2 to 5.5 bar with no cleaning of the membranes at the time of this work. The 20% concentrate waste stream was directed to two 600 L polyethylene (PE) holding tanks with continuous overflow from which a smaller volume of concentrate was taken for the Trial 1 and 2 FF tests. Antiscalant (Ameroyal 363 Ashland Corp. USA) was added to the feedwater to provide a concentration of  $3.0 \text{ g m}^{-3}$  in the feedwater before entering the NF. Samples were taken for the NF process raw water, after biofilter, NF feedwater, NF permeate, NF concentrate. Flowrates for feedwater, concentrate, recycle, and concentrate along with inlet and outlet pressure and water temperature were continuously monitored and automatically logged.

### 2.2. Batch FF lab column testing

FF testing in batch mode was performed using the setup shown in Fig. S2 using concentrate from the holding tanks. Removal of PFAS from the concentrate using FF was evaluated, with and without dosing of the co-surfactants listed in Table 1 to determine if PFAS removal could be enhanced.

A 1000 mL surfactant stock solution of 1.1 mM was prepared for each surfactant using ultrapure water (Milli-Q™, IQ 7000) before each set of tests and mixed for 10 min using a magnetic stirrer. Concentrate was collected in 25 L polyethylene high density (PE-HD) plastic containers from the concentrate holding tanks and used to fill the lab scale FF test column. After filling the column with concentrate, the air pump (JBL model A100) introduced air into the bottom of the glass FF column (inner diameter 52 mm, height 550 mm, Saveen and Werner) through a sintered glass filter creating bubbles which moved upwards through the water column. Airflow was measured through a rotameter (ZYIA

**Table 1**  
Surfactants tested in Trial 1 batch lab scale FF, CAS (Chemical Abstracts Service) number, surfactant classification, additional information provided in Table S3.

Name	CAS nr.	Surfactant classification
Montaline™ C 40	164288-56-6	cationic
Marlinat™ TM 242/28	68891-38-3	anionic
Simulsol™ TM SL 10	110615-47-9	non-ionic
Linear alkylbenzene sulfonate (LAS)	68584-22-5	anionic

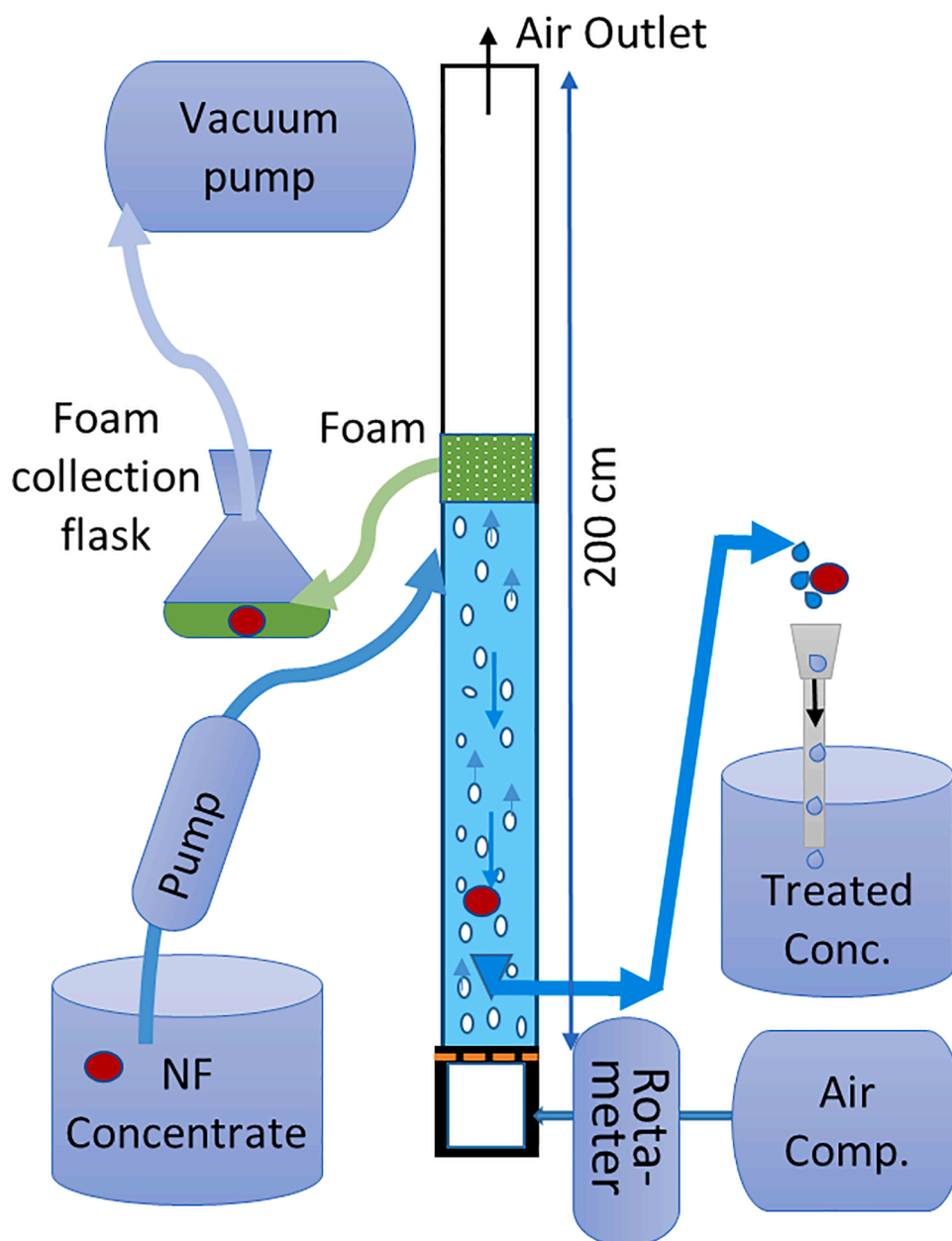
Instrument Company, FL3-1) and maintained approximately  $1 \text{ L min}^{-1}$ . To determine a minimum dose for each surfactant, a series of pre-tests were performed to determine the surfactant dose required to form a foam cushion approximately 2 cm above the water level. After all runs, the column was rinsed with tap water until no surfactant bubbles were evident, then rinsed and filled with ultrapure water, shaken, then emptied.

After determination of the minimum dose, single runs were made using no surfactant, the minimum dosage, two and five times the minimum dose (Table S4) for the 4 surfactants for a total of 16 experimental runs. For each run, the column was filled with 250 mL, the desired co-surfactant dose was added using a plastic pipette, air bubbles were introduced at  $1.7 \text{ L min}^{-1}$ . Foam forming on the water surface was removed from the column using a vacuum pump (GAST, Model DOA-P504-BN) with silicone suction tube inserted from the top of the column for a period of 2 to 3 min until the water level in the column was reduced to 200 mL. The column water was emptied directly into 250 mL

polyethylene high density (PE-HD) plastic sample bottles. Foamate was emptied from the foam collection bottle directly into 250 mL PE-HD sample bottles. The sampled foamate volumes were weighed using a Mettler Toledo® scale (Model PB602-S/FACT) and ultrapure water added to provide 250 mL of sample for analysis by a commercial laboratory (ALS OV-34aQ). Between each run, the column and foam collection bottle were rinsed with tap water until no surfactant bubbles were evident, then filled with ultrapure water, shaken, then emptied. The column was then inverted and any water remaining in the sintered glass filter was blow out using the air pump for approximately 2 min. Samples were taken of both the ultrapure and tap water (Table S5) for PFAS analysis to detect PFAS contamination of the experiment.

### 2.3. Continuous FF pilot column testing

Continuous tests using zero co-surfactant, minimum dose, two and three times the minimum dose of the cationic surfactant Montaline™



**Fig. 1.** Experimental setup with for continuous pilot foam fractionation. Red dots are sampling locations for untreated NF concentrate (in tank and column before start), foamate, and treated concentrate effluent.

C40 were run, each test in triplicate for a total of 12 runs. Montaline™ C40 was utilized in the continuous testing since it was assessed as the most effective surfactant based on the Trial 1 testing. The FF column shown in Fig. 1 was operated with air flow counter-current to water flow provided using a peristaltic pump (Watson Marlow Pumps Group 323E/D 400RPM) at the flowrate determined to provide a 10 min theoretical contact time (CT).

Oil-free air was introduced into the bottom of the column (acrylic plastic, inner diameter 54 mm, height = 2.0 m) through a membrane (Xylem Silver Series II ®). Airflow was measured and maintained at 4 L min<sup>-1</sup> using a rotameter (ZYIA instrument company, FL3-1) with air bubbles moving upwards through the water column with air exiting the column. Foam forming on the water surface was removed from the column using a vacuum pump (GAST, Model DOA-P504-BN) with silicone suction tube inserted through a sampling port. Retentate samples were collected from the exit tube at 5, 10 and 20 min intervals. Untreated concentrate samples were taken directly from the holding tanks and column sample before the start of the experiment and foamate was sampled directly from the foamate collection flask at the end of the runs. The untreated concentrate and treated retentate were measured for pH (Knick Portavo 902 PH), temperature, and conductivity (WTW Cond 340i). In Table 2 is shown the operation parameters for Trial 1 and 2 tests and in Table S6 is shown the surfactant dose and surfactant to PFAS mole ratio for each of the 12 runs. Samples were taken of both the ultrapure and tap water (Table S5). Trial 2 test parameters were selected based on experience from previous FF treatment of landfill leachate (McCleaf et al., 2021).

#### 2.4. Aerosol release from the continuous FF pilot column

Two additional continuous runs were made to determine the impact of PFAS escape via aerosols from the column in Trial 2. An aerosol water trap containing ultrapure water with a PFAS concentration below the LOD was utilized to detect if PFAS was leaving the column as an aerosol. The column was operated at 4 L min<sup>-1</sup> airflow without surfactant addition with the same operating conditions as other continuous FF tests except the vacuum pump was not used for foam collection and foam was allowed to accumulate at the surface of the water column. The outlet at the top of the column was closed and exhaust air from the top of the column was led via PE tubing to the water trap consisting of a 250 mL HDPE sample bottle. The foam level was monitored so as insure only exhaust air and not foam entered the exhaust tubing. New water trap bottles were used for each test at three time intervals: 0–5, 5–10, and 10–20 min and water analyzed for PFAS to determine if PFAS had been transferred as aerosols from the column exhaust air to the water trap.

#### 2.5. Sampling and analysis

Samples were refrigerated at 8 °C then shipped to ALS Scandinavian located in Stockholm, Sweden, for PFAS analysis (ALS OV-34aQ) using liquid chromatography - tandem mass spectrometry (LC-MS/MS). For quality assurance, one blank sample, one spiked sample, and one duplicate sample was analyzed for every 20 samples. Thirty-four PFAS compounds were included in the laboratory analysis as listed in

**Table 2**

Test parameters for Trial 1 lab scale batch tests and Trial 2 pilot scale continuous tests.

Test parameter	Trial 1	Trial 2
Water column height (m)	0.12	1.0
Water volume treated (L)	0.25	7.1
Air flowrate (L min <sup>-1</sup> m <sup>-2</sup> ) <sup>a</sup>	790	1 750
Concentrate flowrate (mL min <sup>-1</sup> )	Zero - batch	229
Contact time (min)	2–3	10

<sup>a</sup> Air flowrate expressed as air flow per area of the water column.

Table S1. For the determination of PFAS removal calculations and graphing, the PFAS results with concentrations lower than the LOD were treated as having the value of half the LOD which is recognized to introduce bias into calculated removal efficiencies but still indicates the effectiveness of the removal process. General chemical analysis for samples from the NF and biofilter pilot were performed by Uppsala Water and Waste Ltd.'s certified water laboratory for the water quality parameters included in Table S2. General chemical analysis for the FF experiments was performed by ALS Scandinavian (ALS GV-3) for water quality parameters included in Table S7.

The removal efficiency was calculated as shown in the following equation.

$$\text{RemovalEfficiency(\%)} = 100 - \frac{C}{C_0} \quad (1)$$

where  $C$  is the PFAS concentration in the NF permeate, Trial 1 batch column water or the Trial 2 continuous exit water (ng L<sup>-1</sup>).  $C_0$  is the untreated concentration (ng L<sup>-1</sup>) in the NF feedwater, or before the start of the Trial 1 batch test or the water entering the Trial 2 continuous process. For Trial 1 and Trial 2 the average PFAS concentration of the holding tank water was used for  $C_0$ .

Contact time ( $C_t$ ) for the water in the in the continuous column tests was calculated using the following equation.

$$C_t = \frac{\text{Volume}}{\text{Flowrate}} \quad (2)$$

where  $C_t$  is the contact time (min), volume is the volume of water in the column (L) and the flowrate (L min<sup>-1</sup>).

Mole ratio co-surfactant to PFAS in the untreated concentrate was calculated using the following equation.

$$\text{Mole Ratio Surfactant to PFAS} = \frac{\text{Moles surfatant}}{\text{Total Moles PFAS avg}} \quad (3)$$

where moles surfactant is the moles of surfactant added to the concentrate tested in the column, moles PFAS<sub>avg</sub> is the total average moles of PFAS detected in the untreated concentrate for Trial 1 sample point 6 and for Trial 2 sample point 7, respectively. The moles of each individual PFAS (Table 3) were calculated based on their individual concentration and the resulting individual mole values added to give the total moles PFAS for Trial 1 and 2. If concentrations were below the LOD, one-half of the LOD was assumed for that individual PFAS.

### 3. Results and discussion

#### 3.1. NF pilot

The two-stage NF pilot reduced the PFAS concentration in the NF

**Table 3**

The average PFAS concentration for NF concentrate water used in Trial 1 and 2.

PFAS	Trial 1 concentrate (n = 3) (ng L <sup>-1</sup> )	Trial 2 concentrate (n = 24) (ng L <sup>-1</sup> )
PFBA (C <sub>3</sub> )	15±3.8	11±5.8
PFPeA (C <sub>4</sub> )	13±0.8	9.9 ± 3.5
PFHxA (C <sub>5</sub> )	30±1.4	30±2.4
PFHpA (C <sub>6</sub> )	<10	<10
PFOA (C <sub>7</sub> )	13±0.5	18±4.0
PFNA (C <sub>8</sub> )	<10	<10
PFDA (C <sub>9</sub> )	<10	<10
PFBS (C <sub>4</sub> )	27±0.8	28±3.4
PFPeS (C <sub>5</sub> )	24±3.4	26±3.7
PFHxS (C <sub>6</sub> )	162±7.4	184±22
PFOS (C <sub>8</sub> )	44±12	63±16
6:2 FTSA (C <sub>6</sub> )	<10±0	<10
∑PFAS	348±19	389±42

feedwater by 98% from a total of approximately 77 to 1.4 ng L<sup>-1</sup> (Table S8) and reduced water hardness by 44%, uranium-238 by 97%, and DOC from 2.7 to <1 mg L<sup>-1</sup> (Table S2). Total and individual PFAS removal efficiencies (Table S8) are similar to the results given in Zhi et al. (2022) and Liu et al. (2022) for NF90 membranes, while the overall removal efficiency of 98% is the same as found for a two-stage membrane process using NF90 and NF270 membrane (Franke et al., 2021).

The greatest PFAS removal efficiency was found for PFOS-C<sub>8</sub> with 99% followed by PFHxA, PFHxS, and PFPeS with 97%, PFOA and by PFBS with 96%, PFPeA 93% and PFHpA 89% if half the LOD is assumed for permeate values below LOD. The  $\sum$ PFAS<sub>4</sub> concentration was reduced from approximately 56 to 1 ng L<sup>-1</sup> if zero is assumed for individual PFAS which were below the LOD for all samples taken. The concentrate averaged 393 ng L<sup>-1</sup> total detectable PFAS concentration, which is 5 times greater than for the feedwater which is expected with a permeate recovery of 80%. The PFAS concentration of the water used in FF testing for Trial 1 and Trial 2 are shown in Table 3.

### 3.2. Trial 1 batch FF results

The PFAS removal efficiency for FF without co-surfactant addition averaged 78±4% with greater than 94% removal efficiency for PFOA, PFHxS and PFOS as shown in Table S9. Comparison of the four surfactants tested indicates (Table S10) the greatest increase in total PFAS removal efficiency was provided by the cationic surfactant Montaline™ C40 with 17% increase, while Marlinat™ 242/28 provided 2% increase, Simulsol™ SL 10 4% increase, and LAS 3% decrease (Fig. 2, Fig. S3). As shown in Fig. 2, removal efficiency improved with increased dosage of the co-surfactant Montaline™ C40 with an increase of 67%, 59%, 83% and 44%, respectively, for PFPeA, PFHxA, PFBS, and PFPeS at the maximum dosage of the co-surfactant. Longer chain PFAS PFOA, PFHxS, and PFOS, had greater than 95% removal efficiency without the addition of co-surfactant. Improved removal of PFAS with a cationic co-surfactant corresponds with the findings Yueh-Feng et al. (2021) for wastewater. Improvement of removal specifically for PFPeA, PFHxA, PFBS, and PFPeS with cationic co-surfactant addition confirms the findings of Vo et al. (2023) for landfill leachate. Similar to Vo et al. (2023), the removal of PFBA appears not to be improved or even negatively affected by the addition of the cationic co-surfactant.

Based on the results from Trial 1 the cationic co-surfactant

Montaline™ C40 was chosen for Trial 2 due the improvement in removal efficiency for the PFAS with perfluorocarbon chain C<sub>4</sub> through C<sub>6</sub> which provided a greater total PFAS removal efficiency.

### 3.3. Trial 2 continuous FF results

As shown in Fig. 3 the maximum dosage of cationic co-surfactant increased removal for PFPeA to 59%, PFHxA to 99%, and PFBS to 96% with removal efficiencies increasing with increasing dose of co-surfactant. PFOA, PFPeS, PFHxS, PFOS had greater than 99% removal efficiency without the addition of co-surfactant. Similar to Trial 1, removal of PFBA appears not to be improved by the addition of the cationic co-surfactant. Average removal efficiency for the three runs for each co-surfactant dose is shown in Table S11 along with PFAS concentration in the effluent water and foamate.

The average removal of 90% with air alone and 94% the maximum cationic co-surfactant dose is greater than the 81% removal reported by Dai et al. (2019), 76% by McCleaf et al. (2021), and 60% for Smith et al. (2022) for FF of landfill leachate. The greater average removal achieved in the present study, however, cannot be credited to a greater relative makeup of long-chain PFAS which are more hydrophobic and better removed by FF than short-chains. The present study's 70% long-chain makeup is less than Dai et al. (2019)'s reported 90% long-chain makeup and McCleaf et al. (2021)'s reported 80%. The better removal efficiency may be due to the polymer based NF antiscalant chemical used for chelation with metal ions, e.g., Ca<sup>+2</sup> and Mg<sup>+2</sup>, and dispersion of microcrystal particles to prevent scaling (Yu et al., 2020) of the NF membrane. Due to size exclusion, the antiscalant polymer, which is typically greater than 2000 Daltons, remains on the membrane's concentrate side. The antiscalant polymer in the concentrate may increase the hydrophobicity of the PFAS due to charge interactions between the polymer and PFAS molecule, thereby making FF removal more effective.

A common thread between these studies and the present work is higher removal efficiency of individual long-chain PFOA, PFHxS, PFOS than for short-chain PFAS, which is also reported for FF treatment of AFFF contaminated groundwater and for PFOS in wastewater (Buckley et al., 2021; Meng et al., 2018).

As shown in Table S11, the total PFAS removal efficiency for all three co-surfactant doses was 93% by the 5 min sampling time which indicates

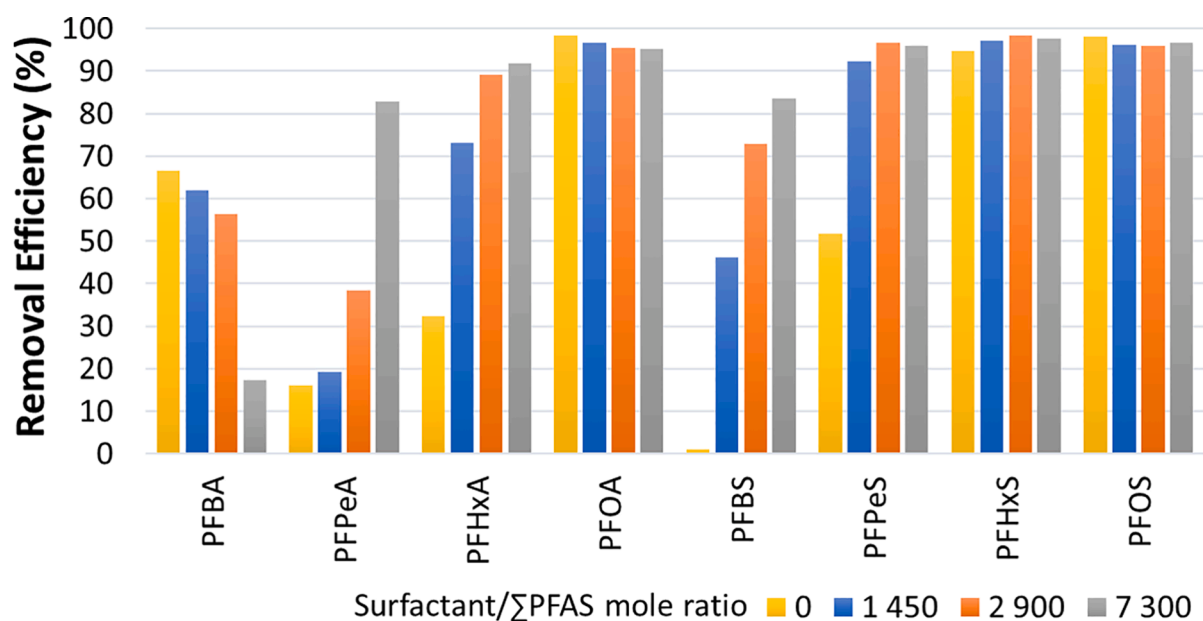


Fig. 2. Trial 1 batch tests removal efficiency for each PFAS for Montaline™ C40 surfactant.

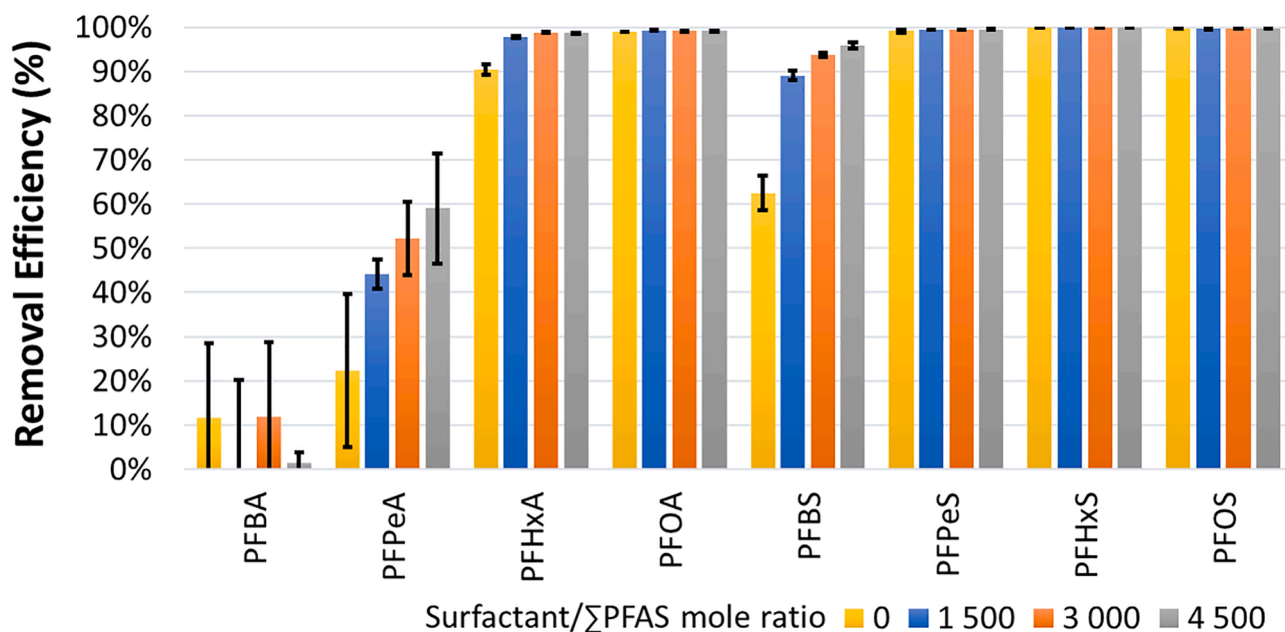


Fig. 3. Average PFAS removal efficiency at the 20 min sample time Trial 2 continuous experiments, four runs with different co-surfactant dosage, each run repeated three times. Standard deviation for each run and dosage ( $n = 3$ ).

removal is initiated quickly. The no dose runs averaged 89–90%±1 for the 5, 10, and 20 min sampling times while for the maximum dosage of 4 500 mole ratio co-surfactant to total untreated PFAS achieved 94%±1, an increase of 4% which is attained chiefly by improved removal of the C<sub>4</sub>–C<sub>5</sub> perfluorocarbon PFAS.

### 3.4. Trial 1 batch mode and trial 2 continuous mode foamate collection parameters

As shown in Tables S12 and S13, the foam to water ratio (FWR) averaged 26% for Trial 1 batch mode and 11% for Trial 2 continuous mode which can be compared to the 21–22% (Robey et al., 2020; McCleaf et al., 2021) reported for FF for PFAS removal from landfill leachate. The Trial 1 foam average enrichment factor 2.5 and Trial 2 factor of 7.7 is less than the 8400 enrichment factor of Meng et al. (2018) which was attained using a non-ionic hydrocarbon surfactant to enhance removal but similar to the factor of 3.7 reported by Robey et al. (2020) and 2.9 from McCleaf et al. (2021) for landfill leachate where no co-surfactants were added. The greater foam to water ratio and lower foam enrichment factor for Trial 1 compared to Trial 2 is indicative of the improved removal efficiency provided by Trial 2's higher water column, 1 m depth versus in Trial 1's 12 cm depth, and Trial 2's longer contact time of 20 min compared to 2–3 min in Trial 1.

PFAS foamate recovery averaged 66% with decreasing foamate recovery with increasing co-surfactant dose for Trial 1 and averaged 82% for Trial 2 and appeared not correlated to co-surfactant dose. The average PFAS mass balance of 78% for Trial 1 and 89% for Trial 2 is greater than the 72% reported by McCleaf et al. (2021) for landfill leachate. For Trial 1, the decreasing enrichment factor and foamate PFAS recovery with increasing co-surfactant dose, may be due to that greater dosage of co-surfactant resulted in PFAS more readily adhering to the walls of the FF column, thereby not being included in the mass balance.

Analysis of the aerosol trap water during Trial 2 indicated that PFOS was captured and averaged  $0.5 \pm 0.2 \text{ ng L}^{-1}$  for the two aerosol runs with a total of six samples. All other PFAS were below the LOD. This indicates that aerosols are created by the study's FF process as has been established for aeration processes by Ahrens et al. (2011), however the Trial 2's utilization of vacuum collection of the foam or the greater

column height (200 cm versus 55 cm in Trial 2) may mitigate aerosol release from the column (Smith et al., 2022) resulting in higher foamate recovery for Trial 2 than Trial 1. This raises the possibility of the release of more volatile PFAS such as FTASs and FTOHs which were not analyzed in this work. Similarly, the variability in the removal efficiency of short chained PFBA and PFPeA compared to the other PFAS in Trial 1, standard deviation 41% and 26%, respectively (Table S9) and compared to the variability in Trial 2 efficiencies (Table S11) may also be attributable to aerosolization.

The fate of the co-surfactants in Trial 1 and 2 was not investigated, however, the co-surfactants may be partly captured during the FF process in the foamate while residual co-surfactant may remain in the FF retentate. Release of co-surfactant into the environment could be problematic. For example, Montaline™ 40, the cationic co-surfactant in Trial 2, is readily biodegradable with low bioaccumulation potential, however, the maximum dosage of  $1 600 \mu\text{g L}^{-1}$  in Trial 2 is only slightly lower than the lethal concentration 50 (LC50) of 1.7 to 5 mg L<sup>-1</sup> for fresh water fish and is in general considered very toxic to aquatic life (SDS Montaline™ C40).

## 4. Conclusions and future work

Based on results of the present work the novel combination of NF for drinking water treatment coupled with FF for concentrate treatment was able to reduce total detectable PFAS in the permeate from approximately  $77 \text{ ng L}^{-1}$  to approximately  $1.4 \pm 0.4$  and  $\sum\text{PFAS}_4$  from  $56 \text{ ng L}^{-1}$  to  $1.0 \pm 0.2 \text{ ng L}^{-1}$  so as to comply with more stringent drinking water regulations, for example less than  $4 \text{ ng L}^{-1}$  in Sweden and less than  $2 \text{ ng L}^{-1}$  in Denmark for  $\sum\text{PFAS}_4$  (PFOA, PFNA, PFHxS, and PFOS). Additionally, the continuous FF treatment of the PFAS laden concentrate using air alone appears to effectively remove PFAS from concentrate water by approximately 90±1%. The work confirmed NF coupled with FF for concentrate treatment is a viable treatment process train for municipal water providers who are required to meet lower regulatory limits for PFAS in drinking water while addressing the discharge of PFAS back into the environment.

The addition of a cationic co-surfactant to the concentrate before FF increased the PFAS removal via FF to 94%. The FF process with air alone appears to provide effective PFAS removal for longer chained PFAS

wherein PFOA, PFPeS, PFHxS, PFOS each attained 99% removal efficiency. Addition of the cationic co-surfactant increased removal efficiencies for shorter chain PFAS, specifically by 37% for PFPeA, 9% for PFHxA, 34% for PFBS resulting in 59%, 99% and 96% removal efficiency, respectively. Removal of PFBA appeared not to be positively affected by addition of co-surfactant possibly due to the shorter chain length being less amenable to interaction with the longer chained co-surfactant.

For the NF process, greatest rejection was found for PFOS with 99% followed by PFHxA, PFHxS, and PFPeS with 97%, PFOA and PFBS with 96%, PFPeA 93% and PFHpA 89% which is similar to the results found by others and has been associated with the size exclusion mechanism inherent for membrane processes, i.e., larger molecular weight molecules are more effectively rejected.

Based on the Trial 1 batch results, the cationic co-surfactant Montaline™ C40 was found to improve removal efficiency of PFAS from the NF concentrate by 17% compared to an increase of 2% for an anionic surfactant Marlinat™ 242/28, increase of 4% for a non-ionic surfactant Simulsol™ SL 10, and decrease of 3% for an anionic surfactant LAS. The PFAS with greatest improvement in removal efficiency using FF with the cationic co-surfactant were PFPeA, PFHxA, PFBS, and PFPeS with increases of 67%, 59%, 83% and 44%, respectively. The improved

removal efficiency may be due to the interaction of the cationic co-surfactant's positive charged head group with the short-chain PFAS negatively charged head group leading to increased hydrophobicity and better removal with FF.

The study's overall results for NF combined with continuous FF for concentrate treatment is depicted in Fig. 4 which illustrates process flow streams, relative volumes and PFAS concentrations afforded by the process. The study's treatment of NF concentrate using FF results in approximately 2% of the NF feedwater volume being captured as foamate with greater than 3 000 ng L<sup>-1</sup> PFAS. This volume could be further decreased if secondary and tertiary FF is applied (Burns et al., 2021; Buckley et al., 2022) which would be more amenable to destruction techniques such as electrochemical oxidation or evaporation with incineration of residual solids.

The retentate from the continuous FF process was approximately 18% of the NF feedwater and had a PFAS concentration of 35 ng L<sup>-1</sup> which decreased with application of the cationic co-surfactant to 21 ng L<sup>-1</sup>. As shown in Fig. 4, the makeup of the retentate is predominantly composed of PFBA, PFBS, PFPeA and PFHxA, short-chain PFAS as opposed to the greater long-chain makeup of the NF feedwater. Polishing of the FF retentate may be achieved using anion exchange with specialized resin optimized for short-chain PFAS removal. In any case

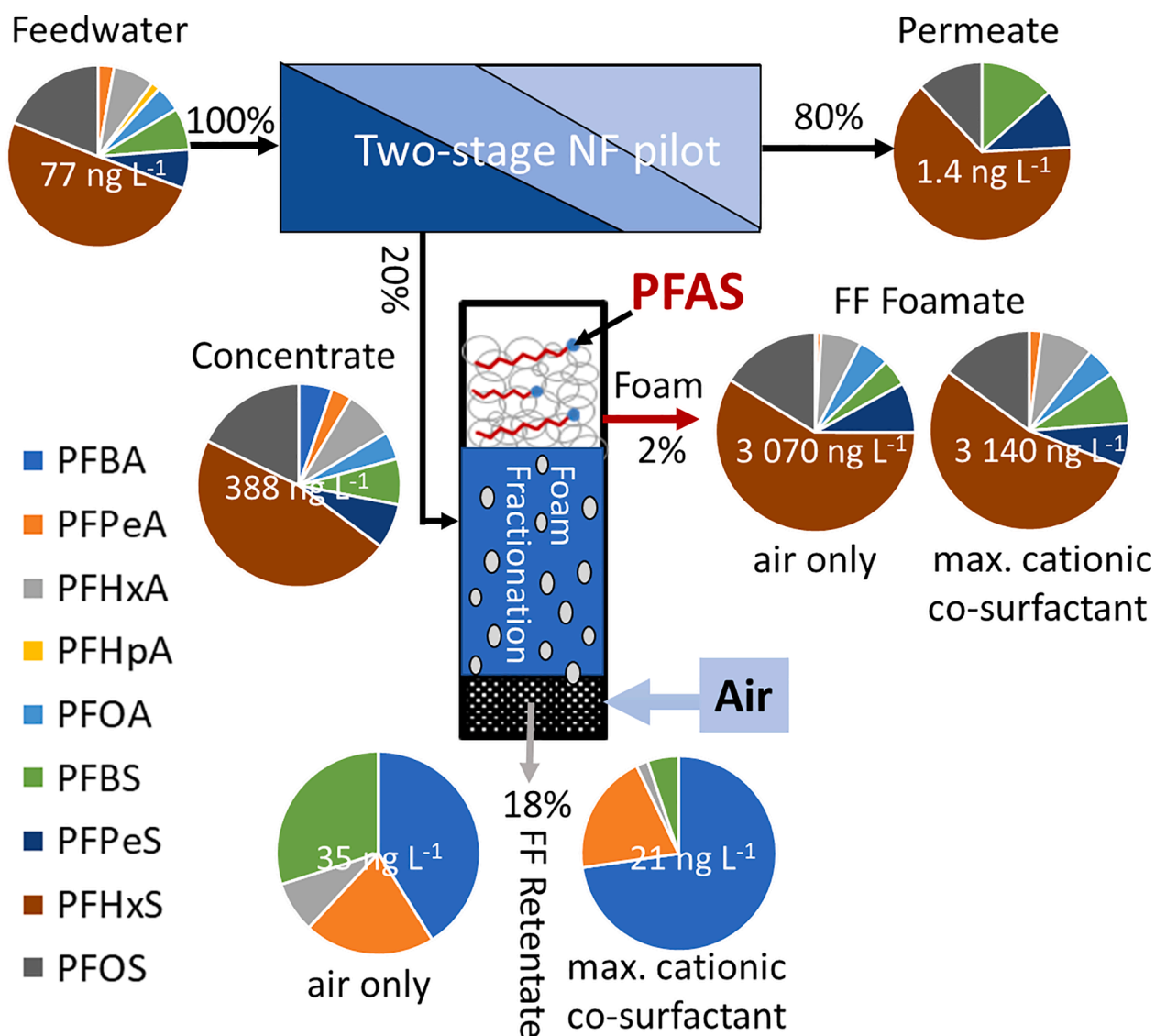


Fig. 4. PFAS process stream volumes (% of feedwater) and PFAS concentrations for NF drinking water treatment process combined with FF for concentrate treatment based on the present study's findings.

the removal of 90% of the PFAS in the FF foamate, decreases the mass of PFAS required to be polished from the retentate before release to the environment which should reduce operation costs of resin regeneration.

Future work should concentrate on investigation of FF process parameters, the effects of co-surfactants, and additional cationic co-surfactants of different chain lengths or membrane antiscalant chemicals that improve the PFAS removal efficiency of the FF process for membrane concentrate and are environmentally friendly and low cost. The trade-off of using co-surfactants for PFAS removal while affecting overall water quality should be investigated along with the fate and toxicity of the co-surfactants. The application of FF in series should be developed for a continuous process for flowrates associated with concentrate streams from full-scale NF/RO applications along with PFAS destruction technologies for FF foamate such as electrochemical oxidation. Polishing processes for FF retentate, for example tailored AIX resins, should be further developed which are effective for high ionic strength applications to provide economical regeneration and avoid PFAS discharge into the environment.

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## CRediT authorship contribution statement

**Philip McCleaf:** Conceptualization, Funding acquisition, Project administration, Resources, Formal analysis, Methodology, Supervision, Visualization, Writing – original draft. **William Stefansson:** Conceptualization, Formal analysis, Investigation, Visualization, Writing – review & editing. **Lutz Ahrens:** Conceptualization, Funding acquisition, Project administration, Supervision, Validation, 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.

## Data availability

Data will be made available on request.

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## Supplementary materials

Supplementary material associated with this article can be found, in the online version, at [doi:10.1016/j.watres.2023.119688](https://doi.org/10.1016/j.watres.2023.119688).

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