#### **ORIGINAL ARTICLE**



# Retention of per- and polyfluoroalkyl substances by syringe filters

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#### Abstract

Per- and polyfluoroalkyl substances (PFAS) are being increasingly measured in water and wastewater due to emerging toxicity concerns and strict regulatory limits. Previous studies have filtered water samples to remove suspended solids before PFAS analysis. However, filtration may introduce negative bias to measured PFAS concentrations. Using a well-controlled syringe pump assembly, we evaluated retention of six perfluoroalkyl carboxylates, three perfluoroalkyl sulfonates, one fluorotelomer sulfonate, and two perfluorooctane sulfonamides by glass-fiber, glass-fiber cellulose acetate, nylon, polyethersulfone, polypropylene, polyvinylidene fluoride/ difluoride, and surfactant-free cellulose acetate filters. The impacts of water quality and operational parameters were also investigated for select filter types. We found that PFAS were retained on all filters, with the glass-fiber cellulose acetate filters demonstrating the lowest retention. For all filters, PFAS retention was linearly related to chain length and hydrophobicity above certain thresholds (*i.e.*, log D higher than 1.5). Importantly, more PFAS were retained at low filtrate volumes, and ~ 30 mL filtrate was required before the retention efficiencies stabilized. Solution pH only affected the retention of perfluorooctane sulfonamides. Pore size (*i.e.*, 0.20, 0.45, 0.70  $\mu$ m), filtration rate (*i.e.*, 0.5, 1.0 mL min<sup>-1</sup>), and PFAS concentration (*i.e.*, 10, 100  $\mu$ g L<sup>-1</sup>), did not exert major influences on PFAS retention. The presence of dissolved organic matter improved PFAS permeation. Based on the reported results, filtration introduces bias and is not recommended for sample pretreatment.

Keywords PFAS · Forever chemical · Filtration · Membrane · Retention · Micropollutant

### Introduction

Per- and polyfluoroalkyl substances (PFAS) encompass over 14,000 unique molecules (EPA 2023a). These contaminants are mobile and persistent in the environment due to their high solubility and the chemical, biological, and thermal stability of the C–F bond (Ahrens and Bundschuh 2014; Krafft and Riess 2015). These properties, along with extensive use of PFAS in industrial and consumer products for the last 50+years, have resulted in widespread PFAS detection in the environment (Dadashi Firouzjaei et al. 2022; Li et al. 2022; Viticoski et al. 2022) and raised concerns about impacts on human health (Ehrlich et al. 2023; Padula Amy

Lee Blaney blaney@umbc.edu et al. 2023). The US Environmental Protection Agency recently proposed maximum contaminant levels of 4 ng L<sup>-1</sup> for perfluorooctanoate (PFOA) and perfluorooctanesulfonate (PFOS), along with a hazard index for four additional PFAS (EPA 2023b). To enforce these goals, sensitive and robust analytical methods are required. Interference or bias introduced from the analytical protocols can greatly influence measured PFAS concentrations. Two primary challenges involve (i) introduction of PFAS from laboratory materials (Denly et al. 2019; Rodowa et al. 2020) and (ii) loss of PFAS due to interactions with sample containers, glassware, and filters (Lath et al. 2019; Zenobio et al. 2022). This article specifically focuses on PFAS retention to filters.

Syringe filters are often used to remove suspended particles from experimental and environmental samples prior to chemical analysis. Previous studies have shown that PFAS interact with common filter materials (Gao et al. 2021; Lundquist et al. 2019; Mulabagal et al. 2018), suggesting that filtration can influence the accuracy of measured PFAS concentrations. To correct for PFAS losses to the filter, some

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analysts add surrogate standards before filtration (Babayev et al. 2022), but this strategy may involve (i) low recoveries for certain PFAS and (ii) high uncertainty stemming from the use of few surrogates for many analytes. To improve PFAS recovery, organic solvents may be employed to rinse the sample container and elute PFAS captured by the filter (Bai and Son 2021; Yao et al. 2022), although this approach would also elute PFAS from retained solids, potentially biasing the measured aqueous-phase concentrations. EPA draft Method 1633 indicates that whole samples should be analyzed without filtration except the de facto filtration that occurs during solid-phase extraction (US EPA 2021). Nevertheless, Table S1 highlights that many recent studies have employed filters, without surrogates or solvent rinses, to prepare aqueous samples for PFAS analysis (Cantoni et al. 2021; Chirikona et al. 2022; Coggan et al. 2019a, b; Gao et al. 2021; Gonzalez et al. 2021; Li et al. 2021; Lundquist et al. 2019; Mulabagal et al. 2018; Saleeby et al. 2021; Wang et al. 2018; Yang et al. 2022). Prior work has confirmed that PFAS interact with filters (Chandramouli et al. 2015; Lath et al. 2019; Sörengård et al. 2020), but more comprehensive investigation is needed to quantify PFAS retention for variable water quality and operating conditions.

Our main objective was to measure the retention of diverse PFAS on common filters during treatment of synthetic and real waters. Six perfluoroalkyl carboxylates, three perfluoroalkyl sulfonates, one fluorotelomer sulfonate, and two perfluorooctane sulfonamides with varying physicochemical properties were chosen as representative PFAS. The impacts of filter chemistry, filtrate volume, pore size, filtration rate, solution pH, water matrix, and PFAS concentration were investigated using a well-controlled syringe pump assembly. The outcomes confirm PFAS losses during filtration, highlight the relative performance of different filter chemistries, and underscore the importance of filtrate volume and water matrix on PFAS retention.

# **Experimental**

# Standards and analysis of per- and polyfluoroalkyl substances

Analytical and mass-labeled PFAS standards were purchased from Wellington Laboratories (Guelph, ON, Canada). Bulk PFAS were obtained from Sigma-Aldrich (St. Louis, MO, USA), Fisher Scientific (Hampton, NH, USA), or Toronto Research Chemicals (North York, ON, Canada) for use in filtration experiments. The 12 targeted PFAS included perfluorobutanoate (PFBA), perfluorohexanoate (PFHxA), PFOA, perfluorodecanoate (PFDA), perfluorododecanoate (PFDoA), perfluorotetradecanoate (PFTeDA), perfluorobutanesulfonate (PFBS), perfluorohexanesulfonate (PFHxS), PFOS, 6:2 fluorotelomer sulfonate (6:2 FtS), perfluorooctanesulfonamide (PFOSA), and N-ethyl perfluorooctane sulfonamidoacetic acid (N-EtFOSAA). These PFAS were selected to inform the potential impacts of chain length, head group, and other structural features on retention to filters. More details of the standards are provided in Text S1, and key physicochemical properties of the targeted PFAS are available in Table S2.

A detailed description of PFAS analysis by liquid chromatography with tandem mass spectrometry (LC–MS/ MS) is available in Text S2, with analytical parameters and performance metrics summarized in Table S3. All measurements were conducted in triplicate, and average concentrations were used for all calculations. The relative standard deviations on experimental data generally ranged from 2% for PFHxA to 10% for PFTeDA.

# **Syringe filters**

Syringe filters containing glass-fiber, glass-fiber cellulose acetate, nylon, polyethersulfone, polypropylene, polyvinylidene fluoride/difluoride, and surfactant-free cellulose acetate membranes were investigated. More details of the specific filters are provided in Table S4. These filter types were selected due to their prevalent use in preparation of experimental and environmental samples for PFAS analysis (Table S1).

# **Experimental design**

Filtration experiments were conducted using a multi-channel syringe pump to (i) carefully control the filtration rate and (ii) enable sample collection as a function of filtrate volume. The experimental solutions were added to 60-mL high-density polyethylene syringes and pumped through the filters at a flow rate of 1 mL min<sup>-1</sup>. Samples (1 mL) were collected in 5 min intervals, namely 0-1, 5-6, 10-11 ... 50–51 min; then, the filter was removed, and another sample was collected at 51–52 min as a quality control. All samples were collected into high-density polyethylene vials prefilled with 1 mL methanol to minimize sorption to the container walls. Those mixtures were vortexed, and 100 µL aliquots were immediately added to 200 µL of methanol, 50 µL of an internal standard solution, and 150  $\mu$ L of a 20 mM ammonium acetate solution before LC-MS/MS analysis (Text S2).

Solutions containing 100  $\mu$ g L<sup>-1</sup> of the 12 targeted PFAS were used to study the impact of filter chemistry, pore size, filtration rate, solution pH, and matrix effects on PFAS retention. The seven filter chemistries described in Sect. "Syringe filters" were evaluated. The influence of 0.20-and 0.45  $\mu$ m pores on PFAS retention were determined with glass-fiber cellulose acetate and polyethersulfone filters.

Two filtration rates, namely 0.5 and 1.0 mL min<sup>-1</sup>, were assessed with the glass-fiber and surfactant-free cellulose acetate filters. Solution pH was maintained at 5.7, 7.6, or 9.0 with 5 mM phosphate buffer to capture the pH range of most environmental waters. Matrix effects were investigated in tap water, groundwater, and deionized water containing 0, 2, or 20 mg<sub>C</sub> L<sup>-1</sup> of Suwannee River natural organic matter (SRNOM). In addition, PFAS levels were varied to examine retention at environmentally (10 µg L<sup>-1</sup>) and experimentally-(100 µg L<sup>-1</sup>) relevant concentrations (Dasu et al. 2022; Li et al., 2020). The solution pH, matrix effects, and PFAS concentration studies were conducted with glass-fiber filters, which have been prevalently used in prior studies (Table S1). In all cases, PFAS retention by the filter was calculated using Eq. 1.

$$\eta_{\text{ret},i}(t) = \frac{1}{\text{PFAS}_{i,\ j=0}} \frac{1}{\mathbf{V}_{t}} \left( \left[ (\text{PFAS}_{i,\ j=0} - \text{PFAS}_{i,\ j=1}) \left( \mathbf{V}_{j=1} - \mathbf{V}_{j=0} \right) \right] + \sum_{j=2}^{t} \left[ \frac{1}{2} \left( \text{PFAS}_{i,\ j=0} - \text{PFAS}_{i,\ j-1} \right) \left( \text{PFAS}_{i,\ j=0} - \text{PFAS}_{i,\ j} \right) \left( \mathbf{V}_{j} - \mathbf{V}_{j-1} \right) \right] \right) 100\%$$
(1)

In Eq. 1,  $\eta_{ret,i}(t)$  is the percent of PFAS *i* retained by the filter at time *t*, PFAS<sub>*i*,*j*=0</sub> is the initial concentration of PFAS *i* in the influent solution, PFAS<sub>*i*,*j*</sub> and PFAS<sub>*i*,*j*-1</sub> are the concentrations of PFAS *i* in the filtrate for sampling events *j* and *j*-1, respectively, V<sub>t</sub> is the filtrate volume at time *t*, and V<sub>*j*</sub> and V<sub>*j*-1</sub> are the filtrate volumes at sampling events *j* and *j*-1, respectively.

#### **Data analysis**

All statistical analyses were performed with OriginPro 2023 (Northampton, MA, USA). A one-way analysis of variance followed by Tukey's honest significant difference test was used to compare normalized PFAS concentrations  $\left(\frac{PFAS_{i,j}}{PFAS_{i,j=0}}\right)$  in the stabilized filtrates for the tested conditions. The normalized PFAS concentrations were employed for statistical analysis because they were more variable than the fraction of retained PFAS mass in the stabilized filtrates. Differences were considered significant if p < 0.05.

## **Results and discussion**

# Effects of filter chemistry, pore size, and filtration rate

For the variable filter chemistries, the total percent of retained PFAS mass is plotted as a function of filtrate volume in Fig. 1. For most conditions, the PFAS retention stabilized after filtration of 30 mL; therefore, the data collected for the 31-51 mL filtrate volumes (n=5) were used to assess apparent equilibrium between the

12 targeted PFAS and seven filter types. The columns in Fig. 2a show the average retention for each PFAS on each filter. PFBA (0-16%), PFOA (0-24%), PFBS (1-24%), PFHxS (0-12%), and PFOS (17-38%) exhibited low retention for all filters. In contrast, PFDoA (63-100%), PFTeDA (83-100%), PFOSA (51-82%), and N-EtFOSAA (72-83%) were well retained for all conditions. The overall performance of the glass-fiber cellulose acetate and surfactant-free cellulose acetate filters was similar to polypropylene (p > 0.05, Table S7), which is encouraged for use with PFAS-containing solutions (US EPA 2021). As indicated by the error bars, PFOS retention to surfactantfree cellulose acetate, nylon, polypropylene, and polyvinylidene fluoride/difluoride filters was more variable than for other PFAS and filters. The average PFAS retention was slightly higher for surfactant-free cellulose acetate, nylon, and polypropylene filters compared to glass-fiber cellulose acetate and polyvinylidene fluoride/difluoride; however, polyvinylidene fluoride/difluoride filters contain -CH<sub>2</sub>CF<sub>2</sub>- groups and raise concerns about introduction of unknown PFAS to the filtrate (Lohmann et al. 2020; Newton et al. 2017). Glass-fiber and polyethersulfone filters retained significantly more PFHxA, PFDA, PFBS, and 6:2 FtS than the other options and should therefore be avoided. Lath et al. had previously reported greater PFOA retention by polyethersulfone filters compared to glass-fiber and polypropylene after a 2-mL conditioning step (Lath et al. 2019). In contrast, Sörengård et al. found that less PFAS were retained in polyethersulfone filters compared to glass-fiber and polypropylene options after passing 5 mL solutions with 21 PFAS (Sörengård et al. 2020). Similarly, Chandramouli et al. reported that 0.45um nylon filters retained more PFAS than glass-fiber and polyethersulfone for pretreatment of 100-500 mL solutions (Chandramouli et al. 2015). We hypothesized that the variable outcomes may stem from differences in filter conditioning and filtrate volume.

The diamonds in Fig. 2a show PFAS mass retention for the first mL of filtrate. Gaps between the diamonds and columns indicate that PFAS were preferentially retained in low-volume filtrates before equilibrating at lower retention efficiencies following filtration of over 30 mL. These outcomes were also apparent from the mass retention profiles in Fig. 1. The greatest differences in retention were observed for perfluoroalkyl sulfonates (*e.g.*, PFHxS, PFOS) and perfluorooctane sulfonamides with environmentally relevant pK<sub>a</sub> values (*e.g.*, PFOSA, N-EtFOSAA) for the nylon, polypropylene, and polyvinylidene fluoride/ difluoride filter chemistries. These findings suggest that filters should be conditioned with at least 30 mL of sample to avoid excessive retention. While retention of long-chain (*e.g.*, PFDoA, PFTeDA) and partially neutral (*e.g.*, PFOSA,



◄Fig. 1 Percent of per- and polyfluoroalkyl substances (PFAS) mass retained as a function of filtrate volume for different filter chemistries. The data were collected by passing solutions with 100  $\mu$ g L<sup>-1</sup> PFAS at pH 7.6 through (a) 0.70-µm glass-fiber, (b) 0.45-µm glassfiber cellulose acetate, (c) 0.45-µm nylon, (d) 0.45-µm polyethersulfone, (e) 0.45-µm polypropylene, (f) 0.45-µm polyvinylidene fluoride/ difluoride, or (g) 0.45-µm surfactant-free cellulose acetate filters. Data are the average of three replicates; for clarity, error bars were not included. The curves are included to highlight the trends for each PFAS. A full data summary is available in Table S5. Acronyms: perfluorobutanoate (PFBA); perfluorohexanoate (PFHxA); PFOA; perfluorodecanoate (PFDA); perfluorododecanoate (PFDoA); perfluorotetradecanoate (PFTeDA); perfluorobutanesulfonate (PFBS); perfluorohexanesulfonate (PFHxS); PFOS; 6:2 fluorotelomer sulfonate (6:2 FtS); perfluorooctanesulfonamide (PFOSA); N-ethyl perfluorooctane sulfonamidoacetic acid (N-EtFOSAA)

N-EtFOSAA) PFAS is unavoidable, glass-fiber cellulose acetate filters exhibited the highest overall PFAS permeation.

Previous studies identified a chain-length dependence on PFAS interactions with various materials (Blaney and He 2021; Chandramouli et al. 2015; Du et al. 2014). In this case, the six perfluoroalkyl carboxylates were used to evaluate trends between PFAS mass retention and log D, which is the log K<sub>ow</sub> value corrected for acid-base speciation; note, log D increases with chain length and is a common measure of hydrophobicity. Figure S1 shows that retention of shortchain PFAS (i.e., PFBA, PFHxA) was generally independent of log D, but removal of long-chain PFAS (i.e., PFOA, PFDA, PFDoA, PFTeDA) was linearly related to log D. The outcomes suggested a log D threshold of approximately 1.5-2.0 before increased PFAS retention to filters, in agreement with conventional definitions of hydrophobicity. The relationships were similar for the glass-fiber cellulose acetate, nylon, polypropylene, polyvinylidene fluoride/ difluoride, and surfactant-free cellulose acetate filters, but the linear trends for the glass-fiber and polyethersulfone filters were shifted to higher retention, presumably due to the additional interaction mechanisms suggested by Fig. 2a. The validity of these relationships could be further confirmed via testing with perfluoropentane carboxylate (PFPeA), perfluoroheptane carboxylate (PFHpA), and perfluorononane carboxylate (PFNA).

We hypothesized that PFAS would be better retained in filters with smaller pores, but Fig. 2b, Figure S2, and Table S8 confirmed that pore size did not significantly affect PFAS retention by glass-fiber cellulose acetate or polyethersulfone filters. One exception was PFOS, which was better retained by the 0.45-µm polyethersulfone filters than the 0.20-µm version (Fig. 2b). The absence of a universal trend was attributed to the much smaller molecular diameters of PFAS, namely 7.4–10.8 Å (ChemSpider, 2022), compared to the 0.20–0.45 µm pores of the filters. However, this outcome contrasted with results from Sörengård et al. (2020), who reported less PFAS retention in polyethersulfone filters with smaller pores. The methodological differences related to filtrate volume between the two studies may have influenced the conflicting outcomes and thereby reinforced the importance of filter conditioning.

Given the high and low PFAS retention by glass-fiber and surfactant-free cellulose acetate filters, respectively, we evaluated these two options for the impacts of filtration rate. Figure 2c shows the results for the 0.45-µm glass-fiber and surfactant-free cellulose acetate filters; the full profiles are available in Figure S3. For glass-fiber filters, PFHxA, PFDA, PFDoA, PFBS, PFOSA, and N-EtFOSAA retention significantly decreased (p < 0.05) when the filtration rate was changed from 1.0 to 0.5 mL min<sup>-1</sup> (Table S9). Similar outcomes were observed for 6:2 FtS and N-EtFOSAA with the surfactant-free cellulose acetate filters; however, PFDA and PFDoA retention significantly increased under these conditions. As a result, no universal rules were proposed for the impact of filtration rate on PFAS retention, but our findings suggest the possible influence of analyst technique (e.g., variable filtration rate when processing samples by hand) on PFAS losses; therefore, controlled filtration systems that maintain a constant filtration rate are recommended to reduce uncertainty.

#### Effects of water quality parameters

Solutions were prepared at pH 5.7, 7.6, and 9.0 to evaluate the impact of pH on PFAS retention by 0.70-µm glassfiber filters (Figure S4). The retention of PFBA, PFHxA, PFOA, PFDA, PFDoA, PFTeDA, PFBS, PFHxS, PFOS, and 6:2 FtS, which predominantly exist as anions at the tested conditions, did not vary with solution pH (Fig. 3a). However, PFOSA and N-EtFOSAA retention did significantly vary as a function of pH (Table S11) due to acid dissociation reactions. From pH 5.7 to 9.0, PFOSA retention decreased from 97 to 56%. This difference was attributed to the prevalence of the neutral PFOSA species, which varied from 77.6% at pH 5.7 to 0.2% at pH 9.0 due to its pK<sub>a</sub> at 6.24 (Rayne and Forest 2009). Similar phenomena were noted for N-EtFOSAA, which has a pK<sub>a</sub> at 3.90 (Rayne and Forest 2009). The greater retention of neutral PFOSA and N-EtFOSAA molecules was explained by the higher hydrophobicity. At pH 5.7, the log D values for PFOSA and N-EtFOSAA were calculated to be 4.75 and 3.33, respectively, but those values decreased to 3.92 and 1.62, respectively, at pH 9.0 due to the aforementioned deprotonation reactions. As noted in Sect. "Effects of filter chemistry, pore size, and filtration rate", PFAS retention decreased for lower log D values (Figure S1).

To inform matrix effects on PFAS retention, filtration tests were conducted with 100  $\mu$ g L<sup>-1</sup> PFAS in deionized water with 5 mM phosphate buffer, tap water, groundwater, and solutions with SRNOM (Figure S5). For most



**∢Fig. 2** Retention of the 12 targeted per- and polyfluoroalkyl substances (PFAS) by filters with different (a) chemistry, (b) pore size, and (c) filtration rate. In (a), all membranes contained 0.45µm pores, except for glass-fiber (0.70µm). The nominal PFAS concentrations in the influent solution were 100  $\mu$ g L<sup>-1</sup> for all conditions, and the influent pH was  $7.6 \pm 0.1$ . Columns are the mean PFAS mass retention calculated from PFAS concentrations in the filtrate after passing 31, 36, 41, 46, and 51 mL of solution through the filters, except for the  $0.5 \text{ mL min}^{-1}$  conditions in (c), when the 15.5, 18.0, 20.5, 23.0, and 25.5 mL samples were used; error bars are the corresponding standard deviation (n=5). The diamonds are PFAS mass retention after passing 1 mL of solution through the filter. The full profiles for (a), (b), and (c) are available in Fig. 1, Figure S2, and Figure S3, respectively; a full data summary is provided in Table S6. The statistical significance of differences in the normalized PFAS concentrations in the filtrate for conditions reported in (a), (b), and (c) are available in Table S7, Table S8, and Table S9, respectively. Acronyms: glass-fiber (GF); glass-fiber cellulose acetate (GFCA); nylon (NL); polyethersulfone (PES); polypropylene (PP); polyvinylidene fluoride/difluoride (PVDF); surfactant-free cellulose acetate (SFCA); perfluorobutanoate (PFBA); perfluorohexanoate (PFHxA); PFOA; perfluorodecanoate (PFDA); perfluorododecanoate (PFDoA); perfluorotetradecanoate (PFTeDA); perfluorobutanesulfonate (PFBS); perfluorohexanesulfonate (PFHxS); PFOS; 6:2 fluorotelomer sulfonate (6:2 FtS); perfluorooctanesulfonamide (PFOSA); N-ethyl perfluorooctane sulfonamidoacetic acid (N-EtFOSAA)

conditions, PFAS retention to glass-fiber filters decreased in the real water matrices (Fig. 3b). This result contrasted with previous findings for nylon filters (Chandramouli et al. 2015). The most significant differences in PFAS retention were observed for solutions containing SRNOM (Table S12), in agreement with previous conclusions (Sörengård et al. 2020). In the deionized water matrix, eight PFAS exhibited greater than 20% retention; however, fewer PFAS met this criterion for solutions with 2 and 20 mg<sub>C</sub>  $L^{-1}$  SRNOM. Compared to deionized water, the average PFAS retention was 5%, 11%, 17%, and 22% lower in tap water, groundwater, the  $2 \text{ mg}_{\text{C}} \text{ L}^{-1}$  solution, and the  $20 \text{ mg}_{\text{C}} \text{ L}^{-1}$  solution, respectively. The variable retention could be attributed to (i) competitive adsorption of organic matter by the filters (Campos Pereira et al. 2018) and/or (ii) PFAS interactions with metals and organic matter in the real waters (Cai et al. 2022; Qi et al. 2022). Based on our aggregate results, PFAS interaction with SRNOM was not considered to be a major cause for the lower retention. Instead, we posit that SRNOM coated the membrane pores and led to lower PFAS capture by the filters. The tap water and groundwater matrices contained less than  $1 \text{ mg}_{C} \text{ L}^{-1}$ SRNOM, suggesting that PFAS interactions with dissolved metals may have enhanced PFAS permeation. Future work is recommended to confirm these mechanisms. Long-chain (e.g., PFDoA, PFTeDA) and partially neutral (e.g., PFOSA,

N-EtFOSAA) PFAS were well retained regardless of the background water quality.

The influent PFAS concentrations were changed from 100  $\mu$ g L<sup>-1</sup> to 10  $\mu$ g L<sup>-1</sup> to determine potential effects on PFAS retention (Figure S6). While some compounds exhibited minor, but significant, changes in retention (Table S13), no clear trends were noted in Fig. 3c. This observation aligns with previously reported results (Chandramouli et al. 2015; Lath et al. 2019) and confirms that PFAS retention was independent of concentration for the tested conditions. We acknowledge that the presence of variable water quality (*e.g.*, dissolved organic matter, metals) may cause differential retention for PFAS across concentration levels. In addition, the effects of water quality parameters on PFAS retention may vary for other filter types. The overall takeaways from this work are summarized in Fig. 4.

### Conclusion

Previous researchers have filtered experimental and environmental samples to remove suspended solids before PFAS analysis. Through carefully designed and controlled studies, we determined that PFAS were retained regardless of filter chemistry. The glass-fiber and polyethersulfone filters retained more PFAS than the other options, while glass-fiber cellulose acetate filters demonstrated the lowest overall PFAS retention and, therefore, best performance. Greater than 50% retention was observed for long-chain (e.g., PFDoA, PFTeDA) and partially neutral (e.g., PFOSA, N-EtFOSAA) PFAS for all filters. PFAS retention was frequently higher for low filtrate volumes, and the retention efficiency did not stabilize until after conditioning with~30 mL solution. Pore size, filtration rate, and PFAS concentration did not exert major influences on PFAS retention. Real water matrices tended to improve PFAS permeation, especially in solutions with more dissolved organic matter. Based on these results, filtration is not recommended for aqueous samples even if surrogates are used to calculate recovery efficiency, because PFAS recovery from the filter will be less than 70% for most compounds. In cases where filtration is unavoidable, solvent could possibly be used to elute PFAS retained on the filter into the filtrate; however, further work is needed to confirm the required solvent volume, calculate recovery efficiencies, and determine the contribution from suspended solids retained by the filter. Instead, centrifugation followed by solid-phase extraction of the supernatant is recommended as a more appropriate approach.



◄Fig. 3 Retention of the 12 targeted per- and polyfluoroalkyl substances (PFAS) for variable (a) solution pH, (b) water matrix, and (c) concentration level. In all cases, 0.70µm glass-fiber filters were employed with a 1.0 mL min<sup>-1</sup> filtration rate. The nominal PFAS concentrations in the influent solution were 100  $\mu$ g L<sup>-1</sup> for all conditions, except in (c). The influent pH was  $7.6 \pm 0.1$ , except in (a). Columns are the mean PFAS mass retention calculated from PFAS concentrations in the filtrate after passing 31, 36, 41, 46, and 51 mL of solution through the filters; error bars are the corresponding standard deviation (n=5). The diamonds are PFAS mass retention after passing 1 mL of solution through the filter. The full profiles for (a), (b), and (c) are available in Figure S4, Figure S5, and Figure S6, respectively; a full data summary is available in Table S10. The statistical significance of differences in the normalized PFAS concentrations in the filtrate for conditions reported in (a), (b), and (c) are available in Table S11, Table S12, and Table S13, respectively. Acronyms: perfluorobutanoate (PFBA); perfluorohexanoate (PFHxA); PFOA; perfluorodecanoate (PFDA); perfluorododecanoate (PFDoA); perfluorotetradecanoate (PFTeDA); perfluorobutanesulfonate (PFBS); perfluorohexanesulfonate (PFHxS); PFOS; 6:2 fluorotelomer sulfonate (6:2 FtS); perfluorooctanesulfonamide (PFOSA); N-ethyl perfluorooctane sulfonamidoacetic acid (N-EtFOSAA)



Fig. 4 Key findings on per- and polyfluoroalkyl substances (PFAS) retention to syringe filters

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Author contributions Ke He contributed to conceptualization, data curation, formal analysis, investigation, methodology, validation, and writing the original draft; Anna Feerick and Hongyue Jin performed investigation; Jahir A. Batista Andrade helped with data curation, formal analysis, methodology; Marylia Duarte Batista and Caitlyn Dugan helped in writing, reviewing, and editing; Lee Blaney contributed to conceptualization, data curation, formal analysis, funding acquisition, methodology, project administration, supervision, resources, visualization, writing the original draft, and writing, reviewing, and editing.

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Availability of data and material All data are reported in the main manuscript or supplementary material. The supplementary material includes the following: PFAS standards and bulk chemicals; LC–MS/ MS protocols for PFAS analysis; summary of previous studies that employed filters for pretreatment of aqueous samples; details of the tested syringe filters; statistical significance of differences in PFAS retention for the tested conditions; total mass retention profiles for PFAS as a function of filtrate volume for the tested conditions; and PFAS retention trends with log D.

Code availability Not applicable.

#### Declarations

Competing interests Not applicable.

Ethics approval Not applicable.

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Consent for publication Not applicable.

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#### References

- Ahrens L, Bundschuh M (2014) Fate and effects of poly- and perfluoroalkyl substances in the aquatic environment: a review. Environ Toxicol Chem 33:1921–1929
- Babayev M, Capozzi SL, Miller P, McLaughlin KR, Medina SS, Byrne S et al (2022) PFAS in drinking water and serum of the people of a southeast Alaska community: a pilot study. Environ Pollut 305:119246
- Bai X, Son Y (2021) Perfluoroalkyl substances (PFAS) in surface water and sediments from two urban watersheds in Nevada, USA. Sci Total Environ 751:141622
- Blaney L, He K (2021) Ion exchange membranes and fibers as passive samplers for chemically-diverse PFAS. Final report for ER20–1073 (limited scope project). 2021
- Cai W, Navarro DA, Du J, Ying G, Yang B, McLaughlin MJ et al (2022) Increasing ionic strength and valency of cations enhance sorption through hydrophobic interactions of PFAS with soil surfaces. Sci Total Environ 817:152975
- Campos Pereira H, Ullberg M, Kleja DB, Gustafsson JP, Ahrens L (2018) Sorption of perfluoroalkyl substances (PFASs) to an organic soil horizon—effect of cation composition and pH. Chemosphere 207:183–191
- Cantoni B, Turolla A, Wellmitz J, Ruhl AS, Antonelli M (2021) Perfluoroalkyl substances (PFAS) adsorption in drinking water by granular activated carbon: influence of activated carbon and PFAS characteristics. Sci Total Environ 795:148821

- Chandramouli B, Benskin JP, Hamilton MC, Cosgrove JR (2015) Sorption of per- and polyfluoroalkyl substances (PFASs) on filter media: implications for phase partitioning studies. Environ Toxicol Chem 34:30–36
- ChemSpider (2022) Available at http://www.chemspider.com/; accessed on July 2, 2022.
- Chirikona F, Quinete N, Gonzalez J, Mutua G, Kimosop S, Orata F (2022) Occurrence and distribution of per- and polyfluoroalkyl substances from multi-industry sources to water, sediments and plants along Nairobi River basin, Kenya. Int J Environ Res Public Health 19(15):8980
- Coggan TL, Anumol T, Pyke J, Shimeta J, Clarke BO (2019a) A single analytical method for the determination of 53 legacy and emerging per- and polyfluoroalkyl substances (PFAS) in aqueous matrices. Anal Bioanal Chem 411:3507–3520
- Coggan TL, Moodie D, Kolobaric A, Szabo D, Shimeta J, Crosbie ND et al (2019b) An investigation into per- and polyfluoroalkyl substances (PFAS) in nineteen Australian wastewater treatment plants (WWTPs). Heliyon 5:e02316
- Dadashi Firouzjaei M, Zolghadr E, Ahmadalipour S, Taghvaei N, Akbari Afkhami F, Nejati S et al (2022) Chemistry, abundance, detection and treatment of per- and polyfluoroalkyl substances in water: a review. Environ Chem Lett 20:661–679
- Dasu K, Xia X, Siriwardena D, Klupinski TP, Seay B (2022) Concentration profiles of per- and polyfluoroalkyl substances in major sources to the environment. J Environ Manage 301:113879
- Denly E, Occhialini J, Bassignani P, Eberle M, Rabah N (2019) Per- and polyfluoroalkyl substances in environmental sampling products: Fact or fiction? Remediat J 29:65–76
- Du Z, Deng S, Bei Y, Huang Q, Wang B, Huang J et al (2014) Adsorption behavior and mechanism of perfluorinated compounds on various adsorbents—a review. J Hazard Mater 274:443–454
- Ehrlich V, Bil W, Vandebriel R, Granum B, Luijten M, Lindeman B et al (2023) Consideration of pathways for immunotoxicity of per- and polyfluoroalkyl substances (PFAS). Environ Health 22:19
- EPA. CompTox Chemicals Dashboard v2.2.1, PFAS Master List of PFAS Substances. Available at: https://comptox.epa.gov/dashb oard/chemical-lists/PFASMASTER. Accessed on June 30, 2023. (2023a)
- EPA. Per- and polyfluoroalkyl substances (PFAS): Proposed PFAS National Primary Drinking Water Regulation. Available at: https://www.epa.gov/sdwa/and-polyfluoroalkyl-substances-pfas. Accessed on June 30, 2023. (2023b)
- Gao P, Cui J, Deng Y (2021) Direct regeneration of ion exchange resins with sulfate radical-based advanced oxidation for enabling a cyclic adsorption—regeneration treatment approach to aqueous perfluorooctanoic acid (PFOA). Chem Eng J 405:126698
- Gonzalez D, Thompson K, Quiñones O, Dickenson E, Bott C (2021) Assessment of PFAS fate, transport, and treatment inhibition associated with a simulated AFFF release within a wastewater treatment plant. Chemosphere 262:127900
- Krafft MP, Riess JG (2015) Selected physicochemical aspects of polyand perfluoroalkylated substances relevant to performance, environment and sustainability—part one. Chemosphere 129:4–19
- Lath S, Knight ER, Navarro DA, Kookana RS, McLaughlin MJ (2019) Sorption of PFOA onto different laboratory materials: filter membranes and centrifuge tubes. Chemosphere 222:671–678
- Li F, Wei Z, He K, Blaney L, Cheng X, Xu T et al (2020) A concentrate-and-destroy technique for degradation of perfluorooctanoic acid in water using a new adsorptive photocatalyst. Water Res 185:116219
- Li R, Alomari S, Islamoglu T, Farha OK, Fernando S, Thagard SM et al (2021) Systematic study on the removal of per- and polyfluoroalkyl substances from contaminated groundwater using metal–organic frameworks. Environ Sci Technol 55:15162–15171

- Li X, Fatowe M, Cui D, Quinete N (2022) Assessment of per- and polyfluoroalkyl substances in Biscayne Bay surface waters and tap waters from South Florida. Sci Total Environ 806:150393
- Lohmann R, Cousins IT, DeWitt JC, Glüge J, Goldenman G, Herzke D et al (2020) Are fluoropolymers really of low concern for human and environmental health and separate from other PFAS? Environ Sci Technol 54:12820–12828
- Lundquist NA, Sweetman MJ, Scroggie KR, Worthington MJH, Esdaile LJ, Alboaiji SFK et al (2019) Polymer supported carbon for safe and effective remediation of PFOA- and PFOS-contaminated water. ACS Sustainable Chemistry Engineering 7:11044–11049
- Mulabagal V, Liu L, Qi J, Wilson C, Hayworth JS (2018) A rapid UHPLC-MS/MS method for simultaneous quantitation of 23 perfluoroalkyl substances (PFAS) in estuarine water. Talanta 190:95–102
- Newton S, McMahen R, Stoeckel JA, Chislock M, Lindstrom A, Strynar M (2017) Novel polyfluorinated compounds identified using high resolution mass spectrometry downstream of manufacturing facilities near Decatur. Ala Environ Sci Technol 51:1544–1552
- Padula Amy M, Ning X, Bakre S, Barrett Emily S, Bastain T, Bennett Deborah H et al (2023) Birth outcomes in relation to prenatal exposure to per- and polyfluoroalkyl substances and stress in the environmental influences on child health outcomes (ECHO) program. Environ Health Perspect 131:037006
- Qi Y, Cao H, Pan W, Wang C, Liang Y (2022) The role of dissolved organic matter during per- and polyfluorinated substance (PFAS) adsorption, degradation, and plant uptake: A review. J Hazard Mater 436:129139
- Rayne S, Forest K (2009) Comment on "Perfluoroalkyl contaminants in an arctic marine food web: trophic magnification and wildlife exposure." Environ Sci Technol 43:6112–6112
- Rodowa AE, Christie E, Sedlak J, Peaslee GF, Bogdan D, DiGuiseppi B et al (2020) Field sampling materials unlikely source of contamination for perfluoroalkyl and polyfluoroalkyl substances in field samples. Environ Sci Technol Lett 7:156–163
- Saleeby B, Shimizu MS, Sanchez Garcia RI, Avery GB, Kieber RJ, Mead RN et al (2021) Isomers of emerging per- and polyfluoroalkyl substances in water and sediment from the Cape Fear River, North Carolina, USA. Chemosphere 262:128359
- Sörengård M, Franke V, Tröger R, Ahrens L (2020) Losses of poly- and perfluoroalkyl substances to syringe filter materials. J Chromatogr A 1609:460430
- US EPA. US Environmental Protection Agency, Draft Method 1633: Analysis of per- and polyfluoroalkyl substances (PFAS) in aqueous, solid, biosolids, and tissue samples by LC-MS/MS. EPA 821-D-21-001. (2021)
- Viticoski RL, Wang D, Feltman MA, Mulabagal V, Rogers SR, Blersch DM et al (2022) Spatial distribution and mass transport of perfluoroalkyl substances (PFAS) in surface water: a statewide evaluation of PFAS occurrence and fate in Alabama. Sci Total Environ 836:155524
- Wang XF, Wang Q, Li ZG, Huang K, Li LD, Zhao DH (2018) Determination of 23 perfluorinated alkylated substances in water and suspended particles by ultra-performance liquid chromatography/tandem mass spectrometry. J Environ Sci Health Part A 53:1277–1283
- Yang Y, Liu S, Wang R, Li C, Tang J, Chen T et al (2022) Diffusive gradients in thin films (DGT) probe for effectively sampling of per- and polyfluoroalkyl substances in waters and sediments. J Environ Sci 121:90–97
- Yao J, Sheng N, Guo Y, Yeung LWY, Dai J, Pan Y (2022) Nontargeted identification and temporal trends of per- and polyfluoroalkyl substances in a fluorochemical industrial zone and adjacent Taihu Lake. Environ Sci Technol 56:7986–7996

Zenobio JE, Salawu OA, Han Z, Adeleye AS (2022) Adsorption of perand polyfluoroalkyl substances (PFAS) to containers. J Hazard Mater Adv 7:100130 **Publisher's Note** Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.