Advanced Filtration Membranes for the Removal of Perfluoroalkyl Species from Water

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ABSTRACT: Perfluorooctane sulfonic acid (PFOS) and perfluorooctanoic acid (PFOA) are two perfluoroalkyl substances that have been shown to result in several adverse health effects, including birth defects, kidney/testicular cancer, as well as liver and thyroid damage. The surfactant nature of PFOS and PFOA in water makes these compounds extremely difficult to remove from drinking water. In this paper, an efficient method to remove PFOS and PFOA from drinking water using linear fluorinated silane-functionalized aluminum oxide hydroxide (γ-AlOOH) nanowiskers was developed. Filters functionalized with linear fluorinated silanes containing 13–17 fluorine atoms were able to remove >90% of the PFOS/PFOA at a very high flux of 1223 L/m²·h. However, due to the hydrophobicity of these linear fluorinated silanes, high pressure drop was also noted across the membrane thickness during the filtration process. To reduce the back-pressure drop, linear fluorinated silanes with appended hydrophilic poly(ethylene glycol) units were synthesized, and it was further demonstrated that the new hydrophilic linear fluorinated silane dramatically reduced the pressure drop of the γ-AlOOH filter while maintaining 99.9% PFOS and PFOA reduction. Adsorption tests were performed to understand the removal mechanism.

INTRODUCTION

Clean water is a vital resource for life. This need has been realized since ancient times where civilizations would emerge and settle near sources of clean water. With the growth of industrial, materials, and agrochemical production, the contamination of aquatic sources is becoming more prevalent worldwide. Many contaminants have been reported in water, including pesticides, heavy metals, biological species, pharmaceutical residues, and polyfluoroalkyl substances (PFAS).1–6 In particular, PFAS (formerly known as perfluorochemicals) have emerged as an increasingly common contaminant in drinking water that are very difficult to remove and persist in the environment.7,8

PFAS are synthetic compounds with multiple C–F bonds that are used in industrial processes for the preparation of fire-resistant foams, protective coatings, and poly(tetrafluoroethylene) products. Perfluorooctane sulfonic acid (PFOS) and perfluorooctanoic acid (PFOA) are two eight-carbon PFAS that are widely observed in water supplies (Figure 1).7 Both PFOS and PFOA are employed for a wide range of applications, including aqueous film-forming foams for firefighting, nonstick cookware, and water-resistant coatings for carpets, leather, and furniture. Due to the long human body accumulation times for PFOS (5.4 years) and PFOA (3.8 years),8 both chemicals have been linked to obesity, cancer, hormone disruption, and high cholesterol levels.9

PFOS and PFOA are typically introduced into the environment from the waste streams of industrial, military, or urban regions. The current state-of-the-art techniques for PFAS removal are adsorption on porous media, membrane filtration, or chemical degradation.9–19 Adsorption on highly porous-activated carbon is the most commonly used method today; however, this approach is both nonselective for PFAS and expensive to implement, which limits its application for large-scale filtration.20

Previously, we developed a cost-effective technology for removing heavy metal ions from drinking water using a thiol-functionalized porous aluminum oxide hydroxide (γ-AlOOH) filter media.21–23 In this work, we utilized the same γ-AlOOH filtration media and investigated the feasibility of fluorinated surface functionalization for PFOS and PFOA removal from contaminated water. The γ-AlOOH substrate was chosen due to its high specific surface area as well as its unique surface characteristics upon contact with water.24 That is, in aqueous solutions, the aluminol group on γ-AlOOH leads to the formation of (Al(OH))2+. As a result, γ-AlOOH carries a high level of positive charge and is commonly used to sequester negatively charged microbes from water.25,26 Thus, our
ultimately, the goal is to develop a multifunctional filtration media that can simultaneously remove toxic heavy metal ions, biological species, and PFOS/PFOA from drinking water.

RESULTS AND DISCUSSION

Our initial approach for the removal of PFOA and PFOS was based on the hypothesis that the perfluorinated side chains would have a favorable fluorophilic C−F···F−C interaction and adsorb onto a surface functionalized with perfluorinated chains (Figure 2). Using commercial perfluorinated silanes, 4603 filters were functionalized and tested for the removal of PFOA and PFOS. The silanes utilized to functionalize the 4603 filters were (tridecafluoro-1,1,2,2-tetrahydrooctyl) trimethoxysilane (F13), (heptadecafluoro-1,1,2,2-tetrahydrodecyl) trimethoxysilane (F17), and (perfluoro(polypropyleneoxy))-methoxypropyl trimethoxysilane (F133−283) (Figure 3). Several functionalization conditions for the functionalization of 4603 filters with fluorinated silanes include solvents, temperatures, and reaction times. We found that using previously optimized functionalization conditions (95% EtOH, cat. AcOH) resulted in filters that performed poorly on these substrates, presumably due to the aggregation of the silanes in solution, which prevented efficient functionalization of the substrate. As perfluorinated compounds are often soluble in fluorinated solvents, we tested trifluoroethanol, trifluorotoluene (TFT), and Novec 7700 for filter conjugation.

A silane concentration screen of the F17-functionalized filters was tested under dynamic filtration conditions at silane concentrations of 0.1, 0.5, and 1%, and the results are shown in Table 1. PFAS removal improved with increasing silane concentration but also resulted in a large pressure drop. This is most likely due to the increased hydrophobicity of the filters.

These fluorinated silane-modified filters performed better than a filter loaded with a cake layer of granular activated carbon (GAC) Filtrasorb 300. Compared to the F13 silanized filter, the F17 silanized filter displayed almost quantitative removal at a similar silane loading. Note that the higher performance of the fluorinated silane-modified filters in this test may have been due to faster kinetics rather than higher capacity since the empty bed contact time of 1 s is less than the typical GAC empty bed contact time for PFAS (>10 min).27

Encouraged by our initial data using commercial silanes, we focused on improving the filter performance by reducing the back pressure under increased flow. The highly fluorinated surface produced high back pressure and prevented efficient filtration. It was hypothesized that a silane containing a polar poly(ethylene glycol) (PEG) terminus and a perfluorinated region would effectively reduce any associated pressure drop while maintaining the effective removal of PFAS. The structure of this amphiphilic silane is shown in Figure 4.

A series of silanes with varied perfluorinated lengths were prepared according to Scheme 1. The MPEG-4-OH (2) was activated using tosyl chloride. The diol (4a−c) was deprotonated using sodium hydride and alkylated with the tosylate (3) to provide alcohol (5a−c) in modest yields. Alcohols 5a−c were activated as the triflate using trifluoromethane sulfonyle chloride and displaced with 3-aminopropyl trimethoxysilane at 50 °C to give the desired products (1a−c) in good yields. Analogs were prepared with 4-, 6-, and 8-perfluorinated carbons giving 8, 12, and 16 fluorines, respectively. Compounds 1a−1c were used directly for the filter conjugation reaction without further purification. Interestingly, these products were soluble under our initial conjugation conditions (95% EtOH, cat. AcOH) and did not require the use of fluorinated solvents. Gratifyingly, the PEG-substituted fluorosilanes proved highly effective in removing PFOA and PFOS with no increase in pressure drop (Table 2). Overall, the PEG silanes with fluorination lengths of F8−F16 were highly effective in removing PFOS/PFOA with the F16−PEG silane (1c) performing the best overall.

Batch-Mode Adsorption for PFOA and PFOS. A static batch-mode adsorption experiment was performed at 25 °C with PFOA and PFOA challenge solutions and the 4603 control, F17-functionalized 4603, and 1c-functionalized 4603 (F16−4PEG), using a challenge solution with 15 000 ppt PFOS and 8800 ppt of PFOA. The total time of the experiment was controlled to be 48 h. The results are shown in Table 3. Functionalization with silane 1c resulted in the highest adsorption of PFOA under the batch-mode conditions, and the F17 had the highest adsorption of PFOS with almost complete removal from the challenge solution. The high adsorption capacity of PFOS is believed to be due to the extra fluorne atoms, which can lead to more fluorine–fluorine interactions, and the results are consistent with our molecular dynamic (MD) simulation. Preliminary data suggests that...
adsorbed PFOS and PFOA do not leach out of used filters when flushed with deionized water; however, more experimentation is needed to confirm this result. The MD simulation, as well as the leaching of the adsorbed PFAS on the membrane, will be reported in future reports.

**CONCLUSIONS**

Filters functionalized with commercial fluorinated silanes proved highly effective at removing PFAS contaminants from the challenge water, however, resulting in a large pressure drop. To improve the performance of these filters, a series of modified amphiphilic silanes were synthesized and evaluated under dynamic and static filtration conditions. Gratifyingly, these silanes provided excellent PFAS removal with no increased pressure drop. The amphiphilic silanes have a high capacity for PFAS removal under static adsorption conditions. Evaluation of these filters under realistic water conditions is currently underway.

**EXPERIMENTAL SECTION**

**Materials.** A commercial γ-AlOOH filtration media 4603 was provided by Ahlstrom Filtration LLC (Mt Holly Springs,
PA). The filter media was used without further pretreatment. Oxygen plasma treatment was accomplished using a Harrick Plasma Cleaner PDC-001. Commercial perfluorosilanes and 3-amino propyl trimethoxysilane were obtained from Gelest Inc. (Morrisville, PA) and used in the as-received form. PFOS, PFOA, trifluorotoluene, Novec 7100, and ethanol were obtained from Sigma-Aldrich (St. Louis, MO) and used without any further purification. Reagents for synthesis were obtained from Sigma-Aldrich or Oakwood Chemicals and used as received. Anhydrous solvents were purchased from Sigma-Aldrich. Filtrasorb 300 granular activated carbon was obtained from Calgon Carbon Corporation (Moon Township, PA).

**Surface Functionalization.** Two functionalization procedures were developed for filter functionalization. All silane functionalization reactions were performed in plastic containers.

**Fluorinated Silane Functionalization.** The base filter is 4603, which consists of γ-AIOOH nanowhiskers sintered onto fiberglass and forming a nonwoven filter. The effective pore size in these filters is around 1 μm. All filters were subjected to an oxygen plasma treatment for 20 min. The filters were then placed in a 1% (v/v) solution of silane in trifluorotoluene with 0.1% (v/v) acetic acid and placed on a shaker table for 24 h (150 rpm). The filters were collected by vacuum filtration, washed with trifluorotoluene and isopropanol, and dried under vacuum.

**PEG-Fluorinated Silane Functionalization.** The 4603 filters were subjected to an oxygen plasma treatment for 20 min. The filters were then placed in a 1% (v/v) solution of silane in 95% ethanol with 0.1% (v/v) acetic acid and placed on a shaker table for 24 h (150 rpm). The filters were collected by vacuum filtration, washed with ethanol, and dried under vacuum.

**Water Filtration Tests.** Artificial water solutions containing PFOS and PFOA were prepared with deionized water spiked with a target 860 ppt of PFOS and 390 ppt of PFOA according to the NSF method 53. The pH of the water was controlled to be around 7.5. Two types of filtration tests were performed: (a) dynamic filtration test: a previously developed set was used for this study. Briefly, a filter with a diameter of 25 mm, a thickness of 0.67 mm, and an average weight of 120 mg was encapsulated inside a 25 mm filter holder made of polycarbonate. A flux of 1223 L/m²h was selected using the recommended flow rate based on NSF protocol P248 (Military Operations Microbiological Water Purifiers) as well as NSF P473-2016 (drinking water treatment units, PFOA and PFOS). Prior to collecting the sample, the filter was flushed with the challenge water for 100 mL, and then a total of 200 mL sample was collected. The total filtration time was around 30 min. To avoid any cross-contamination, all testing tubing was fluorine-free. (b) Batch-mode filtration test: this test was performed by soaking three filters (the same size as used in the dynamic filtration process) in Erlemeyer flasks containing 15 000 ppt PFOS and 8800 ppt PFOA in deionized water. The flask was then placed on a shaker table for 48 h operated at 200 rpm at room temperature. After the desired duration, the concentration of PFOS and PFOA of the water was then analyzed and the adsorption capacity was then calculated. For relative comparison, the GAC was ground to less than 250 μm in particle size to fit inside the polycarbonate filter holder. Approximately 60 mg of F400 was supported onto the 4603 base membrane for the dynamic state testing. The empty bed contact time was around 1 s using the empty bed divided by the flow rate of 10 mL/min. The bed volume after the sample collected was around 0.16 mL.

**PFOS and PFOA Testing.** PFOS and PFOA were analyzed using solid phase extraction (SPE) and LC/MS/MS according to the EPA method 537 version 1.1 (2009). Briefly, the C13 labeled analogs of the target compounds were added to the sample container. The container was agitated, and then the contents of the container (water sample) were passed through a solid phase extraction (SPE) cartridge with a weak anion exchange (WAX) sorbent. Once the PFAS compounds were adsorbed onto the WAX cartridge, the cartridge was eluted with the solvent and the solvent solution was reduced in volume. An aliquot of the final volume was then injected onto an LC/MS/MS operated in electrospray ionization, negative ion mode. Target analytes were then quantified off of a primary transition ion used to generate a calibration curve of relative response (target/isotope) versus concentration. The minimum reporting levels (MRLs) for PFOS and PFOA are 0.4 and 0.2 ppt, respectively.

**Synthesis of PEG Silanes.** Compounds 3, 4a, 4b, 5a, and 5b were prepared according to literature procedures. Briefly, a solution of tetraethyleneglycol monomethyl ether (10.0 g, 48.0 mmol) and triethylamine (6.8 mL, 48.0 mmol) in CH2Cl2 (100 mL) was treated with p-toluenesulfonyl chloride (8.70 g, 45.6 mmol) portionwise at 20 °C under nitrogen. The resulting reaction mixture was stirred for 16 h at 20 °C. The reaction was diluted with water (200 mL), and the aqueous layer was extracted with CH2Cl2 (150 mL × 2). The combined organic fractions were dried (MgSO4) and concentrated under reduced pressure to yield tosylate 3 (15.2 g, 41.9 mmol, 87%) as a colorless oil. 3H NMR (400 MHz; CDCl3): δ 7.80 (d, J = 8.2 Hz, 2H), 7.34 (d, J = 8.0 Hz, 2H), 4.16 (t, J = 4.8 Hz, 2H), 3.68 (t, J = 4.9 Hz, 4H), 3.63 (t, J = 4.5 Hz, 8H), 3.58 (s, 5H), 3.54 (t, J = 4.6 Hz, 3H), 3.37 (s, 3H), 2.45 (s, 3H).

**General Procedure for the Alkylation of Fluorinated Diols 4a–c with 3 To Give 5a–c.** A solution of diol 4a–c (14.5 mmol) in dry dioxane (50 mL) was treated with sodium hydride (60% dispersion in mineral oil, 10.4 mmol) under nitrogen, stirred for 30 min at room temperature, and warmed to 90 °C for 2 h. A solution of tosylate 3 (6.90 mmol) in dry dioxane (19 mL) was added dropwise and the mixture was stirred at 90 °C overnight. The reaction was cooled down to room temperature and quenched with 1 M HCl (2 mL), and the solvent was removed under reduced pressure. The crude compound was dissolved in dichloromethane (200 mL), and a white precipitate was removed via filtration. After solvent removal, the crude product was purified by chromatography on SiO2 (2:1 ethyl acetate/hexanes) to yield 5a–c as the monosubstituted alcohols.

16,17,18,19,20,21,22,23,24,25,26-Hexadecafluoro-2,5,8,11,14-pentaoxatetracosan-24-ol (5c). 3H NMR (400 MHz; CDCl3): δ 4.13–3.99 (m, 4H), 3.79–3.77 (m, 2H), 3.66 (q, J = 7.7 Hz, 12H), 3.56–3.52 (m, 2H), 3.38 (s, 3H); 19F NMR (376 MHz, CDCl3): δ = −123.5 (s), −122.4, −122.0, −119.8.

**General Procedure for Triflation and Aminolysis of Alcohols 5a–c To Give Silanes 1a–c.** A solution of PEG-fluorinated alcohol 5a–c (2.41 mmol) in dry THF (5 mL) was cooled to 0 °C and treated with trifluoromethanesulfonfyl chloride (4.83 mmol) followed by triethylamine (6.03 mmol) under a N2 atmosphere. The reaction was stirred for 30 min at 0 °C and allowed to warm to room temperature overnight, diluted with EtOAc, and washed with water. The organic
extract was dried (MgSO4), filtered, and concentrated in vacuo to give the crude product. The crude product was purified by chromatography on SiO2 (1:1 to 4:1 ethyl acetate/hexanes) to give the intermediate triflate as a tan, oily liquid. The intermediate triflate (2.41 mmol) was dissolved in 3-amino-propyltrimethoxysilane (7.25 mmol) and heated to 50 °C overnight. The reaction was concentrated to a viscous oil, and silanes 1a–c were carried on directly for the functionalization of the membranes.

16,17,18,19,20,21-Dodecafluoro-N-(3-(trimethoxysilyl)propyl)-2,5,8,11,14-pentaoxadocosan-20-amine (1b). 1H NMR (400 MHz; CDCl3): δ 6.01 (t, J = 13.5 Hz, 2H), 3.76–3.75 (m, 2H), 3.66–3.60 (m, 12H), 3.54–3.50 (m, 9H), 3.34 (s, 3H), 3.23 (t, J = 15.7, 1.8 Hz, 1H), 2.71 (t, J = 7.0 Hz, 2H), 1.57 (t, J = 15.6, 7.6 Hz, 2H), 0.64 (t, J = 8.2 Hz, 1H).

16,17,18,19,20,21,22,23,23,24,24,24-Hexadecafluoro-N(3-(trimethoxysilyl)propyl)-2,5,8,11,14-pentaoxtetracosan-24-amine (1c). 1H NMR (400 MHz; CDCl3): δ 3.96 (t, J = 13.9 Hz, 2H), 3.70 (s, 2H), 3.57 (s, 12H), 3.49 (s, 9H), 3.29 (s, 3H), 3.17 (t, J = 16.2 Hz, 2H), 2.65 (t, J = 6.9 Hz, 1H), 1.52 (t, J = 7.7 Hz, 2H), 0.58 (t, J = 7.9 Hz, 2H).

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Notes
The authors declare no competing financial interest.

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