EXECUTIVE SUMMARY

Remediation of Per- and Polyfluoroalkyl Contaminated Groundwater Using Cationic Hydrophobic Polymers As Ultra-high Affinity Sorbents

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SERDP Project ER18-1052

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ACRONYMS AND ABBREVIATIONS

ACF	activated carbon fiber
AFFF	aqueous film-forming foams
ATR	attenuated total reflectance
BET	Brunauer-Emmett-Teller
CMC	critical micelle concentration
DoD	Department of Defense
EPA	U.S. Environmental Protection Agency
FTIR	Fourier transformed infrared
FTS 6:2	fluorotelomer sulfonate 6:2
GAC	granular activated carbon
Na_3PO_4	trisodium phosphate
NaCl	sodium chloride
NaOH	sodium hydroxide
NH_4Cl	ammonium chloride
ng L^{-1}	nanogram(s) per liter
NOM	natural organic matter
PAC	powdered activated carbon
PANI	polyanilines
PFAS	per- and polyfluoroalkyl substances
PFBA	perfluorobutanoic acid
PFBS	perfluorobutane sulfonate
PFHpA	perfluoroheptanoic acid
PFHxS	perfluorohexane sulfonate
PFNA	pefluorononaoic acid
PFOA	perfluorooctanoic acid
PFOS	perfluorooctane sulfonate
POT	poly-o-toluidine
PPy	polypyrroles
RSSCT	rapid small-scale column test
SERDP	Strategic Environmental Restoration and Development Program
SSA	specific surface area
UCMR	Unregulated Contaminant Monitoring Rule
U.S.	United States

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1.0 INTRODUCTION

Per- and polyfluoroalkyl substances (PFAS) such as perfluorooctanoic acid (PFOA) and perfluorooctane sulfonic acid (PFOS) are persistent, bioaccumulative and highly toxic emerging pollutants that are present in the subsurface at many Department of Defense (DoD) facilities due to the use of aqueous film-forming foams (AFFF) in firefighting at airports and training facilities (1–4). In 2016, the U.S. Environmental Protection Agency (EPA) established nationwide drinking water health advisory levels for PFOA and PFOS of a combined 70 nanograms per liter (ng L^{-1}). On June 15, 2022, EPA released revised targets for PFOA (0.004 ng L^{-1}) PFOS (0.02 ng L^{-1}) that were far more stringent than those identified in 2016. In addition, EPA set drinking water health advisory levels for two other PFAS chemicals: perfluorobutane sulfonate (PFBS, 2000 ng L^{-1}) and hexafluoropropylene oxide dimer acid and its ammonium salt (often referred to as "GenX" chemicals, 100 ng L^{-1}). GenX chemicals are considered a replacement for PFOA, and PFBS is considered by the chemical industry as a replacement for PFOS. These health advisory levels are many orders of magnitude lower than PFOA/PFOS concentrations observed in groundwater at numerous historical fire training areas 1 (5–8). Therefore, there is thus an urgent need for effective remediation of PFAS impacted groundwater.

At present, ex situ treatment with granular activated carbon (GAC) is the most commonly used technology for PFAS removal from water, but this approach is very costly and relatively inefficient for removing PFOS, PFOA and shorter length PFAS analogues. In situ and ex situ remedial options for PFAS destruction are limited by the high recalcitrance of these compounds. Numerous studies, including the project team's research, have shown that microbial destruction of perfluorinated PFAS is not a viable treatment approach (9–13). Other known technologies (e.g., photolysis, electrochemical treatment, chemical oxidation, chemical reduction, thermally induced reduction, sonolysis, pyrolysis, etc. [14–20]) have multiple drawbacks (e.g., expensive, involve extreme temperature or pressure conditions, difficult to scale-up). Given these constraints, sorptive removal of PFAS using ultra-high affinity sorbents engineered to achieve long-lasting PFAS sequestration offer promising opportunities for application in ex situ remediation technologies. Likewise, DoD can benefit by using these sorbents in in situ remediation, i.e., mixable media for source zone treatment, media for permeable adsorptive barriers, or an injectable powder similar to colloidal activated carbon. Regeneration can extend the service life and improve the economics of the sorbents. Incineration of spent sorbents can be considered as one option if polymer production is sufficiently low cost.

The thermodynamics and kinetics of surfactant adsorption at solid-water interfaces depends on: (i) sorbent surface chemistry (charge and hydrophobicity) and porosity, (ii) solution chemistry, and (iii) physico-chemical properties of the target molecule (PFAS). Mechanisms of adsorption can include covalent (inner-sphere) or electrostatic (outer-sphere) complexation at surface sites, hydrophobic interaction (including sorbate intermolecular associations such as surface admicelle and hemimicelle formation), ion or water bridging, and hydrogen bonding (Figure ES-1). PFAS sorption is governed by electrostatic, covalent, or hydrophobic interaction. The first two mechanisms involve outer-sphere and inner-sphere adsorption of anionic polar head groups, respectively, at positive-charged sites on sorbent particles.

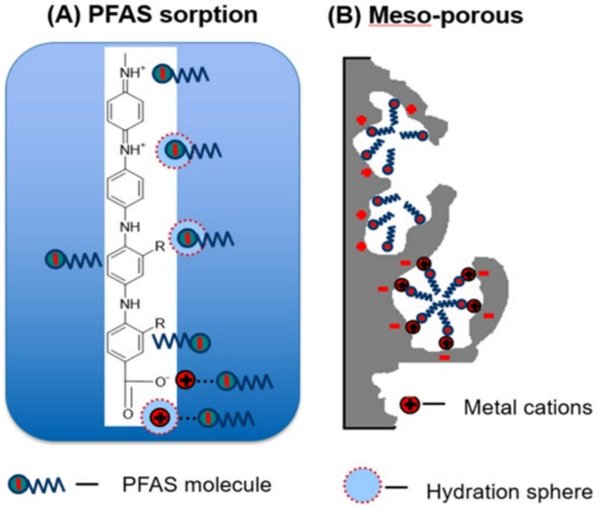

Figure ES-1. Mechanisms of Adsorption

(A) Potential sorption mechanisms of anionic PFAS molecules at moieties of cationic synthetic polymers include (i) inner-sphere or (ii) outer-sphere complexation at quarternized (cationic) N atoms, (iii) H_2O-bridging to polar O-containing sites, and (iv) hydrophobic interaction with apolar domains, and (v) cation bridging or (vi) H_2O-bridging through complexed metal ions. (B) Conformational changes in polymeric sorbents can create mesoporous domains that promote the formation of hemimicelle and admicelle (intermolecular PFAS associations) below the surfactant critical micelle concentration (CMC).

Nano-scale sorbent environments within the sorptive media that combine electrostatic/covalent and hydrophobic bonding modes, such as the mesoporous cationic polymeric sorbents proposed here, are rare in nature, but are postulated to present high-affinity bonding environments for PFAS. The energetics of surfactant adsorption can, therefore, effectively combine the additive effects of multiple bonding modes and Gibbs energies of molecular hydration and intermolecular association.

2.0 OBJECTIVES

PFAS are persistent pollutants present in the subsurface at many DoD facilities, often due to the past use of AFFF in firefighting. PFAS pose a human health threat, necessitating the development of feasible technologies for their removal. At present, ex situ treatment of groundwater by adsorption to GAC is the most commonly used technology for treating PFAS-impacted water. However, this approach is very costly and relatively inefficient at removing PFOA and shorter chain length analogues. Ultra-high affinity sorbents are promising for application in ex situ pump-and-treat adsorption systems. In addition, they offer further opportunities for in situ remedial technologies, including injection as a fine powder or use as filling in subsurface permeable adsorptive barriers.

The objective of this study is to determine the feasibility of utilizing a sorptive remediation approach that exploits multiple, complementary bonding modes (e.g., electrostatic and hydrophobic interactions) for the remediation of PFAS-impacted groundwater. The innovative sorbents are cationic polyaniline (PANI) and polypyrrole (PPy) polymers containing hydrophobic moieties. The hypothesis of this study is that the unique structure of these polymeric materials will allow both strong electrostatic interaction with the anionic head group present in many PFAS compounds and hydrophobic interactions with the fluorinated tail, allowing them to be more selective than GAC and to adsorb a wider range of compounds than anion exchange resins. By using suitable polymer precursors, the charge density and hydrophobicity of these polymers can be tailored to enhance PFAS removal.

3.0 TECHNICAL APPROACH

To meet the project goals, the research plan included six research tasks that address the technical objectives:

1. Develop, characterize, test, and optimize cationic hydrophobic polymers and activated carbon-cationic polymer composites as ultra-high affinity sorbents to sequester PFAS.
2. Develop cationic hydrophobic polymers grafted on GAC and activated carbon fibers and evaluate the capacity of these composites to adsorb PFAS.
3. Evaluate the impact of common co-occurring chemicals and aqueous chemistry on the sorptive removal of PFAS.
4. Assess the feasibility of regenerating the engineered sorbents to enable sorbent reuse.
5. Elucidate the molecular-scale adsorption mechanisms of PFAS on the most effective engineered sorbents under various geochemical conditions to gain insights on how to improve the sorbents.
6. With the most promising sorbents, demonstrate the continuous ex situ removal of PFAS in laboratory columns and develop cost-effective and implementable strategies for the ex situ and in situ remediation of PFAS in impacted groundwater.

This study examined the sorptive remediation of the six anionic PFAS compounds listed in the EPA’s Unregulated Contaminant Monitoring Rule, UCMR3, list (i.e., perfluoroheptanoic acid (PFHpA), PFOA, perfluorononaoic acid (PFNA), PFBS, perfluorohexane sulfonate (PFHxS), PFOS), and two additional compounds often detected in impacted water, i.e., perfluorobutanoic acid (PFBA), and a fluorotelomer compound (fluorotelomer sulfonate 6:2 [FTS 6:2]). Screening studies and kinetic experiments were conducted with PFOA, a ubiquitous chemical of concern in the environment for which EPA established a stringent nationwide drinking water health advisory level in 2019. The project team also evaluated the performance of the polyaniline-based polymers to adsorbed PFAS in two AFFF-impacted groundwater samples collected from a U.S. military facility.

4.0 RESULTS AND DISCUSSION

4.1 SYNTHESIS AND CHARACTERIZATION OF POLYMERIC CATIONIC HYDROPHOBIC ADSORBENTS

An array of pyrrole and several aniline derivatives, with different substitution on the aniline ring were synthesized, i.e., PANI, polyanisidine, poly-o-toluidine (POT), PPy, polyethylaniline, poly-sec-butylaniline, and polynaphthylamine (Figure ES-2). Polymers were synthesized using a simple procedure that involved chemical oxidation of the precursors with ammonium persulfate, and they were characterized using a multi-faceted approach, including Brunauer-Emmett-Teller (BET) surface areas, charge densities, and molecular structures using N2-BET adsorption, electrophoretic mobility, as well as infrared- and X-ray spectroscopy techniques. The affinity and selectivity of these polymers for PFOA were then tested across a wide range of aqueous solution pHs and interpreted based on the characterization data. The results obtained suggested that a combination of electrostatic and hydrophobic interactions controlled the adsorption of PFOA to various polymer types. The project team found that a balance between the addition of hydrophobic substitutions to the polymer backbone and the presence of cationic amine groups provided the highest affinity of the new adsorbents to PFOA. The data also indicated that the affinity of these polymers for the adsorption of PFAS could be tailored by altering substituents on the aniline ring. Among the tested polymers POT and PANI were the most promising adsorbents.

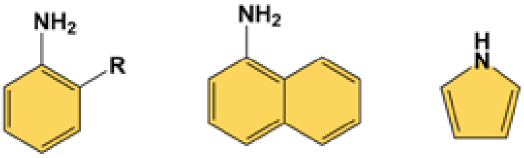

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Figure ES-2. Chemical Structure of the Polymer Precursors Used to Synthesize Polypyrrole and an Array of Aniline Derivatives with Different Substitution on the Aniline Ring

(1) Aniline or substituted anilines: Aniline (R=−H), o-toluidine (R= −CH3), o-anisidine (−OCH3), 2-ethylaniline (−CH2CH3); 2-sec-butylaniline (R= –(CH3)2); (2) 1-naphtylaniline; and (3) pyrrole.

4.2 EVALUATION OF ENGINEERED POLYMERIC MATERIALS AS PFAS ADSORBENTS

This work tested the adsorption of a PFAS mixture (including short-chain congeners) on polyaniline-derived polymers under simulated environmentally-relevant conditions including a range in natural organic matter (NOM) and ion concentrations, mineral anionic and cationic species, and multi-component PFAS solutions. Results were benchmarked against commercial activated carbon (Calgon Filtrasorb 400) and an anion exchange resin (IX, Calgon Calres 2304). The adsorption capacity and binding affinity of the most effective polymers (POT and PANI) at environmental PFAS concentrations was found to be competitive with that of GAC, as well as with recently reported data for novel polymeric and aminated sorbents. The tailored polymers performed similarly or better than GAC and the anion exchange resin tested at elevated NOM and ion concentrations. The engineered adsorbents showed higher sequestration for perfluorosulfonates than their corresponding perfluorocarboxylates, and PFAS adsorption increased with increasing fluorocarbon chain length.

The polymeric adsorbents developed in this project have other technical properties that make them promising for PFAS remediation, including their rapid PFAS uptake kinetics, and the low desorption of adsorbed PFAS to aqueous media. PANI and POT displayed fast PFOA uptake kinetics and their pseudo-second-order rate constants (k2) were in the range of the values obtained for the GAC and ion exchange resin tested in this study, and comparable to those reported for sorbents capable of rapid PFOA removal such as a β-cyclodextrin polymer (21) and poly(ethylenimine)-functionalized cellulose (22) at low PFOA concentrations (1–100 μg L^{-1}). These results suggest that continuous flow treatment systems implementing these polymeric adsorbents would accommodate a short contact time, which is a crucial requirement for the efficient operation of a water treatment facility. Additional experiments demonstrated that PFOS and PFOA exhibited significant desorption hysteresis, indicating that, once adsorbed, the PFAS molecules are strongly retained on the polymer surfaces, even if the solution concentration is significantly reduced. Such properties may be useful for in situ utilization of these adsorbent in the progressive remediation of impacted groundwater matrices.

4.3 ADSORPTION MECHANISMS OF PFAS ON THE MOST EFFECTIVE ENGINEERED SORBENTS

Attenuated total reflectance (ATR) Fourier transform infrared (FTIR) spectroscopy experiments were conducted to investigate the molecular mechanisms of PFAS adsorption on the cationic polymeric adsorbents. The benefit of ATR-FTIR was that it enabled the observation of vibrational modes of bonds present in the adsorbate and the adsorbent, simultaneously, and in the presence of solvent water. Interactions between the "probe molecules"—PFOA and PFOS and "reporter groups"—the IR active functional groups of PANI and POT were observed "in situ" (i.e., in aqueous suspension) in order to better understand the nature of adsorbate-adsorbent molecular bonding. The results supported the hypothesis that both hydrophobic and electrostatic interactions governed the adsorption of anionic PFAS on the cationic polymers, and they elucidated mechanisms of adsorbate-adsorbent interaction that facilitated understanding of the impact of solution chemistry (pH and ionic strength) on PFAS adsorption by the polymeric adsorbents.

4.4 EVALUATION OF THE REGENERATION POTENTIAL OF THE ENGINEERED POLYMERIC ADSORBENTS

Studies were undertaken to explore the regeneration potential of the newly developed adsorbents. Experiments using different alcohols (methanol, ethanol, isopropanol) confirmed that methanol provided the highest PFAS recovery efficiency. Additional regeneration tests performed with aqueous methanol solutions under different conditions (e.g., varying methanol concentration, different salt additives (NaCl, NaOH, NH_4Cl, Na_3PO_4, and sodium acetate), and a wide range of regeneration times and temperatures) showed that the polymeric adsorbent could be regenerated effectively using methanol containing 1% NaCl at ambient temperature. Following five sequential cycles of PFOA loading and regeneration, the polymer maintained both high removal of PFOA and close to complete polymer regeneration.

4.5 ASSESSMENT OF THE STABILITY OF THE ENGINEERED POLYMERIC ADSORBENTS

Additional testing confirmed that the chemical stability of the polymers when stored under ambient conditions and their resistance to microbial attack. The long-term stability of the most promising sorbents (PANI and POT) stored as dry powders at room temperature in the dark for three years was assessed on a yearly basis. Periodic FTIR spectroscopy measurements and adsorption isotherm results indicated that the adsorbents are chemically stable. Furthermore, the results of batch bioassays inoculated with clean soil/sediment obtained from a DoD impacted site, and activated sludge collected from a local municipal wastewater treatment plant, confirmed that the polymeric adsorbents are not susceptible to microbial degradation even in the presence of activated sludge, an inoculum known to harbor a highly active microbial population. The presence of PFAS in the bioassay medium did not have an impact on the rate of polymer degradation.

4.6 SYNTHESIS AND CHARACTERIZATION OF HIGH-SURFACE AREA POLYMERIC ADSORBENTS

Project results have shown that PANI and related amine-containing polymeric materials are high affinity, selective adsorbents for PFAS remediation, with surface area normalized adsorbed mass significantly exceeding that of GAC. However, most of these materials have relatively low specific surface area (SSA), suggesting that increasing their SSA could result in further enhancement of their ability to sequester PFAS. The project team evaluated the effectiveness of various approaches previously shown to yield PANI or PANI-related polymers with high SSA (i.e., chemical crosslinking, synthesis of polymer nanostructures, solvent treatment, thermal treatment; 23–26) in enhancing the PFAS adsorption capacity of the polymeric sorbents. The results obtained confirmed that crosslinking with paraformaldehyde was an effective method to synthesize hyper-crosslinked, permanently porous polymers with SSA (490 m^2 g^{-1} for the best crosslinked polymer compared to 26 m^2 g^{-1} for the original PANI). Crosslinked PANI also exhibited rapid adsorption kinetics and high removal efficiency toward PFOA at environmentally relevant concentrations. However, relative to PANI, the crosslinked polymer showed lower removal efficiency of multicomponent PFAS mixtures and slower sorption kinetics, which were likely due to the narrowed pores created during crosslinking that were easily blocked by preferentially adsorbed long-chain PFAS molecules.

4.7 SYNTHESIS AND CHARACTERIZATION OF GRANULAR POLYMER COMPOSITES

The project investigated the feasibility of grafting novel PANI and PANI-related sorbents onto larger particles to generate polymer composites with a size suitable for application in continuous-flow packed-bed adsorbers. Three different support materials were considered: GAC, activated carbon fiber (ACF), and cellulose crystals. The results obtained showed that cationic polymeric sorbents were effectively grafted onto GAC and ACF in a single synthetic step, as demonstrated by elemental and spectroscopic analysis, and that grafting increased the PFOA adsorption capacity of the hybrid adsorbents when compared to the original polymeric adsorbent. However, the PFAS adsorption capacity of the most effective polymer-GAC composites was comparable to that of GAC. The low PANI content in the best GAC-PANI composites (1.2–2.9%) suggests that the adsorption capacity of the hybrids was mainly attributed to GAC.

4.8 PERFORMANCE OF THE NEW ADSORBENTS IN BENCH-SCALE COLUMN EXPERIMENTS WITH SYNTHETIC GROUNDWATER: IMPACT OF AQUEOUS CHEMISTRY

Rapid small-scale column tests (RSSCT) were conducted to evaluate the performance of PANI to adsorb PFAS (PFBS, PFHxS, PFOS, PFBA, PFHpA, PFOA, PFNA, FTS 6:2, 1 $\mu g\ L^{-1}$ of each) under different aqueous chemistry conditions, including varying levels of NOM (1 versus 5 mg L^{-1}) and ionic strength (0.5 versus. 5.0 mM NaCl), and the presence of other organic co-occurring chemicals (trichloroethylene and toluene, each at 30 $\mu g\ L^{-1}$) that might be present in DoD impacted sites. In each experiment, columns packed with crushed GAC (Filtrasorb 400) were run in parallel to allow benchmarking of the performance of the polymeric adsorbents. The results obtained confirmed that the performance of the polymeric adsorbent was superior to that of GAC for both short chain and long chain PFAS compounds and that PFAS adsorption by GAC was more impacted by increasing NOM- and ionic strength levels compared to PANI. These results are in agreement with previous experiments reported by the project team showing that PFAS adsorption by the sorbents developed in this project is less affected by common competing solutes (NOM and inorganic ions) compared with GAC (27).

4.9 EVALUATION OF THE PERFORMANCE OF THE NEW ADSORBENTS TO TREAT AFFF-IMPACTED GROUNDWATER IN BENCH-SCALE COLUMN EXPERIMENTS

The project team evaluated the performance of the polymeric adsorbent PANI to remove PFAS in RSSCT experiments using two different groundwater samples collected from an AFFF-impacted site at a U.S. military facility ("Hangar 680" and "Site 5"). The average concentrations of the main PFAS detected by liquid chromatography–Tandem Mass Spectrometry in these samples were 19.1 and 2.5 $\mu g\ L^{-1}$, respectively, and the dominant PFAS compounds were PFOS, PFHxS, and PFOA. The PFAS breakthrough curves obtained in these experiments indicated that PANI generally outperformed powdered activated carbon (PAC). Desorption of some PFAS adsorbed onto PAC was observed with time, probably due to competitive adsorption by chemicals with high sorptive affinity (e.g., PFOS). In contrast, the concentration of PFAS in the treated effluent typically did not exceed their respective concentrations in the influent (i.e., $C/Co \leq 1$) even for chemicals that were breaking through completely. These results suggest that PFAS are more strongly adsorbed by PANI than GAC.

5.0 IMPLICATIONS FOR FUTURE RESEARCH AND BENEFITS

This project has led to the development of engineered materials with outstanding adsorptive capacity and binding affinity for PFAS at environmentally relevant concentrations. These novel cationic hydrophobic polymers display fast PFAS uptake kinetics, excellent performance in the presence of elevated natural organic matter and ion concentrations, low desorption of adsorbed PFAS to aqueous media, and facile regeneration at ambient temperature. Moreover, the polymers can be customized to enhance adsorption of PFAS compounds under the wide range of solution chemistry conditions encountered in environmental systems. Collectively, these findings indicate that the tailored polymeric adsorbents offer great promise for the development of effective adsorptive process for the remediation of PFAS impacted groundwater.

The project team is already working on one field application under Strategic Environmental Restoration and Development Program (SERDP), *In Situ Sequestration of PFAS from Impacted Groundwater using Injectable High Affinity Cationic Hydrophobic Polymers*, ER22-3155.[1] It is anticipated that this work will lead to an in situ pilot study and the technology will be applied as a permeable adsorptive barrier to control migration of low PFAS concentrations in groundwater in conjunction with source treatment and management. The research team is also planning to seek funding to demonstrate the feasibility of the new adsorbents at the pilot-scale in an onsite packed-bed filtration system. For field-scale implementation, further work is required to develop a granular porous material with high PANI content in order to prevent the elevated pressure drops associated with powdered adsorbents.

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