

March 31, 2024

James C. Kenny
Cabinet Secretary
New Mexico Department of Environment
Harold Runnels Building
1190 St. Francis Dr. Suite N4050
Sante Fe, NM 87505

Via email to NMED's contractor, Janet.Carpenter@erg.com.

Dear James C. Kenny:

On behalf of Renewable Technology Corporation (RTC), we thank you for your commitment to making environmental changes for the strategic water supply of New Mexico. At RTC, our mission is to use advanced technologies to efficiently, sustainably, and safely convert non-potable water sources and waste solids to clean water and usable energy for the wellbeing of all communities. We are dedicated to achieving this transformation with a strong commitment to innovation, efficiency, and environmental stewardship. RTC recognizes the need for a strategic water supply and continues to develop technology to address core requirements. Our technology provides comprehensive turnkey solutions that allow industries and communities to focus their time and energy on business.

One of our foremost concerns for the water and wastewater industries is that of cybersecurity. New Mexico has unique requirements for strategic water supplies that have many cybersecurity similarities with existing Water and Waste Systems (WWS). Many of the rural locations lack modern security systems and some rural locations lack cell phone coverage, making communications difficult.

In the following pages, we briefly outline issues and solutions that have long histories of success. RTC is suggesting that these technologies—already are in place in other industries—would benefit all water infrastructure in the State of New Mexico in oil fields, wastewater treatment plants and agricultural water supplies. These state-of-the-art technologies are both affordable and ready to be deployed in a matter of weeks.

RTC also specializes in the development of funding sources from Federal, State and local governments to coordinate and maximize the necessary integration of resources for complete solutions.

Cybersecurity, Supervisory Control and Data Acquisition (SCADA), and 5G should be implemented in the first phases of water infrastructure projects to ensure that design and construction phases are properly protected and integrated. We will submit additional responses via additional emails for water filtration and contaminant destruction including PFAS.



We look forward to our continued commitment to New Mexico in these upcoming opportunities to strengthen the Strategic Water Supply for future generations.

Kind regards,

Charlene Thomas

Charlene Rainey Thomas
CEO Renewable Technology Corporation
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Cybersecurity for Strategic Water Supply

By Renewable Technology Corporation (RTC)

March 31, 2024

Response to Request for Information for Strategic Water Supply issued by New Mexico Environment Department issued on Jan 19, 2024

The Urgent Cybersecurity Threat: cyberattacks are targeting Water and Wastewater Systems (WWS) nationwide. This is endangering clean water access, water applications, and imposing significant costs on affected communities and industries. The U.S. Federal Government continues to take action, sends out alerts/notices, and has classified WWS as national critical functions.

- The disruption or corruption of WWS operations would “have a debilitating effect on security, national economic security, national public health or safety, or any combination thereof.”
- US agencies have issued and continue to issue many alerts, warnings, and advisories.
 - EPA, FBI, CISA, NSA
- Attacks are on-going against WWS IT and Operational Technology (OT) networks — launched by both known and unknown threat actors.
- Attacks by adversaries impact the WWT Supervisory Control and Data Acquisition (SCADA) systems.
 - Adversaries could gain control of critical processing equipment, chemical doses, flow rates, and vital functions.
 - Endanger public health, service interruptions, malfunctions, and life-threatening contamination events.

The Cybersecurity Threat Overview - Tactics, Techniques, and Procedures (TTP)

- Adversaries can attack and go undetected from anywhere in the world into cyberspace.
- Command-and-control linkages between cyberspace and physical systems vulnerabilities can enable remote manipulation or sabotage of WWS and infrastructure.
- Difficulties of reducing vulnerabilities, increasing security coverage, and sustaining resiliency in complex cyber networks.
- Spearphishing personnel that lack cybersecurity training are used to deliver malicious payloads, including ransomware to the WWS.
- Exploitation of unsupported or outdated operating systems and software where WWS prioritize physical assets (e.g. pipes) rather than IT/OT software.
- Exploitation of control system, SCADA, and IoT (Internet of Things) devices with vulnerable firmware versions and unsecured, unvalidated software components.

Real-World Example Threats

- Iranian Government-affiliated actors exploit default manufacturer passwords in operational WWS.
- People's Republic of China-sponsored cyber group Volt Typhoon compromises IT systems of critical infrastructure for covertly engineered threat
- Adversaries attacked Nevada-based WWS facility with unknown ransomware directly to SCADA monitoring
- Adversaries attacked Maine-based WWS facility SCADA system resorting to manual processes and escalating operator burden
- Adversaries attacked California-based WWS facility SCADA system that displayed a taunting ransomware message.

WWS Recommended Cybersecurity Mitigations

The FBI, CISA, EPA, and NSA recommend WWS and Department of Defense facilities in the US and abroad must use a risk-informed analysis for mitigations that prevent, detect, and respond to cyber threats.

WWS Monitoring for Mitigations

- Authorized personnel and monitoring detection software must check for suspicious activities and indicators with the control system SCADA, IoT, and reporting systems.
- Includes inability to access SCADA devices, unfamiliar data and alerts, abnormal parameter values, access or attempts by unauthorized users for SCADA systems, access at unusual and unscheduled times, unexplained SCADA system restarts

WWS Remote Access Mitigations

- Require strong digital identity authentication such as multi-factor for all remote access and to critical systems and SCADA access points.
- Utilize a collection of best practices.
- Block-listing, logging all remote actions, enabling and automated regular audits, only expose necessary ports and enforce security, limited and least privilege access

WWS Network Mitigations

- Establish strong network segmentation between IT and OT networks to prevent cyber attackers from accessing OT systems through compromised IT networks.

Planning and Operational Mitigations

- Include all cyberattack impacts in the emergency response plan, covering loss or manipulation of data and control, and safety threats.
- Regularly review, test, and update the plan, incorporating third-party needs and testing failover procedures for alternate control systems

Safety System Mitigations

- Implement independent cyber-physical safety systems to physically prevent dangerous conditions if control systems are compromised.

- Used for preventing cyber adversaries from manipulating process or chemical levels to hazardous levels.

WWS Cybersecurity Product Solutions

Cybersecurity, WWS Private, Resilient, Wireless 5G Data Network by Wi5G, Industry 4.0 by Lempko Corporation

- Integrate SCADA and IoT monitoring, command-and-control, data flow
- Wireless regional encrypted, secured, and controlled access network
- Network-level authenticated devices communications and encryption
- 4G and 5G high-performance data communications
- Mobile Edge computing with no single point of failure

SCADA in Water and Wastewater Monitoring Operations by multiple Vendors

- SCADA systems enable system operators to control processes and access automated real-time data
- Centralized, live Digital Dashboards and views screens
- Enabled for efficient management of water treatment processes
- Automation to increase energy-efficiency, adjustments, and alerts for flow changes and processes
- Increased regulatory compliance and reporting

Cybersecurity for SCADA, Operator, and Personnel Identity and Authorizations by DGate Corp and eSentire Cybersecurity

- Passwordless, password-based secured Digital Identity, different multi-factor authentication
- Advanced Web3 digital identities and credential wallets for added authentication
- Zero Trust cybersecurity enforcement for sub-system SCADA accesses and privileges
- Full device and software process monitoring coverage – IT, OPS, SCADA, IoT
- Extended Detection and Response (XDR) Platform for analytics and automated responses
- Rapid Threat Detection with current industry knowledge-bases
- Real-Time Threat Intelligence
- SOC as a Service (SOCaaS) Model increasing cost-savings and shared knowledge-based

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RE: Vortex and Electronic Aeration Water Filtration

Dear James C. Kenny:

On behalf of Renewable Technology Corporation (RTC), we thank you for your commitment to making environmental changes for the strategic water supply of New Mexico. At RTC, our mission is to use advanced technologies to efficiently, sustainably, and safely convert non-potable water sources and waste solids to clean water and usable energy for the wellbeing of all communities. We are dedicated to achieving this transformation with a strong commitment to innovation, efficiency, and environmental stewardship.

RTC recognizes the need for a strategic water supply and continues to develop technology to address core requirements. Our technology provides comprehensive turnkey solutions that allow industries and communities to focus their time and energy on business.

RTC represents Desert Bio's water filtration including Vortex and Electric aeration built in New Mexico and currently running in Farmington among other locations. Desert Bio's system is transportable and has achieved a very high throughput making it ideal for the repurposing of oil brackish and produced water.

In the attachments, we share publications and results of Desert Bio's Vortex and Electro Aeration water filtration. These systems come in both fixed and transportable configurations making them easy to install and cost efficient.

RTC is suggesting that Desert Bio's Vortex and Electro Aeration would benefit all water infrastructure in the State of New Mexico in oil fields, wastewater treatment plants and agricultural water supplies. These state-of-the-art technologies are both affordable and ready to deliver clean water to New Mexico.

RTC also specializes in the development of funding sources from federal, state, and local governments to coordinate and maximize the necessary integration of resources for complete solutions.

We will submit additional responses via additional emails for water SCWO and cybersecurity.

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Summary of the technology:

The basis of the technology is the reduction of pollution in all types of waters. Scalable to any body of water or flow rate, the utility and its associated systems make use of electrical impulses to dissociate the H₂O molecule into micron-sized gas bubbles. These efficiently and long lived catalytically generated gases eliminate organic pollutants through a process known as electro-oxidation.

Deployed for continuous aeration of lakes, ponds, cooling towers, farming lagoons, septic tanks, and aquariums the utility increases dissolved oxygen content and naturally reduces bacteria, organic contaminants, phosphorus, and nitrite compounds with no potential chlorination disinfection by-products. The technology can be modified to inexpensively and chemically-free harvest algae while eliminating odor causing bacteria. It also can be adapted for use in mosquito abatement programs as this form of oxygen will destroy larvae without harming fish.

As a cylinder or reactor, that houses the electro-catalytic cells; pump driven reactors can be scaled up to process thousands of gallons of polluted water a minute on a limited footprint. Operational at low conductivity and without producing chlorine compounds, these reactors can be used as an add-on or optimization of municipal wastewater treatment, industrial recycling systems for reduction of organic chemical compounds, industrial and home grey water system optimization, fish-farm pollutant reduction and many other applications where chlorine disinfection is not possible.

In sea water, the technology as electro-chemistry can be adapted to ship's open-scrubber systems, ballast, and bilge remediation. Reactors create, through the addition of a saltwater slip stream, in-situ generated chlorine compounds for long term disinfection.

In recent testing it appears the technology transforms CO₂ in solution to other non-greenhouse compounds and raises the prospect of this tool for carbon exchange programs. This technology could redefine the role of water remediation as a method of lowering CO₂ in the atmosphere by conversion rather than transitory sequestration.

The earlier, now [patented](#), and licensed version, of a similar approach in oilfield water treatment is currently economically processing millions of gallons per day in FeS, H₂S and bacterial reduction through electro-chemistry or chlorination.

This new utility patent filing concerns itself with the geometry and metal composition that engender at low conductivity, electro-oxidation, or the generation of reactive oxygen species as disinfection agents.

The plates, while cast in China, are assembled here in our UL certified fabrication shop Downey CA. The plates have a long multi year life which more than makes up the Cap-Ex.



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When implementing water filtration for reuse, the most difficult challenge is the destruction of contaminants and the forever chemicals like PFAS. RTC recommends as one of our solutions General Atomics for their experience and EPA certification of PFAS 99% destruction.

In the attachments, we share publications and results of Super Critical Water Oxidation (SCWO) decades of technology research and development with success in multiple full-scale industries and military. SCWO destroys hazardous materials at a molecular level. These systems come in both fixed and transportable configurations making them easy to install and cost efficient.

RTC is suggesting that SCWO integrated with other technologies—already are in place in other industries—would benefit all water infrastructure in the State of New Mexico in oil fields, wastewater treatment plants and agricultural water supplies. These state-of-the-art technologies are both affordable and ready to deliver clean water to New Mexico.

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WASTE DESTRUCTION TECHNOLOGY

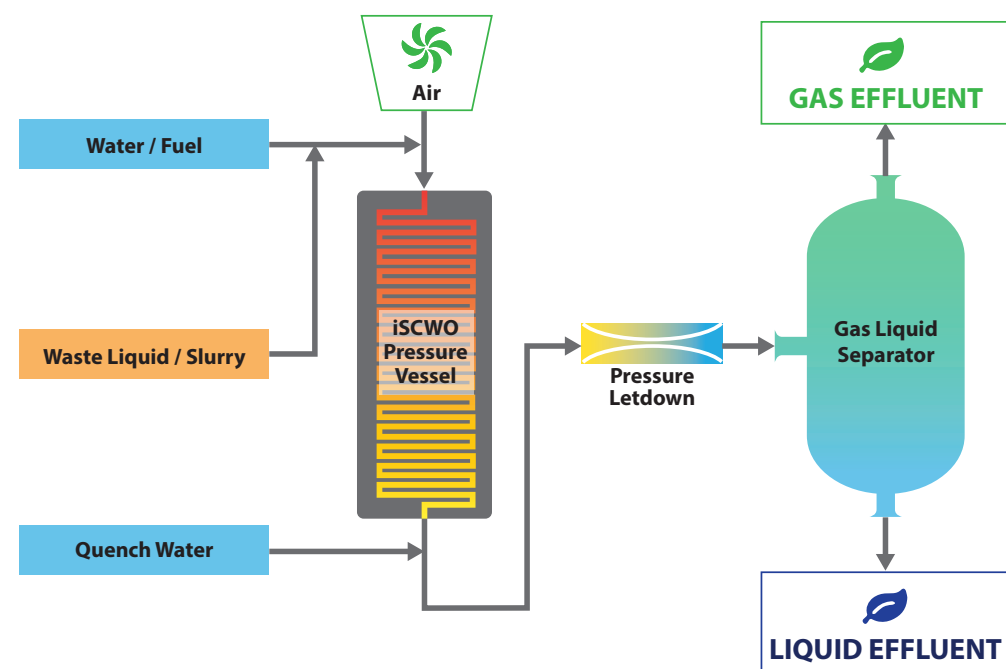
General Atomics Electromagnetic Systems' (GA-EMS) commercially available Industrial Supercritical Water Oxidation (iSCWO) technology has been successfully used to treat a broad range of hazardous and non-hazardous waste, with at least a 99.99% destruction removal efficiency. iSCWO provides a safe, environmentally sound solution to replace alternative technologies that have high emission rates, post treatment requirements, and substantial installation and operation costs.

In addition to offering a fixed site solution, GA-EMS offers the only flexible, transportable iSCWO system available today to facilitate "generate and destroy" on-site capabilities. The system consists of a compact equipment skid housed inside a shipping container to create a convenient, cost effective, mobile platform for waste destruction when and where it's needed.



Two Transportable iSCWO Systems

GA-EMS' iSCWO system uses compressed air rather than more expensive pure oxygen (O_2) in the treatment process for safe, easy, and low cost in-field maintenance. The iSCWO process makes most organic materials, oxidation reactants, and oxidation products miscible in water, allowing complete oxidation reactions to take place at a high rate. The result is the creation of CO_2 , water and salts, all of which can be released into the environment or reused for other industrial purposes without any post treatment. NO_x , SO_x , and particulate concentrations created are also at or below detection limits.



INDUSTRIAL SUPERCRITICAL WATER OXIDATION



- Safe, proven, and environmentally sound
- High throughput, low cost, and commercially available
- Excellent, effective destruction efficiency of energetics, organic compounds, chemical waste, pesticides, petrochemical waste, and fungicides
- Emissions much lower than incineration systems that require pollution abatement systems
- Transportable, skid-based system
- Low-cost confirmation tests for process and environmental regulatory permits

ISCWO HAS SUCCESSFULLY TREATED A WIDE RANGE OF CHEMICALS

Complex Feeds		
Activated carbon (spent)	Diesel fuel	Paint, paint sludges
Adhesives	E. coli	Paper
Aqueous Cleaning Solution	Endotoxin (pyrogen)	Paraffin oil
(hydrolyzed RDX, TNT, Tetryl, NG, NC)	Explosives/energetics/propellants	Pesticide manufacturing wastewater
AFFF	Photographic developer paste	Pharmaceutical waste
Antifreeze	Fermentation byproducts	Photographic developer solutions
Aroclor 1242	Flourinated organics	Polychlorotrifluoroethylene (PCTFE)
Aroclor 1254	Food Waste	PFAS/PFOA
Aroclor 1260	Fuel oil	Pig manure
Bacillus stearothermophilus	GB chemical agent (neat, hydrolyzed)	Propellants (hydrolyzed)
(heat resistant spores)	Gold mining waste with organics	Protein
Brake fluid	Gray water	Pulp/paper mill sludge
Bran cereal	Greases (mixed)	Sawdust
Bromated organics	Human waste	Sewage sludge (black water)
Caprolactam wastewater	Hydraulic fluid	Soil contaminated with organics
Casein	Industrial biosludge	Soybean plants
Cesium Chlorate	Ion exchange resins (styrene/divinyl benzene)	Sulfolobus acidocaldarius
Chlorinated plastics (shredded)	Kerosene	Timber/wood waste
Class 1.1 solid propellant	Lube oil (molybdenum disulfide oil)	Transformer oil
Class 1.3 AP-depleted solid propellant	Malaria antigen	Trimsol cutting oil
Coal	Motor oil	VX chemical agent (neat, hydrolyzed)
Coal waste	Mustard chemical agent (neat, hydrolyzed)	Waste oils (chlorinated and non-chlorinated)
Corn flakes	Navy shore-based wastes	Wheat straw
Corn oil	Olive oil	Wood fibers
Corn starch	Organic compounds with nuclear isotopes	Yeast
CS (Tear Gas)	Organic salts (complex mixtures)	
Inorganic Substances		
Aluminum hydroxide	Fluorides	Potassium hydroxide
Aluminum metal	Hydrochloric acid	Potassium sulfate
Aluminum oxide sodium	Hydrofluoric acid	Silica
Ammonia	Iron chloride	Sodium bicarbonate
Ammonium chloride	Iron oxide	Sodium carbonate
Ammonium nitrate (ANSOL)	Lead chloride	Sodium chloride
Ammonium nitrite	Lead sulfate	Sodium fluoride
Ammonium perchlorate	Lithium hydroxide	Sodium hydroxide
Ammonium sulfate	Lithium sulfate	Sodium nitrate
Ammonium sulfite	Magnesium nitrate	Sodium nitrite
Boric acid	Magnesium oxide	Sodium phosphate
Bromides	Magnesium phosphate	Sodium sulfate
Calcium carbonate	Magnesium sulfate	Sodium sulfite
Calcium chloride	Mercuric chloride	Sulfur, elemental
Calcium oxide	Molybdenum disulfide lube oil	Sulfuric acid
Calcium phosphate	Nitric acid	Titanium dioxide
Calcium sulfate	Phosphoric acid	Zinc chloride
Cerium chloride	Potassium bicarbonate	Zinc sulfate
Cesium chlorate	Potassium carbonate	
Copper chloride	Potassium chloride	

Organic Chemicals		
Acetate acid	4,4-Dichlorobiphenyl	Nitrobenzene
Acetone	Dichloroethylene	2-nitrophenol
Acetylsalicylic acid (aspirin)	Dichlorophenol	4-nitrophenol
Adumbran	Diethanolamine	Nitrotoluene
4[(2-Amino-3, 5-dibromophenyl)-methylamino] cyclohexanol	Dimethylformamide	Octachlorostyrene
Ammonium acetate	Dimethyl methyl phosphonate (DMMP)	Octadecanoic acid magnesium salt
Ammonium formate	Dimethyl sulfoxide	Paracetamol
Ammonium oxalate	4,6-dinitro-o-cresol	Pentachlorobenzene
Benzene	2,4-Dinitrophenol	Pentachlorobenzonitrile
Biphenyl	Dinitrotoluene	Pentachlorophenol
Butanol	Dioxane	Pentachloropyridine
Calcium acetate	1,4 Dioxane	Phenol
Carbon tetrachloride	Dioxin	Polychlorinated biphenyls (PCB)
Carboxylic acids	Dipyridamole	Polychlorotrifluoroethylene
Carboxymethyl cellulose	Diisopropyl ethanolamine	Sodium acetate
Cellulose	Diisopropyl ethylamine	Sodium formate
Cerium acetate	Ethanol	Sodium hexanoate
Chlorinated dibenzo-p-dioxins	Ethyl acetate	Sodium isethionate
6-chloro-2,3,4,5-tetrahydro-3-methyl-1H-3-benzazepine hydrochloride	Ethylene chlorohydrin	Sodium propionate
2-chlorobenzalmononitrile (CS)	Ethylenediamine tetraacetic acid	Sucrose
Chlorobenzene	Ethylene glycol	Surfactant
Chloroform	Fluorescein	Tetrachlorobenzene
2-Chlorophenol	Freon 22	Tetrachloroethylene
o-Chlorotoluene	Glycerol	Tegrpropylene H
Cobalt acetate	Hexachlorobenzene	Thiodiglycol
Corrosive solvent waste	Hexachlorocyclohexane	Toluene
m-Cresol	Hexachlorocyclopentadiene	Tributyl phosphate
Cresolate	Iron acetate	Trichlorobenzene
Cyanide	Isooctane	1,1,1-Trichloroethane
Cyclohexane	Isopropanol	1,1,2-Trichloroethane
DDT	Lead acetate	Trichloroethylene
Decachlorobiphenyl	Mercaptans	Trichlorophenol
Dextrose	Mercaptoethanol	Trifluoroacetic acid
Dibenzofurans	Methanol*	1,3,7-Trimethylxanthine
3,5-dibromo-N0cyclohexyl-N-methyltoluene-,	Methyl acetate	Unsymmetrical dimethyl hydrazine
2-diamine	Methyl cellosolve	Urea
Dibutyl phosphate	Methylene chloride	o-Xylene
Dichloroacetic acid	Methyl ethyl ketone	Zinc acetate
Dichloroanisole	Methylphosphonic acid (MPA)	
Dichlorobenzene	Monoethanolamine	

Validation of supercritical water oxidation to destroy perfluoroalkyl acids

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Funding information

Arcadis; General Atomics

Abstract

Some of the same unique physical and chemical properties that make per- and polyfluoroalkyl substances (PFAS) desirable for a wide range of commercial applications render them recalcitrant to many liquid treatment technologies. As developments in PFAS-related toxicological studies increasingly suggest potential adverse human health effects, our industry has made great progress in the past several years on concentrating PFAS into small volume waste streams via adsorption and separation mechanisms. Coupled with residual PFAS-containing commercial products that are being phased out, management of these concentrated waste streams presents an urgent need for the development and validation of destructive treatment technologies. Here, we field-validate supercritical water oxidation to treat a concentrated waste stream of 12 perfluoroalkyl acids (PFAAs) with liquid and gaseous analysis, adhering to the recent Other Test Method 45 for stack emission sampling from the United States Environmental Protection Agency (USEPA) and USEPA Method 537.1, with quality control and quality assurance protocols from the Department of Defense/Department of Energy Quality Systems Manual 5.3. Results generated suggest greater than 99.999% destruction and removal efficiency of these 12 PFAAs after two ~120-min continuous flow trials, with an overall defluorination percentage of approximately 62.6%.

1 | INTRODUCTION

Multiple reviews in the literature detail the various strengths and weaknesses of treatment technologies for per and polyfluoroalkyl substance (PFAS)-impacted matrices, and are either a general assessment of PFAS-relevant treatment technologies (Anderson et al., 2021; Garg et al., 2021; Mahinroosta & Senevirathna, 2020; Merino et al., 2016; Riegel & Sacher, 2020; Ross et al., 2019, 2018; Wanninayake, 2021) or a focus on a particular mechanism, such as adsorption (Boyer et al., 2021; Dixit et al., 2021; Gagliano et al., 2020;

Uriakhil et al., 2021; Zhang D. Q. et al., 2019) or various forms of destruction (Cui et al., 2020; Horst et al., 2020; Nzeribe et al., 2019; U.S. Environmental Protection Agency, USEPA, 2020a; Winchell et al., 2020; Zhang W. et al., 2019). Independent of the purpose of the review, a similar conclusion is drawn: an appropriate treatment strategy for PFAS-impacted waste includes multiple treatment technologies in series to concentrate PFAS into the smallest possible volume for energy-intensive destruction.

Over the past several years, our industry has benefited from PFAS-related optimization of commercially available adsorbents, such

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as activated carbon (Murray et al., 2019; M. Park et al., 2020; Son et al., 2020) and ion exchange resins (Dixit et al., 2020; Zeng et al., 2020), the application of developing adsorbents for PFAS removal (Kumarasamy et al., 2020; Stebel et al., 2019; L. Xiao et al., 2017; Yan et al., 2020; Zheng et al., 2020), and increased efficiency of PFAS separation from impacted matrices (Chen et al., 2020; Franke et al., 2019, 2021; Guo et al., 2021; Horst et al., 2018). In parallel, the commercial industry is responding to enacted regulations to remove long-chain perfluoroalkyl acids (PFAAs) from products, such as fire-fighting foams and other commercial products. Industrial cleaning to remove residual PFAS within process equipment, stockpiles of commercial products containing long-chain PFAAs, and the concentrated waste streams associated with adsorptive and separative treatment technologies create an urgent need for the validation of practical field-scale destructive technologies.

Advanced reduction processes (e.g., nonthermal plasma [Stratton et al., 2017], electron beam [Wang et al., 2016], and/or ultraviolet activated catalysts/reductants [Tenorio et al., 2020]), sonochemical degradation (Mader et al., 2008), electrochemical treatment (Schaefer et al., 2015), and high-temperature thermal oxidation or pyrolysis (USEPA, 2020a), all provide PFAS-relevant destruction mechanisms. While there is a focus in the literature around the comparative energy consumption (kilowatt-hours per cubic meter; kW-h/m³) of these technologies, the application of any of these technologies to dilute PFAS-impacted matrices at high flowrates seems cost-prohibitive and impractical. On the basis of the pioneering work of Bentel (Bentel et al., 2020, 2019), the bond dissociation energies determined for the PFAA chains within these molecules will necessitate energy-intensive mineralization. Considering the promising developments of PFAS-relevant treatment mechanisms, an evaluation of destructive technologies in batch or continuous closed-loop circulation for concentrated waste destruction based on energy consumption may be more relevant (i.e., kW-h per mass of PFAS destroyed; Kalra et al., 2021).

A common solution for the terminus of a PFAS treatment train is commercial incineration (USEPA, 2020a). Multiple studies related to the thermolysis of specific PFAS adsorbed to, or studied as, solid matrices provide compelling data of at least partial defluorination/decomposition of PFAS (Ellis et al., 2001; Krusic et al., 2005; Krusic & Roe, 2004; Matula, 1968; Tsang et al., 1998; Yamada & Taylor, 2003; Yamada et al., 2005). Recent research has evaluated the efficacy of thermal treatment as it pertains to the reactivation of activated carbon (Sasi et al., 2021; Sonmez Baghirzade et al., 2021; Watanabe et al., 2018; 2016; F. Xiao et al., 2020). Despite the evident thermal decomposition of long-chain PFAAs that are currently considered analytically, observations from the literature referenced here and elsewhere (Altarawneh et al., 2021; Arkenbout, 2018; Horst et al., 2020; Liu et al., 2021; Winchell et al., 2020) suggest incomplete mineralization of PFAS under various thermal treatment conditions may result in undetected gaseous emissions or concentration within the ash. In response to these concerns, the USEPA developed Other Test Method 45 (OTM-45) (Merrill et al., 2021) which incorporates PFAS-specific components to gaseous emission sampling.

Supercritical water oxidation (SCWO) may be a relevant solution for the complete mineralization of PFAS in various liquid and slurry waste streams. For water, the supercritical condition is achieved at pressures exceeding 22 mega Pascals (MPa) and temperatures above 374°C (Cengel & Boles, 2002) and is defined by a single fluid phase that is a fraction of the density of water with a tremendous capacity to dissolve gas and organic compounds while precipitating inorganic species. Since its introduction in the 1980s from work at the Massachusetts Institute of Technology, SCWO has been thoroughly reviewed throughout the literature for various applications and potential challenges, and various patents have been pursued and awarded (Hong & Spritzer, 2002; Krause et al., 2022; Kritzer & Dinjus, 2001; Marrone, 2012; Pinkard et al., 2021; Tester et al., 1993; Yesodharan, 2002). Recent research on the applicability of high-pressure thermal treatment (e.g., SCWO and alkaline hydrothermal treatment) has demonstrated a high degree of certainty for the complete mineralization of perfluorooctane sulfonic acid (PFOS) in water (Krause et al., 2022; Pinkard et al., 2021; Wu et al., 2019), PFAS within wastewater treatment sludge (Yu et al., 2020), and PFAS associated with aqueous film-forming foam (AFFF) (Hao et al., 2021; Pinkard, 2022). The theorized mechanism of SCWO facilitated destruction is thermal oxidation of PFAS, resulting in fully mineralized carbon dioxide and inorganic fluoride, though instantaneous sequential defluorination reactions related to chemical oxidation and/or reduction via manipulated pH may also occur (Wu et al., 2019).

Our work summarized here attempts to validate a continuous flow SCWO process to mineralize PFAS associated with a PFOS-dominant AFFF by considering both liquid effluent and gaseous emissions. A concentrated source of PFAS was selected to align with the likely application of SCWO. As the ability of SCWO to mineralize PFAS in liquids has been demonstrated (Krause et al., 2022; Pinkard et al., 2021), this validation study pertained to the mineralization of PFAS in both the liquid and gaseous effluents.

2 | MATERIALS AND METHODS

2.1 | Selected AFFF and associated dilution

The AFFF selected for this validation was 3M Lightwater™ ATC Plus™ (manufacture date: November 1989) used to extinguish hydrocarbon fuel and/or polar solvent-related fires. This AFFF was manufactured using electrochemical fluorination, and analyses of the product summarized in the literature (Backe et al., 2013; Houtz, 2013; Lang & Divine, 2020; Place & Field, 2012) suggest the overwhelming majority of the PFAS in 3M Lightwater™ ATC Plus™ are represented by approximately 12 PFAAs (C4–C8 perfluorocarboxylic acids [PFCAs] and C4–C10 perfluorosulfonic acids [PFSAs]). It is acknowledged that there were likely polyfluorinated compounds present (such as perfluoroalkyl sulfonamide amino carboxylates), potentially at concentrations of up to 1 g/L, which would be expected to sequentially transform into some of the 12 PFAAs analyzed under aggressive oxidation conditions (Houtz, 2013) during the mineralization process.

A stock solution of a 1000-fold diluted AFFF was used for this validation. Dilution was selected to avoid excess foaming and related gas-liquid partitioning concerns for confirmation analysis purposes (Brusseau, 2017, 2019a, 2019b; Lyu et al., 2018; Schaefer et al., 2019). The diluted AFFF fed into the SCWO reactor will be referred to as the influent AFFF hereafter.

For discussion purposes herein, long-chain PFAAs ($\Sigma\text{PFAA}_{\text{LC}}$) are defined as six or more fluorine-saturated carbons for PFSA and seven or more fluorine-saturated carbons for PFCAs (Buck et al., 2011). Short-chain PFAAs ($\Sigma\text{PFAA}_{\text{SC}}$) are then referred to as those PFSA and PFCAs with less than six and less than seven fluorine-saturated carbons in the associated alkyl chain, respectively.

2.2 | Test parameters and process configuration

The validation testing was performed at the General Atomics facility in San Diego, CA, in March 2021. Two duplicate test runs (continuous flow) were conducted (Run 1 and Run 2), each lasting approximately 140 min to facilitate sample train maneuvering over 120-min test durations for the purposes of gaseous emissions sample collection. Parameters associated with the two duplicate test runs are summarized in Table 1.

The process equipment used for this test included a high-pressure air compressor, a startup fuel-fired preheater, the high-pressure titanium-lined reactor where the SCWO reactions occurred, a pressure letdown system, a low-pressure gas-liquid separator (GLS), chemically compatible and pressure-rated conveyance piping and connections, and requisite holding tanks for the influent AFFF and treated effluent. A flow schematic of the SCWO process is conceptualized in Figure 1.

The influent AFFF and fuel (low-sulfur diesel) were fed into the high-pressure reactor through a nozzle designed to effectively mix

the oxidant (air) with the influent AFFF/diesel in a reaction zone that is maintained at 650°C. Quench water was introduced around the outside of the high-pressure reactor towards the bottom to decrease the temperature to approximately 300°C before entering the pressure letdown system. Sodium hydroxide (NaOH) was added to the quench water to neutralize anticipated hydrofluoric acid (HF), which blended with the effluent from the high-pressure reactor. The effluent was then conveyed to the GLS under atmospheric pressure, where gas/water vapor was vented to the atmosphere, and the liquid was conveyed through a heat exchanger to further decrease the temperature to approximately 55°C.

2.3 | Liquid and gaseous sample collection

Liquid effluent samples were collected as composite samples of the discharge from the heat exchanger throughout the runs. For Run 1, a single composite sample was collected. For Run 2, two composite samples were collected, each representing one-half of the test run. Samples were collected in laboratory-issued high-density polyethylene (HDPE) bottles and were refrigerated until submission to the analytical laboratory under standard chain of custody procedures.

Gaseous effluent samples were collected using the general isokinetic test procedures defined in USEPA Method 5 (USEPA, 2020b), with modifications similar to USEPA Method 23 (USEPA, 2017) and USEPA Method 0010 (USEPA, 1986). The gaseous effluent sample collection was also consistent with OTM-45. The SCWO exhaust stack was constructed to provide the straight run distances required for isokinetic sampling. Briefly, a continuous sample was withdrawn using a 16-point traverse over the stack cross-sectional area (17.5 cm²) using two perpendicular axes and conveyed into a sample probe, and then through a glass fiber filter, through a packed column

TABLE 1 Test parameters and associated measurements

Test parameter	Run 1	Run 2	Average	Test parameter	Run 1	Run 2	Average
Diesel consumed (L)	107	106	107	Influent air volume (L)	2,310,651	2,310,651	2,310,651
Diesel feed rate (lpm) ^a	0.89	0.88	0.9	Run time (min)	120	120	120
AFFF concentrate (L)	1.1	1.0	1.0	Residence time (s)	10–11	10–11	10–11
Influent AFFF treated (L)	1,085	997	1,041	SCWO temperature (°C) ^a	649.1	648.5	648.8
Influent AFFF feed rate (lpm) ^a	9.1	8.3	8.7	SCWO pressure (MPa) ^a	22	22	22
Liquid effluent volume (L)	1,359	1,301	1,330	% Excess oxygen ^a	10.6%	12.4%	11.5%
Liquid effluent flow rate (lpm) ^a	11.3	10.9	11.1	pH post-Quench ^a	6.16	6.05	6.11
Quench volume (L)	2,844	2,768	2,806	Corrected stack flow rate (dslpm)	20,181	19,512	19,847
Quench feed rate (lpm) ^a	23.7	23.1	23.4	Metered gas volume (dscm)	2.2	2.4	2.3
Influent air flow rate (lpm)	19,255	19,255	19,255	Water collected in sample train (L)	1.9	2.3	2.1

Abbreviations: AFFF, aqueous film-forming foam; dscm, dry standard cubic meters; dslpm, dry standard liters per minute; lpm, liter per minute; SCWO, supercritical water oxidation.

^aData presents averages from continuous measurements recorded at a 3 s frequency over each run (Run 1 = 2695 log entries; Run 2 = 2662 log entries).

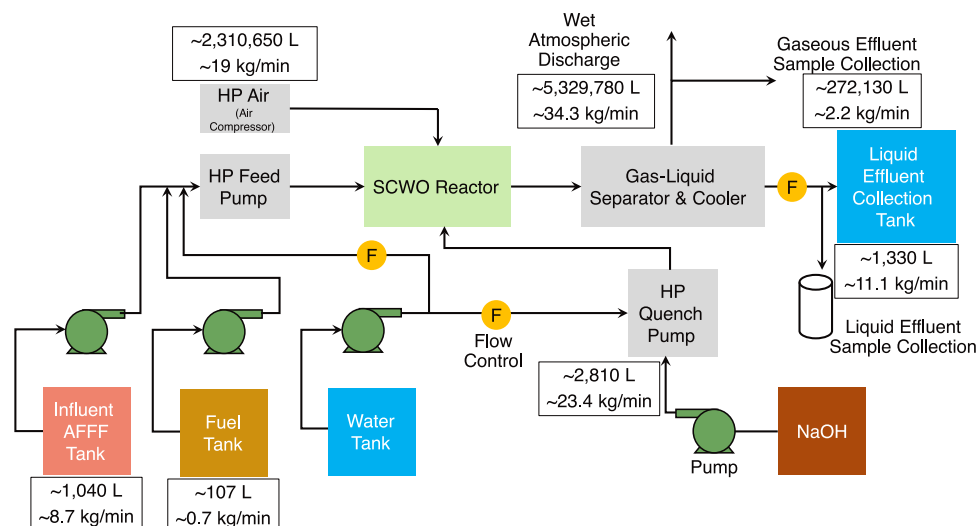


FIGURE 1 SCWO process schematic and mass flow balance for General Atomic's pilot system. AFFF, aqueous film-forming foam; SCWO, supercritical water oxidation; kg/min, kilograms per minute; L, liters; NaOH, sodium hydroxide; HP, high pressure [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.com)]

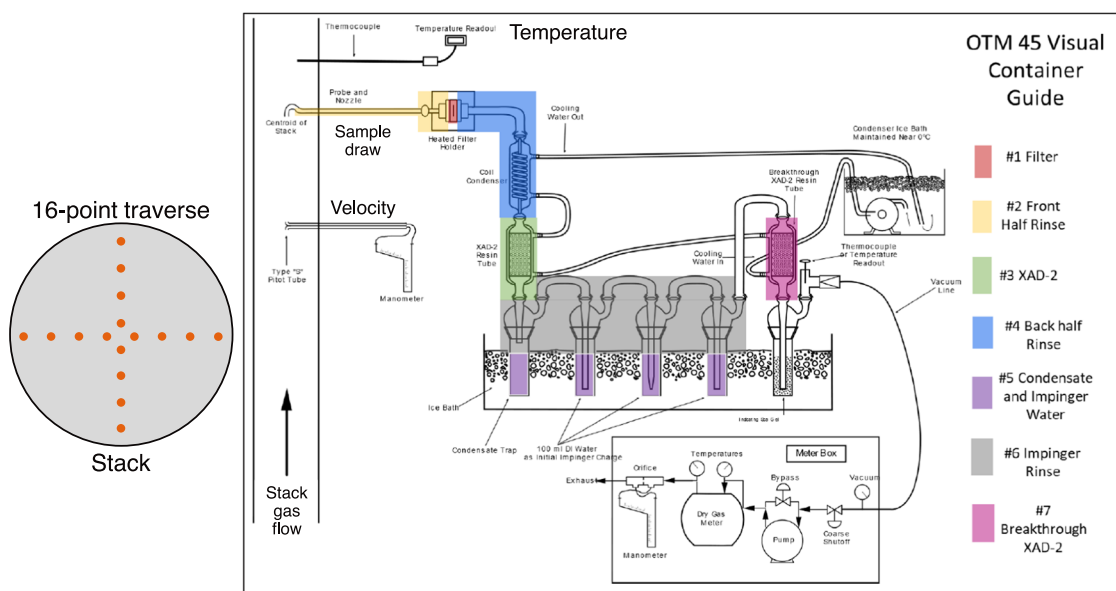


FIGURE 2 USEPA method OTM-45 sample train for PFAS in gaseous emissions from a stack (Merrill et al., 2021). OTM-45, Other Test Method 45; PFAS, per- and polyfluoroalkyl substances; USEPA, United States Environmental Protection Agency [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.com)]

of adsorbent XAD-2 resin and ice-chilled impingers charged with distilled-deionized water (ddH₂O). A second identical XAD-2 adsorbent resin was located at the end of the sample train to evaluate any breakthrough of PFAS during sample collection. A schematic of the sampling train for isokinetic sampling is presented in Figure 2 (Merrill et al., 2021).

At the conclusion of each test run, the sample train condensates, rinses, and filters were recovered into HDPE containers. XAD resin traps were sealed, and all samples were submitted to the analytical

laboratory in icepack-chilled coolers under standard chain of custody procedures.

Glassware preparation and cleaning were performed by Arcadis for the sample train and SGS North America Inc. (SGS) for the XAD-2 resin and quartz fiber filters. Glassware for the sample train was soaked in 50°C soapy (Alconox[®]) water, followed by sequential triplicate rinses using hot tap water, ddH₂O, acetone, dichloromethane, and methanol. After baking for 2 h at 600°C, the glassware was triple rinsed with a methyl alcohol/5% ammonium hydroxide

(MeOH/NH₄OH) solution. XAD-2 resin was cleaned with methanol, then soaked in MeOH/NH₄OH solution until use. Following cleaning and drying, all glassware, filters, and XAD-2 resin traps were capped and sealed for transport to the test site. The sampling train was assembled without vacuum grease in a clean, dust-free area.

2.4 | Laboratory analysis

All laboratory analysis was conducted by SGS, using USEPA OTM-45 modified method for the measurement of PFAS in stack gas sample components. The method uses isotope-dilution high-performance liquid chromatography with tandem mass spectrometry (LC-MS/MS). Analytical methods for PFAS in stack gas samples are not currently accredited due to lack of availability.

Sample extractions were performed based on the component. The filter and XAD-2 traps were fortified with isotopically labeled standards and extracted sequentially using MeOH/NH₄OH solution. Following extraction, evaporation was carried out to remove methanol and reconstitute the extract in water. Impinger samples (water) were spiked with isotopically labeled standards, adjusted to a pH of 7 ± 1. Water samples and filter/XAD extracts were subject to further cleanup using weak anion exchange.

Target PFAS were analyzed by LC-MS/MS and quantified using a multipoint calibration (minimum of 5 points). The analytes were measured relative to their isotopically labeled/surrogate counterparts. Final data were recovery corrected. A blank sample was analyzed using a clean reference XAD-2/filter and processed simultaneously with the samples. In addition, spiked ongoing performance and recovery samples were created in the same fashion and fortified with both the isotopically labeled extraction standards and native analytes to ensure accurate quantitation. Additional quality control included the use of isotopically labeled field standards that were added to the XAD-2 trap before sampling. The analysis included the 12 PFAAs reported as a total mass in the sample train, with anticipated nominal analytical reporting limits between 1 and 12.5 ng for each individual PFAA.

No advanced analytical techniques (such as the Total Oxidizable Precursor Assay) were used because the selected AFFF was well established in the literature to have minimal precursor contribution. Fluoride analysis was performed on the liquid effluent only (i.e., not on the steam) using USEPA Method 300/SW846 9056A. The total number of samples and associated analytical methods are summarized in Table 2.

3 | THEORY/CALCULATION

For all calculations, where analytical results were reported as less than the reporting limit, the reporting limit was used. There was negligible variation between the reporting limits for the duplicate tests.

The destruction and removal efficiency (DRE) calculation was based on the percent difference between the influent mass flow rate and the sum of the liquid and gaseous effluent mass flow rates. The influent mass flow rate was calculated as follows:

$$M_{x,inf} = C_{x,inf} \times Q_{inf} \times \frac{1 \mu\text{g}}{1000 \text{ ng}},$$

where $M_{x,inf}$ is the influent mass flow rate (micrograms per minute [$\mu\text{g}/\text{min}$]) for an individual PFAA, $C_{x,inf}$ is the analytically determined concentration for an influent PFAA (nanograms per liter [ng/L]), and Q_{inf} is the influent AFFF flow rate (liters per minute [lpm]).

The liquid effluent mass flow rate was calculated as follows:

$$M_{x,liq} = C_{x,liq} \times Q_{liq} \times \frac{1 \mu\text{g}}{1000 \text{ ng}},$$

where $M_{x,liq}$ is the liquid effluent mass flow rate ($\mu\text{g}/\text{min}$) for an individual PFAA, $C_{x,liq}$ is the analytically determined concentration for a PFAA in the liquid effluent in ng/L , and Q_{liq} is the liquid effluent flow rate (lpm).

The gaseous effluent mass flow rate was calculated as follows:

$$M_{x,gas} = \frac{C_{x,gas}}{V_{gas}} \times Q_{gas} \times \frac{60 \text{ min}}{1 \text{ h}} \times \frac{1 \mu\text{g}}{1000 \text{ ng}},$$

where $M_{x,gas}$ is the gas effluent mass flow rate ($\mu\text{g}/\text{min}$) for an individual PFAA, $C_{x,gas}$ is the sum of quantified masses for an individual PFAA from the sample train digestion in ng , V_{gas} is the total volume of stack gas passed through the sample train (dry standard cubic meters), and Q_{gas} is the gaseous effluent flow rate based on measured parameters during sampling and corrected to dry standard cubic meters per minute.

The DRE% was then calculated as follows:

$$\text{DRE} = \frac{M_{x,inf} - (M_{x,liq} + M_{x,gas})}{M_{x,inf}} \times 100.$$

The overall defluorination ratio (%deF_x) was then calculated after Bentel et al. (2019) as follows:

$$\sum_{X=1}^{12} \text{overall } \%deF_X = \frac{C_{F^-}}{\sum_{X=1}^{12} (C_{x,inf} \times N_{C-F})} \times 100\%,$$

where C_{F^-} is the molar concentration of fluoride in the liquid effluent (analytically determined), and the initial molar concentration of organofluorine was calculated as the summation of the molarity of each PFAA considered multiplied by the number of carbon-fluorine bonds within the respective PFAA (N_{C-F}). This calculation was corrected using the background fluoride concentration (400 $\mu\text{g}/\text{L}$) in the dilution and quench water, obtained from the City of San Diego Annual Drinking Water Quality Report (City of San Diego, 2020).

Sample identification	Analysis	Methods
Run 1 AFFF Feed	PFAS	USEPA Method OTM-45 ^a
Run 1 SCWO Eff	PFAS, F	USEPA Method OTM-45 ^a ; USEPA 300/SW846 9056A
Run 1 Quench	PFAS	USEPA Method OTM-45 ^a
Run 1 filter and rinses-FR	PFAS	USEPA Method OTM-45 ^a
Run 1 XAD front—XAD 1	PFAS	USEPA Method OTM-45 ^a
Run 1 condensate—IMP	PFAS	USEPA Method OTM-45 ^a
Run 1 XAD back—XAD2	PFAS	USEPA Method OTM-45 ^a
Run 2 AFFF Feed	PFAS	USEPA Method OTM-45 ^a
Run 2A SCWO Eff	PFAS, F	USEPA Method OTM-45 ^a ; USEPA 300/SW846 9056A
Run 2B SCWO Eff	PFAS, F	USEPA Method OTM-45 ^a ; USEPA 300/SW846 9056A
Run 2 Quench	PFAS	USEPA Method OTM-45 ^a
Run 2 filter and rinses-FR	PFAS	USEPA Method OTM-45 ^a
Run 2 XAD front—XAD1	PFAS	USEPA Method OTM-45 ^a
Run 2 condensate—IMP	PFAS	USEPA Method OTM-45 ^a
Run 2 XAD back—XAD2	PFAS	USEPA Method OTM-45 ^a
FB XAD front—XAD1	PFAS	USEPA Method OTM-45 ^a
FB condensate—IMP	PFAS	USEPA Method OTM-45 ^a
FB XAD back—XAD2	PFAS	USEPA Method OTM-45 ^a

TABLE 2 Sample identification and associated analysis

Abbreviations: OTM-45, Other Test Method 45; PFAS, polyfluoroalkyl substances; USEPA, United States Environmental Protection Agency.

^aUSEPA method OTM-45 (Merrill et al., 2021) was followed closely by SGS North America, Inc., with slight modification as discussed in Section 2.4 and remained largely consistent with Quality Systems Manual 5.3 (USDoD/USDoE, 2019).

Energy requirements related to this validation are calculated using an adapted version of the Electric Energy per Mass (E_{EM}) parameter (adapted from Bolton et al., 2001), which represents the electric energy in kW-h needed to degrade a unit mass of the subject constituent in the impacted matrix. The E_{EM} for work presented here is calculated as follows:

$$E_{EM} = \frac{\sum E}{M_i - M_f},$$

where $\sum E$ is the energy consumed (kW-h) and M_i and M_f are the initial and final mass of the sum of all 12 PFAAs calculated as follows:

$$M_i = \sum_{X=1}^{12} (C_x \times V_{AFFF}),$$

$$M_f = \sum_{X=1}^{12} (C_{x,gas} \times V_{gas}) + \sum_{X=1}^{12} (C_{x,liq} \times V_{liq}),$$

where V_{AFFF} is the volume of influent AFFF treated, V_{gas} is the volume of gaseous effluent, and V_{liq} is the volume of liquid effluent.

4 | RESULTS AND DISCUSSION

4.1 | Analytical results

The analytical results for the influent AFFF were in good agreement with the historical PFAS analytical data for 3M Lightwater in the literature (Backe et al., 2013; Houtz, 2013) and were consistent between Run 1 and Run 2 (Table 3). Perfluorohexane sulfonate (PFHxS) and PFOS accounted for an average of 94.7% of the 12 PFAAs analyzed. Approximately 1 L of AFFF concentrate was treated in each Run (Table 1), and the average mass of PFAAs per Run in the influent AFFF was 13.7 g.

The city water used for diluting the AFFF and quenching (i.e., to direct process fluids to the subcritical regime and neutralize HF) was determined to contain four detected PFAAs (perfluorobutanoic acid [PFBA], perfluoropentanoic acid [PFPeA], perfluorooctanoic acid [PFOA], and PFOS) at concentrations ranging from 2.1 to 4.3 ng/L and 10 estimated detections (i.e., J flagged) for other PFAAs ranging from 0.7 J to 2.0 J ng/L (Table 3). In comparison to the influent PFAAs, the PFAAs contribution from the dilution/quench water is insignificant (i.e., $\mu = 53 \mu\text{g}$ or 0.0004% of the influent PFAAs mass). The quench water was added to the liquid effluent after the SCWO

TABLE 3 Analytical results for the influent, quench, and liquid and gaseous effluents

PFAAs	Influent results				Quench					
	Run 1 ng/g	ng/L	Run 2 ng/g	ng/L	Run 1 ng/L	Run 2 ng/L				
PFBA	35.14	35,140	35.13	35,130	2.6	2.5				
PFPeA	35.17	35,170	32.93	32,930	1.96 J ^a	3.1				
PFHxA	86.6	86,600	100.3	100,300	1.93 J ^a	1.87 J ^a				
PFHpA	37.93	37,930	28.71	28,710	0.895 J ^a	0.746 J ^a				
PFOA	72.21	72,210	83.22	83,220	2.1	1.48 J ^a				
PFBS	146.9	146,900	147.8	147,800	1.13 J ^a	1.14 J ^a				
PFPeS	159.4	159,400	167.3	167,300	<0.688 ^a	<0.691 ^a				
PFHxS	717.1	717,100	855.6	855,600	1.52 J ^a	0.914 J ^a				
PFHpS	85.89	85,890	91.99	91,990	<0.511 ^a	<0.513 ^a				
PFOS	10,780	10,780,000	12,590	12,590,000	4.3	3.1				
PFNS	10.56	10,560	14.02	14,020	<0.199 ^a	2 J ^a				
PFDS	<8.734 ^a	<8734 ^a	<7.813 ^a	<7813 ^a	<0.199 ^a	2 J ^a				
PFAAs	Effluent results									
	Run 1 ng/L	Run 2A ng/L	Run 2B ng/L	Average Run 2 ng/L						
PFBA	2.8	3.0	2.8	2.9						
PFPeA	2.5	2.2	2.4	2.3						
PFHxA	2.4	2.1	2.5	2.3						
PFHpA	0.961 J ^a	0.718 J ^a	0.771 J ^a	0.7						
PFOA	2.2	1.79 J ^a	1.45 J ^a	1.6						
PFBS	2.0	1.74 J ^a	1.71 J ^a	1.7						
PFPeS	<0.686 ^a	<0.672 ^a	<0.717 ^a	0.7						
PFHxS	1.67 J ^a	1.44 J ^a	2.3	1.9						
PFHpS	<0.509 ^a	<0.499 ^a	<0.532 ^a	0.5						
PFOS	5.6	3.4	4.6	4.0						
PFNS	<1.98 ^a	<1.95 ^a	<2.08 ^a	2.0						
PFDS	<1.98 ^a	<1.95 ^a	<2.07 ^a	2.0						
PFAAs	Gaseous effluent sample train results									
	Run 1					Run 2				
F/R ng	XAD (F) ng	ddH ₂ O ng	Sum ng/train	Conc. ng/m ³	F/R ng	XAD (F) ng	ddH ₂ O ng	Sum ng/train	Conc. ng/m ³	
PFBA	<1.25 ^a	<1.25 ^a	<5 ^a	7.5	3.43	1.51 J ^a	2.0	<6.25 ^a	9.8	4.15
PFPeA	0.375 J ^a	0.5	<1 ^a	1.8	0.84	0.403 J ^a	<0.25 ^a	1.9 J ^a	2.6	1.09
PFHxA	0.6	0.4	<1 ^a	1.9	0.89	0.7	0.3 J ^a	<1.25 ^a	2.3	0.96
PFHpA	0.368 J	<0.3 ^a	<1.2 ^a	1.9	0.86	0.6	0.332 J ^a	<1.5 ^a	2.4	1.02
PFOA	1.1	0.334 J ^a	<1.2 ^a	2.6	1.21	2.6	<0.3 ^a	<1.5 ^a	4.4	1.87
PFBS	<1 ^a	<1 ^a	<4 ^a	6.0	2.75	<1 ^a	<1 ^a	<5 ^a	7.0	2.98
PFPeS	<0.2 ^a	<0.2 ^a	<0.8 ^a	1.2	0.55	<0.2 ^a	0.2	<1 ^a	1.4	0.61
PFHxS	0.316 J ^a	<0.25 ^a	<1 ^a	1.6	0.72	0.372 J ^a	<0.25 ^a	<1.25 ^a	1.9	0.80
PFHpS	<0.35 ^a	<0.35 ^a	<1.4 ^a	2.1	0.96	<0.35 ^a	<0.35 ^a	<1.75 ^a	2.5	1.04

(Continues)

TABLE 3 (Continued)

PFAAs	Gaseous effluent sample train results									
	Run 1					Run 2				
	F/R	XAD (F)	ddH ₂ O	Sum	Conc.	F/R	XAD (F)	ddH ₂ O	Sum	Conc.
ng	ng	ng	ng/train	ng/m ³	ng	ng	ng	ng/train	ng/m ³	
PFOS	1.8	0.3	1.3	3.4	1.57	1.5	0.7	<1.5 ^a	3.7	1.57
PFNS	<0.5 ^a	<0.5 ^a	<2 ^a	3.0	1.37	<0.5 ^a	<0.5 ^a	<2.5 ^a	3.5	1.49
PFDS	<0.5 ^a	<0.5 ^a	<2 ^a	3.0	1.37	<0.5 ^a	<0.5 ^a	<2.5 ^a	3.5	1.49

Abbreviations: ddH₂O, distilled-deionized water; F/R, filter and rinse; ng/g, nanogram per g; PFBA, perfluorobutanoic acid; PFBS, perfluorobutane sulfonic acid; PFDS, perfluorodecane sulfonic acid; PFHpA, perfluoroheptanoic acid; PFHpS, perfluoroheptane sulfonic acid; PFHxA, perfluorohexanoic acid; PFHxS, perfluorohexane sulfonic acid; PFNS, perfluorononane sulfonic acid; PFOA, perfluorooctanoic acid; PFOS, perfluorooctane sulfonic acid; PFPeA, perfluoropentanoic acid; PFPeS, perfluoropentane sulfonic acid; XAD F, front XAD resin.

^aResult is below the shown reporting limit or J means the result is detected but estimated below the reporting limit.

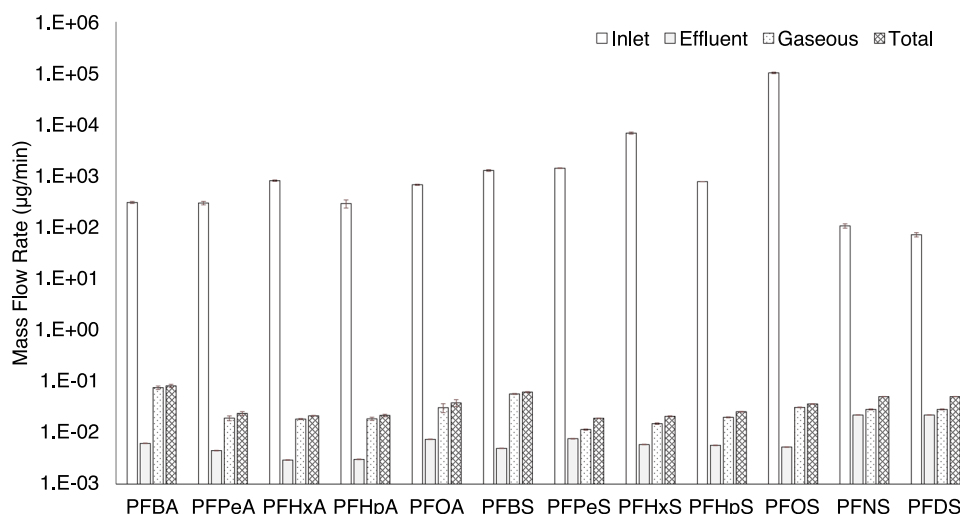


FIGURE 3 Logarithmic comparison of influent and effluent mass flow rates. PFBA, perfluorobutanoic acid; PFBS, perfluorobutane sulfonic acid; PFDS, perfluorodecane sulfonic acid; PFHpA, perfluoroheptanoic acid; PFHpS, perfluoroheptane sulfonic acid; PFHxA, perfluorohexanoic acid; PFHxS, perfluorohexane sulfonic acid; PFNS, perfluorononane sulfonic acid; PFOA, perfluorooctanoic acid; PFOS, perfluorooctane sulfonic acid; PFPeA, perfluoropentanoic acid; PFPeS, perfluoropentane sulfonic acid

process, and the contribution of PFAAs from the quench water should be subtracted from the liquid effluent. However, the flow rate of the quench water in comparison to the liquid effluent flow rate is such that a subtraction results in negative mass flow rates (Table 1). Liquid effluent samples had detections of PFBA, PFPeA, PFHxA, PFOA, PFBS, PFHxS, and PFOS ranging from 2.0 to 5.6 ng/L (Table 3). On the basis of volume of liquid effluent, the PFAAs mass within the liquid effluent was an average of 32 µg or 0.0002% of the measured influent PFAA mass. On the basis of the large difference in concentrations between the influent and the liquid effluent and the negative mass flow rates when trying to correct for the quench water, the quench water PFAAs contribution was partially accounted for by assuming the liquid effluent was equivalent to the reporting limits.

Gaseous effluent results were reported as the mass of PFAAs per component of the sampling apparatus (filters, front XAD resin, and the water in the impingers). In both Run 1 and Run 2, the filters and

front XAD resin had the greatest number of PFAAs detections (Table 3), while the water from the impingers was mostly below analytical reporting limits, with only one detection. On the basis of these results, the XAD resin on the back of the sampling train was unlikely to be exposed to any PFAAs and was not released for analysis. On the basis of the volume of gaseous effluent measured, the PFAA mass within the gaseous effluent was an average of 42 µg or 0.0003% of the measured influent PFAA mass.

4.2 | Influent and effluent mass flow rates

A logarithmic comparison of the average influent versus the average effluent liquid and gas mass flow rates is presented in Figure 3.

The mass flow rates depicted in Figure 3 (µg/min) are a slight divergence from conventional gaseous emissions measurements (typically grams per hour [g/h]) but were purposefully selected due to

the small masses of PFAAs observed in the liquid and gaseous effluent (i.e., average $C_{\text{PFOS, gas}} = 0.03 \mu\text{g}/\text{min}$ or $1.8 \times 10^{-7} \text{g}/\text{h}$). The difference between the influent mass flow rate and the individual and/or combined liquid and gaseous effluent mass flow rates is considerable. These results strongly suggest that the SCWO conditions described here were effective at destroying the PFAAs analyzed in this validation study.

4.3 | Destruction and removal efficiency

The average influent, liquid effluent, gaseous effluent, total effluent, and calculated DRE% for each PFAA analyzed in this study are summarized in Table 4.

The average DRE% for all 12 PFAAs is greater than 99.999%. The highest and lowest DRE% were for PFOS and perfluorodecane sulfonate (PFDS), respectively. Note that PFDS was below detection limits in the influent AFFF.

4.4 | Long-chain PFAAs versus short-chain PFAAs

Data presented here suggest an appreciable percentage of the influent PFAS are not present in the gaseous or liquid effluents, regardless of chain length, post-SCWO. Specifically, for $\Sigma\text{PFAA}_{\text{LC}}$ reported in this study, of the $1.3 \times 10^7 \mu\text{g}$ in the influent, $8.2 \mu\text{g}$ and

$18.5 \mu\text{g}$ were observed in the liquid and gaseous effluents, respectively (DRE% > 99.999%). For $\Sigma\text{PFAA}_{\text{SC}}$ reported in this study, of the $0.5 \times 10^6 \mu\text{g}$ in the influent, $3.5 \mu\text{g}$ and $24.0 \mu\text{g}$ were observed in the liquid and gaseous effluents, respectively (DRE% > 99.99%).

The gaseous effluent mass flow rate accounts for most of the total effluent mass flow rate. This appears to be an artifact of using the reporting limits for calculations (rather than actual detections where there were none). For example, on average PFBS appears to account for approximately 92% of the total PFBS effluent mass flow rate; however, PFBS is below analytical reporting limits for each component of the gaseous sampling apparatus (Table 3). This is an important observation in this data because there are concerns throughout the industry that some high-temperature thermal oxidation applications may not result in complete PFAS mineralization (Altarawneh et al., 2021; Arkenbout, 2018; Horst et al., 2020; Liu et al., 2021; Winchell et al., 2020). $\Sigma\text{PFAA}_{\text{LC}}$ are suspected of defluorinating/transforming into $\Sigma\text{PFAA}_{\text{SC}}$ or unknown intermediates and the concern is that $\Sigma\text{PFAA}_{\text{SC}}$ or unknown intermediates may go undetected because gaseous emission sampling protocols for PFAS have not been fully developed. A key focus of this validation study is the gaseous emission sampling, and the data presented here strongly support SCWO effectively addressing PFAAs considering both liquid and gaseous effluent.

To further explore the potential for $\Sigma\text{PFAA}_{\text{SC}}$ formation under SCWO conditions, a comparison of the percentages of $\Sigma\text{PFAA}_{\text{SC}}$ and $\Sigma\text{PFAA}_{\text{LC}}$ before and after SCWO was performed. Before SCWO

TABLE 4 Average (Avg) and standard deviation (Std Dev) DRE%

PFAA	Avg influent ($\mu\text{g}/\text{min}$)	Std Dev ($\mu\text{g}/\text{min}$)	Average liquid effluent ($\mu\text{g}/\text{min}$)	Std Dev ($\mu\text{g}/\text{min}$)	Average gaseous effluent ($\mu\text{g}/\text{min}$)	Std Dev ($\mu\text{g}/\text{min}$)	Total effluent ($\mu\text{g}/\text{min}$)	Std Dev ($\mu\text{g}/\text{min}$)	Avg DRE%	Std Dev
PFBA	304.9	12.9	0.01 ^a	9.7E-05 ^a	0.08	5.9E-03	0.08	5.8E-03	99.97319%	3.0E-05
PFPeA	295.5	22.2	0.00 ^a	7.1E-05 ^a	0.02	2.1E-03	0.02	2.0E-03	99.99197%	1.3E-05
PFHxA	811.0	25.2	0.00 ^a	4.5E-05 ^a	0.02	4.0E-04	0.02	3.5E-04	99.99737%	3.9E-07
PFHpA	289.2	52.2	0.00 ^a	4.7E-05 ^a	0.02	1.3E-03	0.02	1.3E-03	99.99250%	1.8E-05
PFOA	674.5	19.3	0.01 ^a	1.2E-04 ^a	0.03	6.0E-03	0.04	5.9E-03	99.99434%	7.2E-06
PFBS	1278.8	50.1	0.00 ^a	7.3E-05 ^a	0.06 ^b	1.3E-03 ^b	0.06	1.3E-03	99.99516%	2.9E-06
PFPeS	1417.6	25.6	0.01 ^a	1.2E-04 ^a	0.01	3.7E-04	0.02	2.5E-04	99.99865%	4.2E-07
PFHxS	6824.4	312.7	0.01 ^a	9.0E-05 ^a	0.02	5.3E-04	0.02	4.4E-04	99.99969%	7.6E-08
PFHpS	771.9	6.1	0.01 ^a	8.8E-05 ^a	0.02 ^b	4.7E-04 ^b	0.03	3.8E-04	99.99668%	7.5E-07
PFOS	101,408.5	3570.1	0.01 ^a	7.9E-05 ^a	0.03	5.1E-04	0.04	5.9E-04	99.99996%	1.9E-08
PFNS	106.7	10.5	0.02 ^a	2.9E-04 ^a	0.03 ^b	6.6E-04 ^b	0.05	3.8E-04	99.95257%	4.4E-05
PFDS	71.8 ^a	7.0 ^a	0.02 ^a	3.2E-04 ^a	0.03 ^b	6.6E-04 ^b	0.05	3.5E-04	99.92958%	7.4E-05
Sum	114,254.7		0.10		0.40		0.45		99.99960%	

Abbreviations: DRE%, destruction and removal efficiency; PFAA, perfluoroalkyl acid; PFBA, perfluorobutanoic acid; PFBS, perfluorobutane sulfonic acid; PFDS, perfluorodecane sulfonic acid; PFHpA, perfluoroheptanoic acid; PFHpS, perfluoroheptane sulfonic acid; PFHxA, perfluorohexanoic acid; PFHxS, perfluorohexane sulfonic acid; PFNS, perfluorononane sulfonic acid; PFOA, perfluorooctanoic acid; PFOS, perfluorooctane sulfonic acid; PFPeA, perfluoropentanoic acid; PFPeS, perfluoropentane sulfonic acid.

^aValues calculated based upon the average of analytical reporting limits.

^bValues calculated based upon the average of analytical reporting limits for all components of the sample train.

treatment, the average percentage of $\Sigma\text{PFAA}_{\text{SC}}$ of the total PFAAs measured was 3.8% (vs. 96.2% as $\Sigma\text{PFAA}_{\text{LC}}$). After SCWO treatment, this percentage split was observed to shift to 50.8% $\Sigma\text{PFAA}_{\text{SC}}$ versus 49.2% $\Sigma\text{PFAA}_{\text{LC}}$. Despite the average >99.99% DRE%, the shift in the percentage of $\Sigma\text{PFAA}_{\text{SC}}$ from 3.8% to 50.8% suggests that even in the SCWO conditions described here, there could be some $\Sigma\text{PFAA}_{\text{LC}}$ defluorinating/transforming into $\Sigma\text{PFAA}_{\text{SC}}$. This observation would also be supported by postulated defluorination mechanisms of PFSAs proceeding through PFCAs (H. Park et al., 2009; Wu et al., 2019) and the lower observed DRE% for PFCAs versus PFSAs. Another explanation for this observed percentage split shift could be less effective SCWO for $\Sigma\text{PFAA}_{\text{SC}}$. The DRE% is shown with respect to increasing fluorine saturated carbon chain length in Figure 4.

For both PFCAs and PFSAs, there does appear to be a slight increase in the DRE% with respect to fluorine saturated carbon chain length. An apparent exception to this is PFNS and PFDS, both of which represented the lowest influent concentrations and were below reporting limits for all liquid and gaseous effluent measurements (Table 3). The data presented here does not enable a definitive conclusion as to whether the 3.8%–50.8% $\Sigma\text{PFAA}_{\text{SC}}$ increase is related to incomplete mineralization of $\Sigma\text{PFAA}_{\text{LC}}$ or comparatively less effective SCWO of $\Sigma\text{PFAA}_{\text{SC}}$, and the overwhelming decrease of all 12 PFAAs considered in this study may overshadow single-digit ng/L residual $\Sigma\text{PFAA}_{\text{SC}}$.

4.5 | Fluoride considerations

The theoretical calculation of organic fluorine in the influent was compared to analytical results for fluoride within the liquid effluent. The theoretical organic fluorine determined stoichiometrically based on the

quantified PFAAs in the influent AFFF was an average of approximately 8,465 mg/L. When corrected for the 1000-fold dilution, this calculation (approximately 8.47 g/L) is in good agreement with the lower range of analytical results in the literature for 3M Lightwater manufactured in 1989 (Houtz, 2013). Gaseous measurements of fluoride (i.e., in the steam) were not collected due to budget and time constraints. The average overall defluorination ratio was calculated to be 62.6%. A partial fluorine mole balance is presented in Figure 5.

On average, approximately 28.1% (152.9 mmol) of the anticipated molar fluorine was not recovered. The quench/NaOH addition to the effluent of the high-pressure reactor likely neutralized HF resulting in fluoride dissolved in the steam being discharged from the reactor. Based on the liquid recovered from the gaseous sampling train, an average moisture content of 55.3% was determined in the gaseous discharge. Therefore, approximately 2,948,184 L of the volume discharge (Figure 1) is assumed to be steam. This translates to an approximate concentration of 0.99 $\mu\text{g/L}$ fluoride in the steam, which is plausible. Despite selecting a well-known source of AFFF to conduct this validation study, an alternative explanation for the unrecovered fluoride could be nonionic potentially fluorinated intermediate compounds in the gaseous discharge that would require nontarget analysis to discover. In future testing, it may be appropriate to analyze for previously observed PFAS degradation products, such as formate, acetate, sulfate, and trifluoroacetate (Singh et al., 2019; Thomas et al., 2020).

4.6 | Energy considerations

The E_{EM} determination for this validation study is summarized in Table 5 and compared to some other destructive technologies in the literature (i.e., not an exhaustive comparison).

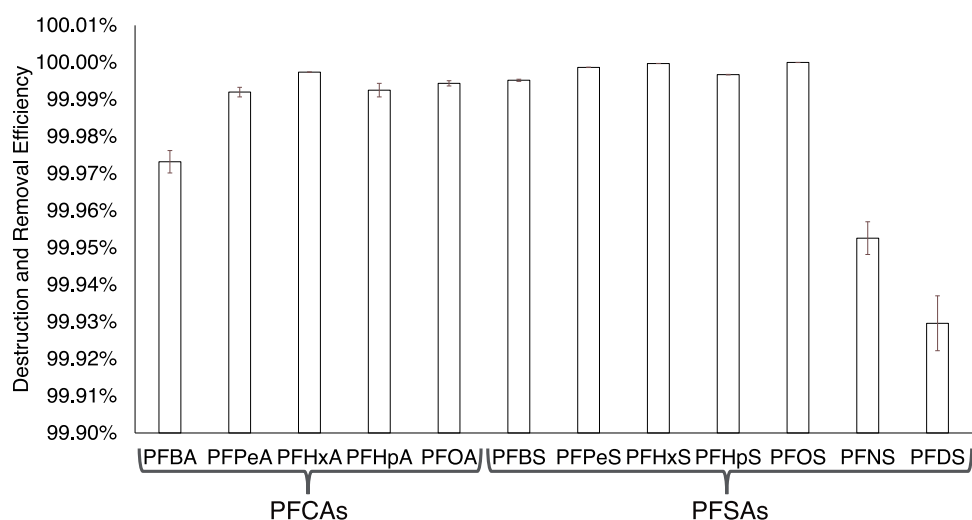


FIGURE 4 Comparison of DRE% with respect to chain length and functional group. DRE%, destruction and removal efficiency; PFBA, perfluorobutanoic acid; PFBS, perfluorobutane sulfonic acid; PFCA, perfluorocarboxylic acid; PFDS, perfluorodecane sulfonic acid; PFHpA, perfluoroheptanoic acid; PFHpS, perfluoroheptane sulfonic acid; PFHxA, perfluorohexanoic acid; PFHxS, perfluorohexane sulfonic acid; PFNS, perfluorononane sulfonic acid; PFOA, perfluorooctanoic acid; PFOS, perfluorooctane sulfonic acid; PFPeA, perfluoropentanoic acid; PFPeS, perfluoropentane sulfonic acid; PFSAs, perfluorosulfonic acid

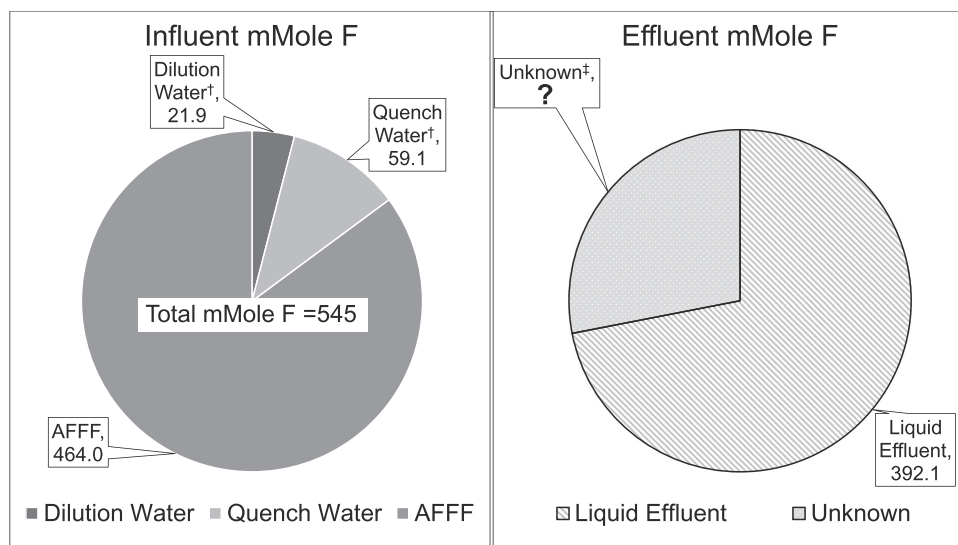


FIGURE 5 Partial fluorine mole balance. [†]Background fluorine concentration of 0.4 $\mu\text{g/L}$ (City of San Diego, 2020). [‡]Fluoride was not sampled in the gaseous effluent

TABLE 5 Calculated E_{EM} for example PFAS destruction technologies

Technology	Reference	Energy consumed kW·h/m ³	Experimental conditions ^a					Mass of PFAS destroyed kg	Energy per mass kW·h/kg
			Time h	Vol L	C_i $\mu\text{g/L}$	C_f $\mu\text{g/L}$	PFAS		
Electrochemical oxidation	Gomez-Ruiz et al. (2017)	256	10.0	2.0	1,623	4.2	Multi	3.2×10^{-6}	1.6×10^5
	Soriano et al. (2017)	15	1.5	1.0	870,000	87,000.0	PFHxA	7.8×10^{-4}	1.9×10^1
	Schaefer et al. (2018)	99	8.0	0.3	451	50.6	Multi	1.0×10^{-7}	2.5×10^5
		136	8.0	0.3	539	126.4		1.0×10^{-7}	3.3×10^5
Sonolysis	Vecitis et al. (2008)	208	3.0	0.6	5,001	0.0	PFOS	3.0×10^{-6}	4.2×10^4
		208	2.0	0.6	4,141	0.0	PFOA	2.5×10^{-6}	5.0×10^4
	Moriwaki et al. (2005)	3,333	1.0	0.1	10,000	7,200.0	PFOS	1.7×10^{-7}	1.2×10^6
		3,333	1.0	0.1	10,000	3,700.0	PFOA	3.8×10^{-7}	5.3×10^5
	Cheng et al. (2010)	448	2.3	0.6	100	5.0	PFOS	5.7×10^{-8}	4.7×10^6
		1,055	2.3	0.6	100	28.0	PFOA	4.3×10^{-8}	1.5×10^7
Plasma	Singh et al. (2019)	294	1.0	4.0	141	9.1	Multi	5.3×10^{-7}	2.2×10^6
Alkaline hydrothermal	Wu et al. (2019)	306	1.5	0.0	50,000	0.1	PFOS	2.7×10^{-7}	6.1×10^3
SCWO Run 1	This study	1,398	2.0	1,085.4	12,176	0.0	Multi	1.3×10^{-2}	1.1×10^5
SCWO Run 2		1,506	2.0	997.4	14,155	0.0		1.4×10^{-2}	1.0×10^5

Abbreviations: C_i , initial concentration; C_f , final concentration; E_{EM} , electric energy per mass; PFAS, per and polyfluoroalkyl substance; PFHxA, perfluorohexanoic acid; PFOA, perfluorooctanoic acid; PFOS, perfluorooctane sulfonic acid; SCWO, super critical water oxidation; Vol, volume.

^aBased on stated experimental conditions.

For conditions described here, the average E_{EM} was determined to be 1.1×10^5 kW·h/kg PFAS. The primary energy source was diesel fuel assumed to have a high-heating value of 45.6 MJ/kg (Staffell, 2011) and an average density of 0.84 kg/L (Staffell, 2011). The energy associated with the air compressor to create the high-pressure

SCWO conditions and the transfer pump were also included but were significantly less than the energy source of the diesel. A 100% efficiency was assumed for the diesel as is a well-established assumption throughout SCWO applications consistent with operation in this study (Tester et al., 1993).

With respect to other forms of destructive technologies (e.g., electrochemical treatment, sonolysis, nonthermal plasma, and alkaline hydrothermal) from a small sample of the available literature, corresponding E_{EM} were calculated based on the various experimental conditions and suggest that the E_{EM} of SCWO is reasonable. It is emphasized that "reasonable" is with respect to the destruction of concentrated PFAS waste streams. A comparison of E_{EM} versus conventional incineration was intentionally not performed due to scale, current unknowns with respect to gaseous emissions from commercial incinerator applications, and the disparate and variable PFAS attempted to be destroyed with incineration. Considering the range and average of the E_{EM} of the non-SCWO destruction technologies (1.9×10^1 – 1.5×10^7 ; $\mu = 2.0 \times 10^6$; $n = 12$; $\sigma = 4.0 \times 10^6$), the E_{EM} of this SCWO validation study is less than the average and considerably more PFAs mass was treated. It is notable that the calculated E_{EM} associated with alkaline hydrothermal treatment ($\sim 350^\circ\text{C}$ and <16.5 MPa) is approximately 94% lower than SCWO for the conditions reported, which is expected given the lower temperature and pressure and enhanced reductive defluorination facilitated under alkaline treatment conditions (Wu et al., 2019) and could be optimized further (Hao et al., 2021).

5 | SUMMARY

Considering the questions that have arisen around the complete mineralization of PFAS in liquid wastes currently being addressed via incineration, the concept of SCWO has high relevance to PFAS. The SCWO technology is not new and its recent application to PFAS destruction of liquids or slurries (Pinkard et al., 2021) benefits from the commercial development of the technology since the early 1980s, leveraging lessons learned from demilitarization and radiological waste disposal (Hong & Spritzer, 2002). This includes sustainability improvements to utilize energy-efficient fuels and harness waste heat to make SCWO more efficient (General Atomics, 2005).

The objective of this validation study for SCWO destruction of a concentrated PFAS waste stream was to evaluate a commercially available and highly relevant technology with respect to gaseous and liquid effluent. Based on the greater than 99.999% DRE% for the sum of 12 PFAAs considered and an average 62.6% overall defluorination ratio, it appears that SCWO can effectively mineralize PFAS at the field scale. The testing conditions studied here could be optimized to include higher temperatures, longer residence times, higher concentrated feeds (as foaming of the 1000-fold dilution influent was negligible), or different oxidant additions. While optimization of SCWO reactors could improve the achievable mineralization of PFAAs, this validation study strongly suggests SCWO is capable of mineralizing PFAAs with a comparable E_{EM} to other relevant PFAS destruction technologies.

This validation study also summarizes a field-scale application of the recently developed OTM-45 for capturing and measuring PFAS within gaseous stack emissions. Isokinetic sampling procedures deployed here are largely consistent with current industry best

practices, but also serve to help validate OTM-45 and the PFAS-associated components to reliably detect PFAAs discharge down to ng per sample train. The method appears to have successfully met its purpose and may have benefited from the aggressive treatment conditions of SCWO. These results should be interpreted carefully, and it may be inappropriate to assume a similar level of accuracy and capture from a manufacturing process or commercial incinerator waste stream in the presence of a greater percentage of competing organics.

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