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Research Paper

Supercritical water oxidation for the destruction of spent media wastes generated from PFAS treatment

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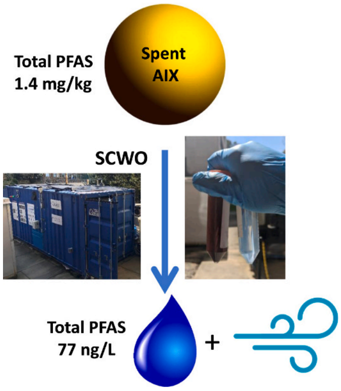
HIGHLIGHTS

- This is the first case study of SCWO application for PFAS-contaminated AIX and GAC.
- A tubular SCWO reactor was used for PFAS destruction on AIX and GAC.
- SCWO eliminated PFAS and completely mineralized the spent GAC and AIX media.
- SCWO demonstrated better PFAS elimination with spent AIX than spent GAC.
- PFCAs elimination was better than PFSAs elimination.

GRAPHICAL ABSTRACT



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ABSTRACT

Granular activated carbon (GAC) and anion exchange resin (AIX) have been successfully demonstrated to remove per- and polyfluoroalkyl substances (PFAS) from contaminated water and wastewater. These treatment technologies, when applied for PFAS removal, generate spent media loaded with a high mass of PFAS requiring further treatment and disposal. This project is the first study on the use of supercritical water oxidation (SCWO) to destroy both the spent media and the PFAS adsorbed onto it. One sample of spent GAC and one sample of spent AIX were collected from full-scale groundwater remediation systems treating PFAS. A second spent AIX sample was collected from a mobile PFAS treatment unit. The total PFAS concentrations reported in the GAC, AIX and second AIX feedstock slurries were 0.21 mg/kg, 1.3 mg/kg and 0.9 mg/kg, respectively. Each feedstock was processed separately in a one (1) wet metric ton per day tubular reactor SCWO system. The study demonstrated that SCWO is a very effective PFAS destruction technology for spent GAC and AIX, derived from water remediation systems treating PFAS. The spent media were completely mineralized to water, carbon dioxide (CO_2) and a negligible amount of residual minerals. Total target PFAS compound concentrations in the SCWO system effluents after treating spent GAC, AIX and second AIX feedstocks were 548, 77 and 796 ng/L, respectively. The

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results indicated that the percentage elimination of perfluorocarboxylic acids (PFCAs) was better than that of perfluosulfonic acids (PFSAs) and long-chain PFAS elimination was better than short-chain PFAS.

1. Introduction

There is an increased public concern over the impact of PFAS on human health and the environment [16,18,22,3,7,27,9], To protect human and ecological health, water utilities and property owners are deploying treatment systems specifically designed to remove PFAS from drinking water supplies. Separation technologies that capture PFAS onto a solid-phase media have been the most widely used methods to remove PFAS from water. The use of media such as granular activated carbon (GAC), anion exchange resin (AIX), or combinations of both is commonplace and has been well documented [19,24,25,32,4]. Although these technologies do not destroy PFAS, they, individually or in combination, have shown to be effective at reducing concentrations of PFAS to the low ng/L range. However, the media have a limited application life typically indicated by PFAS breakthrough in the filter vessel effluent. Once "spent," the PFAS-laden media requires replacement and becomes a residual waste.

Various thermal treatment case studies have demonstrated the potential of incineration to eliminate PFAS [28]. Unfortunately, little is known about the fate of PFAS during incineration and related thermal treatments [2,13,14]. Specifically, there are concerns about whether the organofluorine compounds are fully mineralized and about the formation of incomplete combustion products during thermal treatment yielding possible emissions of short chain and volatile PFAS [8,29]. Thus, alternative PFAS destruction technologies which can fully mineralize organofluorine compounds are in demand for PFAS-laden wastes.

SCWO is a physical-thermal process that relies on the unique reactivity and transport properties of water above its critical point (374 °C and 218 atm). In the unique conditions of supercritical water, organics and oxygen are fully soluble. With the addition of oxygen, highly effective oxidation reactions occur that rapidly and completely mineralizes any organic molecules to carbon dioxide, water, and converts inorganic compounds into insoluble salts [5,11,15,26]. The oxidation reaction is exothermic, releasing energy in the form of heat that can be partially used to pre-heat the feedstock and oxidant inlet stream, and partially converted to electricity. Depending on the concentration and calorific value of the waste feedstock, SCWO can be operated auto-thermally (i.e., no external input of heat). The highly oxidizing environment makes it possible to effectively treat a diverse range of organic compounds and highly complex wastes, such as nerve agents, industrial sludges, biosolid slurries, waste oil, food wastes, and plastics, as well as emerging contaminants such as PFAS or 1,4-dioxane [15]. Typical reaction times are in the order of 5–40 s, resulting in SCWO systems that are quite compact compared to other destruction technologies. When halogenated contaminants are treated, the halogen-carbon bonds are broken and halide anions are released into solution (e.g., fluoride when treating PFAS, or chloride when treating trichloroethylene or tetrachloroethylene). The mechanisms of PFAS mineralization during SCWO are not well understood. It is believed [21,20,29,30] that for perfluoroalkane sulfonates, the initial reaction step is the cleavage of the C-S bond, followed by C-C bond and C-F bond cleavage [6], all mediated by free-radical mechanisms driven by hydroxyl and hydroperoxyl radicals and hydrolysis reactions known to be prevalent in SCWO. Perfluoroalkyl carboxylic acids are believed to be decarboxylated first, followed by similar C-C bond and C-F bond cleavage by free-radical reactions involved in the mineralization of the alkyl moiety of perfluoroalkane sulfonates. Hydrolysis reactions are suspected to play an important role, e.g., in the hydrolysis of carbonyl fluoride [29]. More studies are needed to confirm these reaction mechanisms and to determine the possible roles of the solid media (and their reaction products) on PFAS reaction mechanisms during SCWO.

SCWO is considered a green technology, because of its low energy requirement (e.g., compared to incineration) and it has much cleaner air emission [15,31]. A significant amount of energy is needed to be expended to bring the waste feedstock and the oxidant feed stream above the critical point of water, but a large fraction of this energy and that released by the exothermic reaction can be efficiently recovered in heat exchangers. Compensating for heat losses constrains SCWO to the treatment of concentrated wastes with sufficient organic content for the exothermic reaction to supply the necessary heat. Typically, a minimum calorific content of around 2.4 megajoules per liter (MJ/L), which corresponds to a chemical oxygen demand (COD) of about 120–180 g/L, is needed for autothermal operation. For more dilute streams, external heating or supplemental fuel (e.g., diesel, alcohol, waste oil, etc.) is required, which can increase operating costs, or offset costs if accepted as a waste. SCWO is best suited for the treatment of concentrated organic slurries and concentrates from separation technologies such as GAC and ion exchange, or limited volumes of investigation derived waste. SCWO feedstocks must have a viscosity that renders them pumpable, limiting SCWO, currently, to liquids and slurries. Waste streams that contain excessive grit or abrasive materials and soils cannot currently be processed using SCWO. Lastly, SCWO is currently not economical for very large volumes (e.g., >200 cubic meters per day, m^3/day) of very dilute streams.

While SCWO technology has been reported to destroy PFAS in liquid wastes, such as AFFF ([10,17]), the use of SCWO for the destruction of PFAS captured on spent GAC and AIX has not been investigated before, therefore this is the first case study to examine the elimination of PFAS from the media and the decomposition/mineralization of spent media into water and carbon dioxide. The destruction of PFAS-laden GAC and AIX collected from three different PFAS treatment systems is reported and discussed.

2. Materials and methods

2.1. Spent GAC and AIX sample collection

One spent GAC and two AIX media were collected for this study. The spent GAC and one spent AIX media were collected from full-scale treatment systems to remove PFAS in the extracted groundwater at former Wurtsmith Air Force Base (WAFB). The second AIX media was collected from a mobile treatment train unit installed at the Marine Corps Air Station Miramar (MCAS Miramar) to remove high strength PFAS detected in the base wastewater. The description of spent media collection and PFAS treatment systems are detailed in the Supporting Information (SI) S1. The average PFOS and PFOA mass loading on the spent GAC was calculated to be 60 mg/kg and 11 mg/kg, respectively, based on the full-scale system operation data. The average PFOS and PFOA mass loading on the spent AIX from the full-scale treatment system were calculated to be 13,864 mg/kg and 1659 mg/kg, respectively. The PFAS mass loading on the second spent AIX media from the mobile system could not be calculated due to the lack of system monitoring data. Samples of GAC and AIX spent media were containerized in 20-liter (L) plastic buckets and shipped to Durham, North Carolina for SCWO testing.

2.2. Spent media characterization and preparation of feedstock

The characteristics and the compositions of the PFAS-laden spent media samples (i.e., SCWO feedstock) are presented in Table 1. The AIX samples were used as received for feedstock preparation, whereas the

Table 1
PFAS-laden spent media characterization and feedstock compositions.

Brand, type	Purolite PFA694	Purolite CH-PFAI Treatment Resin	Calgon DSR-A
Spent media	AIX from full-scale system	AIX from mobile system	GAC from full-scale system
Site	WAFB	Miramar AFB	WAFB
Calorific value (MJ/kg$_{dry}$)	34.2	32.6	24.9
Water content (% mass as received, wet basis)	30.0%	45.6%	57.5%
Media concentration in feedstock (g$_{dry}$/L)	80	80	100
Co-fuel additive	none	none	100 g/L hand sanitizer (62% ethanol + <1% Acrylates/C10–13 alkyl acrylates crosspolymers)
Other additives in makeup water	1.2 g/L Na_2CO_3, 7.5 g/L commercial thickener	1.2 g/L Na_2CO_3, 7.5 g/L commercial thickener	1.2 g/L Na_2CO_3, 2.5 g/L commercial thickener

GAC sample was dried in a vacuum oven at 60 °C for 24 h to allow for grinding into a fine powder (particle size <1 mm) using a coffee grinder prior to treatment. Drying and grinding of the GAC was conducted for this demonstration only; future commercial applications will include wet grinding of the GAC if size reduction is needed.

2.3. SCWO System and Operating Conditions

All treatment runs were conducted in a SCWO system located at Duke University, in Durham, North Carolina. The system is a modular SCWO unit, housed in a standard 6-meter shipping container, which can continuously process up to one (1) m^3 of wet feedstock per day. The system had been used before for evaluation of PFAS destruction in AFFF [10]. The SCWO system consists of a tubular reactor, air compressor, tanks, pumps, heat exchangers, etc. and ancillary valves and sensors. A process flow diagram of the system is shown in Fig. 1. The feedstock is pumped into the system using a high-pressure slurry pump which brings the waste to the system pressure of about 240 bar. The pressurized feedstock then passes through a countercurrent heat exchanger where it is preheated to 100–200 °C. This moderate pre-heating avoids charring and gasification of the organics that would occur if slowly heated to the critical temperature. The SCWO system includes an additional supercritical water supply and mixing tee design to rapidly bring the feedstock above the critical temperature, thus minimizing charring, gasification, and corrosion in the subcritical to supercritical transition zone. Compressed air is used as the oxidant because of the risks and costs associated with the use of pure oxygen or hydrogen peroxide. The compressed air, provided by a 15 HP multi-stage air compressor and a mass flow controller, delivers a metered flow of air into the SCWO system. The compressed air is mixed with the pressurized additional water and the air-water mixture is preheated in a countercurrent heat exchanger using heat recovered from the reaction. Additional heating of the air-water mixture is provided during startup and during regular operation if autothermal conditions are not achieved. The supercritical mixture of air and water (typically 550–600 °C) is then mixed with the preheated feedstock at the entrance of the reactor. The flow rates and temperatures of each stream are set such that the feedstock immediately transitions to supercritical conditions upon mixing, thus quickly and efficiently initiating the oxidation reaction.

The SCWO reactor is a horizontal tubular plug flow reactor that is 4 m long with a 1.9 cm internal diameter. As the feedstock passes through the reactor at supercritical conditions, the organics are rapidly oxidized, and the temperature increases due to the released heat from the exothermic oxidation reaction. The resulting fluids pass successively

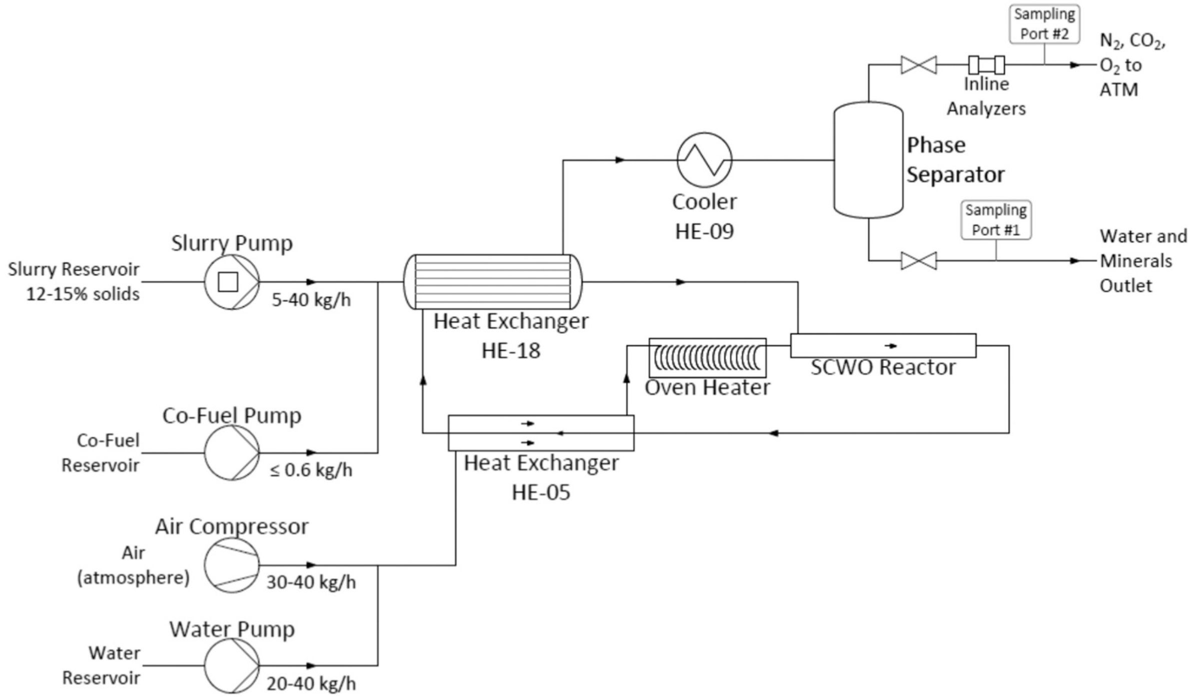

Fig. 1. Schematic of the SCWO pilot system (not to scale, not all details shown).

through two heat exchangers for energy recovery and a final heat exchanger for cooling (to about 25–35 °C). The gas and liquid are then separated and depressurized. The gas (mostly nitrogen, CO_2, water vapor, and unreacted oxygen) is separated and depressurized through a backpressure regulator. The liquid and any inorganic salts formed at supercritical conditions, are depressurized through capillary tubes connected to on/off valves. The valves are actuated by a process controller that maintains the level in the gas/solid-liquid separator. All parts of the system that reach temperatures exceeding 70 °C are thermally insulated to minimize heat losses. The SCWO system uses a programmable logic controller (PLC) from Allen-Bradley (Rockwell Automation, Milwaukee, WI, USA) and a custom code to monitor and control the proper operation of the system. The prototype also includes a number of safety features such as rupture disks and alarms.

The SCWO tests reported herein were conducted during two separate runs, the two AIX in one day and the GAC on another day. During these runs, the system went through its routine startup protocol; the system was heated using the furnace (see Fig. 1) and then brought to a steady state with a starter feedstock (10% v/v isopropyl alcohol in water). At this stage, a control sample (thereafter called effluent blank) was collected to establish baseline effluent concentrations of trace PFAS that exists from previous PFAS tests using the SCWO system. After the warmup, the GAC or AIX slurry was introduced as feedstock replacing the starter materials (see Table 1 for waste feedstock compositions). After a short transient phase, a new steady state (as determined by stable temperatures and run parameters) was established and grab samples of the liquid effluent were collected for analysis, and operation data from the PLC were averaged for data analysis. The important run parameters are listed in Table 2. Note that for pollutant elimination calculations, the feedstock flow rates must be used rather than the concentrations. This is to account for the dilution brought by the additional water supply as mentioned above.

2.4. PFAS analysis and other analytical methods

All PFAS samples were analyzed by a commercial analytical laboratory, SGS AXYS, Sidney British Columbia for 40 PFAS compounds using Draft EPA Method 1633 (EPA, 2021) as described in SI S2. Liquid effluent from SCWO processes often contains various amounts of suspended minerals (depending on the waste being treated). In the current investigations, the concentration of minerals formed was low, and thus minerals formed could not be separated from the liquid effluent (e.g., for separate analysis of PFAS in those minerals). However, since analytical method USEPA Method 1633 uses a whole bottle extraction technique to analyze PFAS in the collected samples, it is expected that PFAS in any suspended mineral were extracted for PFAS analysis and included in the results for the liquid effluent.

Table 2
Run conditions (averages) and characteristics.

Feedstock	WAFB AIX	Miramar AFB AIX	WAFB GAC
Volume of slurry treated (L)	10	10	10
Mass of media treated (g_{dry})	800	800	1000
Feedstock flowrate (L/h)	5.8	5.8	5.5
Water flowrate (L/h)	28.8	28.8	27.4
Air flowrate (Nm^3/h)	12.3 kg/h	12.0 kg/h	11.9 kg/h
Max. reactor temperature ± stdev. (°C)	571 ± 25	550 ± 15	570 ± 8
Reaction time* (s)	7.0	7.6	7.0

* Reaction time is calculated using air and water densities at the reactor conditions (temperature and pressure)

The calorific value of the GAC and ion exchange resins were determined in duplicate samples using a Parr 6100 (Parr Instrument Company, Moline, IL) automated bomb calorimeter following the manufacturer’s instructions.

2.5. Data and performance reporting

PFAS results in the solid matrices are reported in units of nanograms per kilogram (ng/kg). In the liquid effluent, results are reported in nanograms per liter (ng/L)/ Results are reported both as measured and after subtracting the PFAS concentration measured in the steady-state blank. PFAS elimination was calculated on a rate basis (Eqs. 1–3):

$$\text{Rate-based PFAS Elimination} = \frac{\text{PFAS feed rate} - \text{PFAS effluent rate}}{\text{PFAS feed rate}} \times 100\% \tag{1}$$

where

$$\text{PFAS feed rate}\left(\frac{\text{ng}}{\text{hr}}\right) = \text{PFAS conc.in solid}\left(\frac{\text{ng}}{\text{kg}}\right) \times \text{solid conc.in feed}\left(\frac{\text{kg}}{\text{L}}\right) \times \text{feed rate}\left(\frac{\text{L}}{\text{hr}}\right) \tag{2}$$

$$\text{PFAS effluent rate}\left(\frac{\text{ng}}{\text{hr}}\right) = \text{PFAS conc.in effluent}\left(\frac{\text{ng}}{\text{L}}\right) \times \text{effluent flow rate}\left(\frac{\text{L}}{\text{hr}}\right) \tag{3}$$

At the beginning and between SCWO runs for spent media feedstocks, blank samples (10% v/v isopropyl alcohol in water) were run through SCWO system and the SCWO effluents from these blank samples were collected and analyzed for PFAS (blank effluents). PFAS detections in the blank effluents suggest that some residual PFAS may remain in the SCWO reactors from the previous runs and carried over into the blank sample, then subsequently treated and released into the blank effluents. Thus, results are presented two-ways: (1) as absolute concentrations and rate-based elimination (i.e., without correction for blanks and (2) after correction for the blanks subtracting the respective concentrations of the blanks from the measured effluent concentration and calculating corrected rate-based elimination.

3. Results

3.1. COD elimination

The COD of the effluent of the SCWO system during operation was 49 mg/L (n = 3 grab samples during treatment of each solid matrix), 40 mg/L (n = 2 samples each) and 38.3 mg/L for the treatment of WAFB GAC, WAFB AIX and Miramar AIX, respectively corresponding to a minimum of 99.97% COD elimination. The high percentage elimination of COD is consistent with a numerous previous run of SCWO system for treating a variety of wastes streams such as wastewater sludges, food waste slurries, landfill leachates and others (Deshusses, unpublished results).

3.2. Spent GAC feedstock from WAFB

Table 3 presents the results of laboratory analysis of the spent GAC feedstock. Spent GAC contained 14 PFAS out of the 40 PFAS analyzed with a total PFAS concentration of 214,400 ng/kg and individual PFAS concentrations ranging from 289 ng/kg for PFNA to 83,940 ng/kg for

Table 3
PFAS in WAFB spent GAC feedstock and SCWO treated effluent. The corrected effluent is the effluent concentration after subtraction of the effluent blank concentration. PFAS concentrations were rounded to 4 significant digits. ND = non-detect.

WAFB Spent GAC	Feedstock ng/kg	Effluent ng/L	Rate-based elimination %	Effluent blank ng/L	Corrected effluent ng/L	Rate-based (corrected) elimination %
PFBA	15,040	42.6	78.9%	23.4	19.2	90.5%
PFPEA	25,200	25.7	92.4%	13.6	12.1	96.4%
PFHXA	33,510	28.7	93.6%	12.9	15.8	96.5%
PFHPA	3364	1.14	97.5%	0.604	0.536	98.8%
PFOA	13,060	1.1	99.4%	0.913	0.187	99.9%
PFNA	288.8	ND	100.0%	ND	ND	100.0%
PFBS	689.6	41.3	-347%	12.9	28.4	-207.1%
PFPES	514.4	5.09	26.2%	1.76	3.33	51.7%
PFHXS	17,740	50.9	78.6%	14.1	36.8	84.5%
PFHPS	928.4	4.62	62.9%	2.18	2.44	80.4%
PFOS	83,940	243	78.4%	147	96	91.5%
6:2 FTS	12,610	98.7	41.6%	69.1	29.6	82.5%
8:2 FTS	4321	ND	100.0%	ND	ND	100.0%
PFOSA	3165	5.07	88.1%	7.42	0[#]	100.0%
Total PFCA	90,450	99.24	91.8%	51.42	47.82	96.1%
Total PFSA	103,800	344.9	75.2%	177.9	167.0	88.0%
Precursors	20,100	103.8	61.5%	76.52	29.6	89.0%
Short chain (C<6)	41,440	114.7	79.4%	51.66	63.03	88.7%
Total	**214,400**	**547.9**	**80.9%**	**305.9**	**244.4**	**91.5%**
Total PFCA	42%	18%				
Total PFSA	48%	63%				
Precursors	9%	19%				
Short chain	19%	21%				

[#] A zero value was assigned since effluent blank concentration was greater than effluent during GAC treatment.

PFOS. PFCAs (C4-C9, 90,450 ng/kg), PFSAs (C4-C8, 103,800 ng/kg) and precursors (6:2FTS, 8:2FTS and PFOSA; 20,100 ng/kg) were 42%, 48% and 9% of the total PFAS concentration, respectively. The shorter chain PFAS (C<6) including PFBA, PFPeA, PFBS, PFPeS were 19% of total PFAS concentration (Table 3). When comparing the laboratory solids analysis of 83,940 ng/kg of PFOS and 13,060 ng/kg of PFOA to the mass loading determined using the GAC treatment system operation data, which showed 60 mg/kg and 11 mg/kg for PFOS and PFOA, respectively, it is apparent that the laboratory reported data are several orders of magnitude lower than the mass loading on the spent GAC calculated from water treatment data. The discrepancies (about 800-fold lower concentration than expected) could be due to (1) the collection point of the GAC sample, which was at the bottom section of the vessel, and not representative of average PFAS mass loading, or (2) unknown causes of PFAS recovery from the spent GAC during laboratory analysis. For example, it has been suggested that fines generated during GAC extraction can be lost, although it is unlikely that such phenomenon would account for almost 3 orders of magnitude difference. This discrepancy has not been resolved.

Based on the laboratory results presented in Table 3, the elimination efficiencies for PFCAs, PFSA, precursors, short chain PFAS and total PFAS were 91.8%, 75.2%, 61.5%, 79.4% and 80.9%, respectively. The blank sample collected from the effluent prior to introducing the spent GAC exhibited a 306 ng/L PFAS residual from previous investigations,

Table 4
PFAS in WAFB spent AIX feedstock and SCWO treated effluent. The corrected effluent is the effluent concentration after subtraction of the effluent blank. PFAS concentrations were rounded to 4 significant digits. ND = non-detect.

WAFB Spent AIX	WAFB Spent AIX Feedstock ng/kg	Effluent ng/L	Rate-based elimination %	Effluent blank ng/L	Corrected effluent ng/L	Rate-based (corrected) elimination %
PFBA	2646	ND	100.0%	ND	ND	100.0%
PFPeA	17,840	ND	100.0%	ND	ND	100.0%
PFHxA	87,830	1.04	99.9%	1.02	0.02	100.0%
PFHpA	31,156	ND	100.0%	ND	ND	100.0%
PFOA	120,100	ND	100.0%	ND	ND	100.0%
PFNA	686.5	ND	100.0%	ND	ND	100.0%
PFBS	27,150	0.47	99.9%	ND	0.47	99.9%
PFPeS	36,740	0.5	99.9%	ND	0.5	99.9%
PFHxS	555,900	6.45	99.9%	0.874	5.576	99.9%
PFHpS	50,690	0.71	99.9%	ND	0.71	99.9%
PFOS	421,800	62.7	98.9%	97.8	0[#]	100.0%
6:2 FTS	33,226	1.39	99.7%	ND	1.39	99.7%
PFOSA	182.4	3.74[*]	-52.9%	4.97	0	100.0%
Total PFCA	260,200	1.04	100.0%	1.02	0.02	100.0%
Total PFSA	1,092,000	70.83	99.5%	98.67	7.256	100.0%
Precursors	33,260	5.13	98.9%	ND	1.39	99.7%
Short chain (C<6)	84,380	0.97	99.9%	0	0.97	99.9%
Total	**1,386,000**	**77**	**99.6%**	**104.7**	**8.666**	**100.0%**
Total PFCA	19%	1%				
Total PFSA	79%	92%				
Precursors	2%	7%				
Short chain	6%	1%				

[*] PFOSA in the feedstock was not detected. The value in the table is method reporting limit and is used to calculate rate-based PFAS elimination %.
[#] A zero value was assigned since effluent blank concentration was greater than effluent during AIX treatment.

containing 147 ng/L of PFOS. After correcting for the blank, the calculated elimination efficiencies increased to 96.1%, 88.0%, 89.0%, 88.7%, 91.5%, respectively. These values suggest that the PFAS elimination was more effective for PFCAs, than PFSAs or precursors. The corrected elimination efficiencies were all greater than 80% for the detected PFAS compounds, except PFBS, and PFPeS. The effluent concentrations of two compounds (PFNA and 8:2 FTS) were below the detection limits while these two compounds have lower concentrations in the spent GAC comparing to PFOS and PFOA. Regarding PFBS, although its concentration in the effluent was low (41 ng/L or 28.4 ng/L after correction), rate-based calculation shows a 2–3.5 fold formation of this compound. This is contrary to earlier PFAS elimination data with the same pilot [10] that showed no significant formation of PFAS. A possible explanation is the low concentration of PFBS in the influent slurry (equivalent to 55 ng/L, calculated by multiplying the PFBS mass loading on the GAC media by the GAC solids concentration in the slurry) and elimination data being disproportionally affected by the dilution factor of the combined effluent and/or baseline contamination downstream of the reactor (Table 3, blank concentration). A similar phenomenon may have affected PFPeS because of its low concentration in the influent. Generally, the PFCA and PFSA elimination appeared to increase with the carbon chain lengths. However, while many destruction technologies documented lower or no destruction efficiency for shorter chain PFAS, SCWO demonstrated elimination of 88.7% (after correcting for the blank) for shorter chain PFAS.

3.3. *Spent AIX feedstock from WAFB*

Table 4 presents the laboratory results of the spent AIX analyses. Spent AIX feedstock contained 12 out of 40 PFAS compounds analyzed using Draft USEPA Method 1633 with a total PFAS concentration of 1,386,000 ng/kg. This is much higher when comparing to the total PFAS concentration of 214,400 ng/kg found in the spent GAC feedstock sample. PFAS concentrations ranged from 686.5 ng/kg for PFNA to 555,900 ng/kg for PFHxS. PFCAs (C4-C9, 260,200 ng/kg), PFSAs (C4-C8, 1,092,000 ng/kg) and precursors (6:2FTS and PFOSA; 33,260 ng/kg) were 19%, 79% and 2% of the total PFAS concentration. The shorter chain PFAS (C<6) including PFBA, PFPeA, PFBS, PFPeS were 6% of total PFAS concentration. Similar to the GAC observation, when comparing the laboratory reported data of 421,800 ng/kg of PFOS and 120,100 ng/kg of PFOA to the mass loading calculations (13,864 mg/kg and 1659 mg/kg for PFOS and PFOA, respectively) of the AIX treatment system, a large discrepancy was observed. Mass loading calculations yielded concentrations 33,000 and 138,000 times larger than analyzed, for PFOS and PFOA, respectively. The causes for this discrepancy are probably the same as those discussed for the GAC analysis; they deserve further investigation.

Table 4 also reports the PFAS concentrations in the treated effluent water and in the effluent blank and the rate-based elimination efficiencies. PFBA, PFPeA, PFHpA, PFOA and PFNA were not detected in the effluent, while only 4 compounds were detected in the blank (notably 97.8 ng/L PFOS). PFOS and PFOSA concentrations in the blank run were greater than the concentrations in the effluent during the AIX treatment phase. When using rate-based elimination calculation without correcting with blank run results, the results suggest PFOSA was produced in the SCWO treatment. When PFOSA was assumed to be zero, the elimination % becomes 100%. The PFOSA data suggest more cleaning (blank) runs are needed between treatment runs to reduce PFAS carryover from the equipment. The rate-based elimination efficiencies for PFCAs, PFSA, precursors, short chain PFAS and total PFAS were 99.97%, 99.5%, 98.9%, 99.9% and 99.6%, respectively, or 100%, 100%, 99.7%, and 99.9% respectively, if correcting for the blank. This indicates that very efficient treatment was achieved. Shorter chain PFAS were almost completely destroyed. The overall PFAS elimination was better for spent AIX than spent GAC despite the PFAS concentrations on the AIX media being higher than on the GAC media. Examination of the run data for the GAC and AIX treatment runs showed very similar temperature patterns. There are two possible reasons for the better performance when treating spent AIX versus spent GAC: (1) the GAC slurry included additional co-fuel which increased the COD of the influent slurry to a value greater than that of the AIX slurry (285 g/L COD for the GAC-co-fuel slurry vs. 172 g/L for the AIX slurry). As a result, the oxygen excess was slightly greater when treating AIX, which could have improved treatment performance and (2) different rates of PFAS oxidation could be obtained for PFAS adsorbed on GAC and immobilized on AIX, due to the different nature of the binding to the solid matrix.

Table 5
PFAS in Miramar spent AIX feedstock and SCWO treated effluent. The corrected effluent is the effluent concentration after subtraction of the effluent blank concentration. PFAS concentrations were rounded to 4 significant digits. ND = non-detect.

Miramar spent AIX	Miramar Spent AIX Feedstock	Effluent	Rate-based elimination	Effluent blank	Corrected effluent	Rate-based (corrected) elimination
	ng/kg	ng/L	%	ng/L	ng/L	%
PFBA	85,910	7.82	99.5%	ND	7.82	99.5%
PFPEA	102,200	4.74	99.7%	ND	4.74	99.7%
PFHXA	33,980	7.08	98.8%	1.02	6.06	98.9%
PFHPA	1642	1.01	96.3%	ND	1.01	96.3%
PFOA	7516	1.82	98.6%	ND	1.82	98.6%
PFBS	41,280	9.55	98.6%	ND	9.55	98.6%
PFPES	4724	10.8	86.3%	ND	10.8	86.3%
PFHXS	33,660	198	64.8%	0.874	197.1	65.0%
PFHPS	8056	39.5	70.7%	ND	39.5	70.7%
PFOS	491,300	501	93.9%	97.8	403.2	95.1%
PFNS	180.5	ND	100.0%	ND	ND	100.0%
6:2 FTS	77,440	ND	100.0%	ND	ND	100.0%
8:2 FTS	3876	ND	100.0%	ND	ND	100.0%
PFOSA	3493	14.7	74.8%	4.97	9.73	83.3%
Total PFCA	231,200.	22.47	99.4%	1.02	21.45	99.4%
Total PFSA	579,200	758.9	92.2%	98.67	660.2	93.2%
Precursors	84,810	14.70	99.0%	4.97	9.73	99.3%
Short chain (C<6)	234,100	32.91	99.2%	0	32.91	99.2%
Total	**895,200**	**796.0**	94.7%	**104.7**	**691.4**	95.4%
Total PFCA	26%	3%				
Total PFSA	65%	95%				
Precursors	9%	2%				
Short chain	26%	4%				

3.4. Spent AIX from Miramar

Table 5 presents the laboratory results for the spent AIX from Miramar. Spent AIX contained 14 PFAS compounds out of 40 PFAS compounds analyzed using USEPA draft Method 1633 with a total PFAS concentration of 895,200 ng/kg, compared to the total PFAS concentration of 214,000 ng/kg in the spent GAC and 1,386,000 ng/kg in the spent AIX from Wurtsmith AFB. PFAS concentrations ranged from 180.5 ng/kg for PFNS to 491,300 ng/kg for PFOS. PFCAs (C4-C8, 231,200 ng/kg), PFSAs (C4-C9, 579,200 ng/kg) and precursors (6:2FTS, 8:2FTS and PFOSA; 84,810 ng/kg) were 26%, 65% and 9%, respectively, of the total PFAS concentration, which was dominated by PFSAs and PFOS. The shorter chain PFAS (C<6), including PFBA, PFPeA, PFBS, PFPeS, were 26% of total PFAS concentration considering the highest % compared to the feedstock samples collected from Wurtsmith AFB. As mentioned in the methods section, comparison of the PFAS concentration on the spent AIX with the PFAS mass loading could not be conducted for this medium because of incomplete mass loading data.

After SCWO treatment, PFAS concentrations were greatly reduced in the effluent water. As presented in Table 5, the elimination efficiencies for PFCAs, PFSA, precursors, short-chain PFAS and total PFAS were 99.4%, 92.2%, 98.96%, 99.2% and 94.7%, respectively, or slightly greater after correcting the effluent concentrations for the blank. The overall PFAS elimination percentage was greater compared to the treatment of the spent GAC from WAFB but slightly less complete when comparing to the spent AIX from WAFB.

4. Discussion

This study presents the results from SCWO treatment of three different PFAS-laden filtration media taken from PFAS removal systems for treating contaminated groundwater and high-strength wastewater. To our knowledge, this is the first study demonstrating the ability of SCWO to destroy both the spent media and the PFAS adsorbed or immobilized onto it. The PFAS mass in the spent media was calculated based on PFAS treatment system operation and monitoring data. The PFAS mass in the spent media was also examined using the modified PFAS recovery method described in Draft USEPA Method 1633. While PFAS data quality met the criteria, PFAS mass loading from laboratory analysis (Tables 3–5) was orders of magnitude lower than the mass loading determined using the treatment system operation data (described in the Supporting Information). Whether all PFAS in the spent media were fully recovered was not examined, however, if incomplete PFAS recovery occurred, it would have resulted in correspondingly underestimated PFAS elimination efficiencies. The discrepancy between analysis and mass loading data can be a result of limited sample volume (20 L) collected from the bottom section of the lead vessels. PFAS mass distribution within the lead vessel was not characterized, the PFAS mass in the vessel bottom section can be significantly lower than the average PFAS mass loading. It is also difficult to validate whether PFAS adsorbed on spent media were fully recovered for PFAS analysis and if the PFAS recovery rates were comparable between different PFAS treatment systems (GAC or AIX) and different media products. These observations highlight the need for further documentation of the degree of heterogeneity of PFAS loading in spent media and PFAS recovery during analysis and better sampling methods to gather a representative composite sample from the entire length of the vessel.

The feedstock sample data showed abundant shorter-chain PFAAs on both GAC and AIX with shorter-chain PFAS contribution to total PFAS concentrations in the WAFB spent GAC, WAFB spent AIX and Miramar spent AIX at 19%, 6% and 26%, respectively. Both WAFB and Miramar have legacy AFFF as sources of PFAS contamination. The feedstock results confirmed the total PFSA concentrations were higher than PFCA concentrations and the ratios of PFCA:PFSA for WAFB spent GAC (42:48), WAFB spent AIX (19:79), and Miramar spent AIX (26:65). After SCWO treatment, the ratios changed to 18:63, 1:92, 3:95, respectively (Tables 3–5) due to the greater elimination of PFCAs.

PFAS elimination by SCWO treatment was calculated based on the PFAS concentrations, solid density, and flow rates of feeding feedstock, and the effluent. Corrections were then made for a blank sample collected from the effluent prior to introducing the GAC or AIX feedstocks into the SCWO system. Fig. 2 compares the rate-based PFAS elimination efficiencies among the three feedstock samples. Overall, PFCAs were better removed than PFSAs and precursors and long-chain PFAS were better removed than short chain ones. These results are consistent with the recent observations of Li et al. [12], Austin et al. [1] and Scheitlin et al. [23] during treatment of PFAS in solutions and in lab-scale SCWO systems. It has also been suggested that the carboxyl moiety in PFAS is more reactive than the sulfonated one [29]. For the three spent media, greater than 80% of total PFAS elimination was achieved (or greater than 90% if corrected by the blank). The elimination rate when treating spent GAC was lower with 62–89% of total precursors removed during the treatment. Possibly, conditions will need to be optimized to achieve greater elimination of precursors. Total PFAS elimination was 99.6–99.95% for WAFB spent AIX. PFOSA was not detected in the feedstock (albeit at a high limit of detection) but was detected in the effluent at very low level (3.74 ng/L) suggesting it may

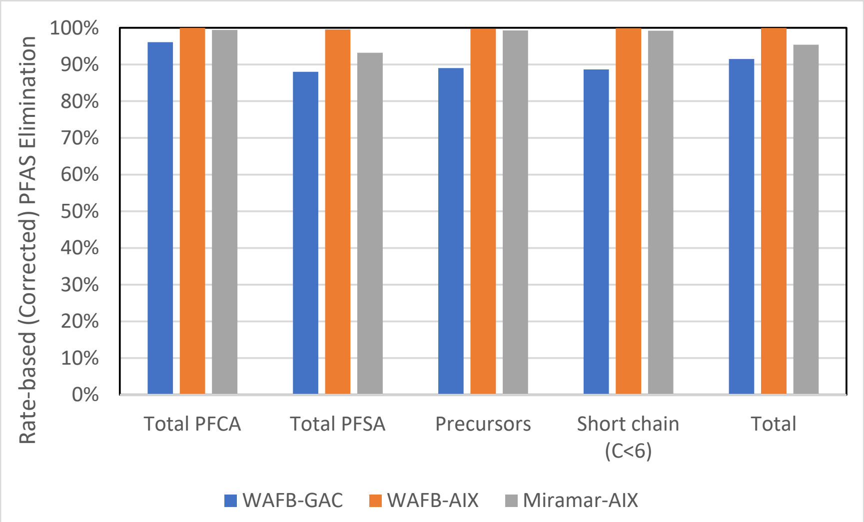

Fig. 2. Rate-based elimination of PFAS in spent GAC and AIX using SCWO treatment.

be in the range of analytical uncertainties or similar to the blank. Further, since influent samples are subject to much greater dilution than effluent samples prior to PFAS analysis, erroneous reporting of formation of specific PFAS compounds can occur due to very different detection limits in the influent and effluent samples. This was reported for PFOSA for the WAFB spent AIX.

The PFAS elimination efficiencies were better for PFAS on spent AIX than GAC. AIX generally contains a polymeric matrix to which different functional groups are attached. The organic polymeric matrix is expected susceptible to destruction via SCWO process comparing to the GAC as a form of carbon. However, there is no specific data-driven explanation on the performance differences between the two types of media. Thus, the kinetics of PFAS elimination in spent media deserves detailed investigations. Overall, SCWO effectively eliminated PFAS in the spent media, and all organics were effectively mineralized during the SCWO treatment into carbon dioxide and water.

SCWO performance and PFAS elimination efficiency can be further improved after by optimizing the performance of the SCWO system. Specifically, temperature and reaction time have a major impact on the elimination of PFAS based on prior experience. The pH, oxidant (oxygen) concentration, and presence of co-oxidized contaminants may also play a role. The large scale, continuous SCWO system used for this study was originally designed for treating significantly less recalcitrant wastes and it was not redesigned or configured for PFAS elimination applications. The underlying SCWO technology (AirSCWO™) is being scaled up for commercial applications at higher throughputs and optimized for elimination of highly recalcitrant organic compounds like PFAS. The resulting system is expected to mineralize PFAS-laden wastes more effectively, such as spent GAC and AIX from PFAS water treatment systems. We note here that the mass balance of fluoride and destruction of PFAS have not been evaluated in this study. This is because of limits of detection and precision of measuring inorganic fluoride in the effluent stream. Despite the fact that the PFAS concentration on the solid matrices tested was up to 1,386,000 ng/kg, complete mineralization would only increase the fluoride concentration by about 0.06 mg/L which was not measurable accurately alongside the fluoride background present in the water. There is however strong evidence that SCWO can fully mineralize organofluorine compounds to inorganic fluoride, especially when operated at temperature in the range of 550–600 °C (Austin et al., 2021; [12]; Deshusses, unpublished). Even so, more studies and research are needed to collect fluoride balance data from SCWO during treatment of solid matrices such as GAC and AIX to confirm PFAS destruction and complete mineralization.

5. Conclusions

PFAS have been detected in water, soil, air, biota, and food and are used in everyday consumer goods that we come into contact with regularly. Scientific research has shown that exposure to PFAS may result in adverse health impacts. US and international regulatory agencies are developing regulations that will require the removal of PFAS from drinking water and contaminated source waters. Thus, far more PFAS treatment systems will be installed, the majority of which will most likely use GAC and AIX, to meet the stringent criteria set for PFAS discharges or PFAS in drinking water. GAC and AIX are the most proven and demonstrated technologies, however, they are separation technologies, removing the PFAS from the aqueous phase and concentrating them on a solid matrix that requires further processing to meet PFAS waste management requirements. Incineration and disposal of spent media are not sustainable; furthermore, the capacity of such waste management units can decrease exponentially with growing regulations. Permanent, effective, and sustainable destruction technologies like SCWO are in demand for eliminating PFAS forever.

In this paper, we demonstrated that SCWO is an efficient technology for the destruction of PFAS-laden spent GAC and AIX. However, this study also revealed the need for further research to confirm the PFAS mass loading on the spent media and a longer-term study for establishing a full mass balance of PFAS and fluorine in SCWO system outputs. In parallel, full-scale commercial SCWO systems, able to demonstrate effective treatment of the entire volume of spent media from a filtration vessel, are required to reduce uncertainties when only treating a discrete sample. The full-scale treatment will also validate the findings in actual operation over extended periods of time. Finally, energy consumption, air quality, carbon footprint, and a life cycle assessment should be conducted to compare the SCWO technology to current practices such as incineration, deep well injection, and landfilling.

Environmental Implication

Some PFAS are proven toxic and hazardous to the human health and environment. The number of treatment systems using filtration media (such as GAC and AIX) to remove PFAS from water sources is increasing globally. The systems are effective a removing PFAS from water, but they generate PFAS-laden spent media requiring further treatment and disposal. PFAS destruction of such wastes is needed to prevent PFAS from reentering the environment. Most PFAS destruction technologies under development aim to destroy PFAS in the liquid wastes. There are very few alternatives for contaminated solid wastes such as spent media. This study demonstrates the use of SCWO technology to mineralize spent media and destroy PFAS adsorbed or bound onto the media.

CRediT authorship contribution statement

Sheau-Yun Dora Chiang: Supervision, Conceptualization, Methodology, Validation, Investigation, Writing-original Draft, Review, Editing, Project Administration, Funding Acquisition. Matthew Saba, Macon Leighton, David Ballenghien: Investigation, Data Curation. Douglas Hatler: Methodology, Writing Review & Editing. Justin Gal: Methodology, Data Curation, Writing Review & Editing. Marc A. Deshusses: Supervision, Conceptualization, Methodology, Validation, Investigation, Writing-original Draft, Review, Editing, Project Administration, Funding Acquisition.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests. Matthew Saba, Macon Leighton, David Ballenghien, Douglas Hatler, and Marc Deshusses are shareholders of 374Water Inc., which is commercializing supercritical water oxidation systems for PFAS destruction.

Data Availability

Data will be made available on request.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jhazmat.2023.132264.

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