Water Treatment Technologies for PFAS: The Next Generation

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Introduction

In May 2009 at the fourth meeting of the Stockholm Convention (Convention) in Geneva, Switzerland, the Conference of Parties added perfluorooctane sulfonic acid (PFOS) to the treaty under Annex B (Stockholm Convention 2018). This restricted production and use on PFOS and initiated global research on a broader list of per and polyfluoroalkyl substances (PFAS). Finding ways to manage PFAS in the environment is a clear focus for many stakeholders and something that has gained intensity because of a combination of forces:

- Regulatory developments such as the long-term health advisory in the U.S. Environmental Protection Agency (USEPA 2016), regulations in Australia and Europe, and the review of perfluorooctanoic acid (PFOA) and perfluorohexane sulfonic acid (PFHxS) for inclusion in the Convention.
- Rapidly evolving toxicology and epidemiological research (Frisbee et al. 2009; Steenland et al. 2010; Halldorsson et al. 2012; Danish Ministry of the Environment 2015; Mastrantonio et al. 2017; Post et al. 2017; He et al. 2018).
- Public awareness due to detection in groundwater resources used for drinking water (Hu et al. 2016).
- The fact that there are more than 3000 compounds including the perfluoroalkyl group in the class of

chemical termed PFAS (Wang et al. 2017).

The factors above are driving interim water treatment applications for public water supplies and informing state regulatory decision-making with respect to groundwater compliance (ITRC 2017; NGWA 2017). However, these are moving targets, and as the scientific community continues to develop a better understanding of the toxicology associated with various PFAS, the list of PFAS subject to regulation is already growing, depending on regulatory attention in differing U.S. states and countries. Further, based on the adoption of nonenforceable and enforceable regulatory standards globally, environmental practitioners should anticipate targeting concentrations in the parts per trillion (ppt) range in drinking water. These ultra-low regulatory standards in matrices other than drinking water have raised questions as to the accuracy of current commercially available laboratory techniques, and analytical chemists are working toward finalizing matrix-specific analysis for PFAS (Mills 2017).

In addition to these challenges, the stability leading to recalcitrance and persistence plus aquifer mobility that were imparted to many PFAS, have fundamentally limited the effectiveness of most conventional water treatment technologies to address them. As with every other contaminant that has emerged to challenge the remediation community, academic and industry professionals are collaborating globally to research and develop innovative new ways to remove PFAS from water or destroy PFAS altogether. Helpful literature review documents summarizing the progress of PFAS treatment technologies provide a state of the practice assessment, but also highlight that even relevant treatment technologies may have considerable hurdles to handle practical treatment applications at the field-scale (Vecitis et al. 2009; Du et al. 2014; Merino et al. 2016; Kucharzyk et al. 2017; Ross et al. 2018) and the inapplicability of conventional treatment technologies (Appleman et al. 2014; Higgins and Dickenson 2016).

In a previous column, we discussed two perspectives for managing this rapidly evolving issue via short-term situational management strategies and long-term site restoration strategies (Suthersan et al. 2016). The focus of this column is to continue that discussion by exploring the next generation of treatment technologies for water that are currently emerging for PFAS—rather than another exhaustive review of available PFAS water treatment technologies. This requires that we differentiate between what is possible and what is practical, so to set the stage we will start with a cursory discussion of both PFAS chemistry and the key challenges with current approaches for PFAS water treatment.

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PFAS Chemistry: Just the Basics

While there are plenty of references that walk through PFAS chemistry in great detail, a brief overview is worthwhile prior to exploring the effectiveness of various available and emerging water treatment techniques. As previously mentioned, the compounds PFOA and PFOS are part of a large class of more than 3000 fluorinated compounds commonly referred to as PFAS. Organofluorine compounds, such as PFAS, have been used to make many different commercial products as a result of their relatively unique properties (Prevedouros et al. 2006; Fujii et al. 2007; Houtz et al. 2013). The terminology used to describe individual molecules in the class of PFAS is typically abbreviated into numerous acronyms which can initially be overwhelming to the nonscientific community; however, this nomenclature is purposeful and informative. Every PFAS contains a common structural element of one or more fully fluorinated alkyl moiety $(C_n F_{2n+1})$, which is referred to as a perfluoroalkyl group (Buck et al. 2011). When these perfluoroalkyl groups are synthesized to create chains of various lengths, numerical abbreviations are used to describe the compound. For example, PFOS has eight (octa) perfluoroalkyl groups. The perfluoroalkyl chains are resistant to dissolution in water and oils, and therefore by affixing a functional group (e.g., carboxyl, sulfonate, phosphate) to the end of a perfluoroalkyl chain this dramatically changes the molecules properties making them water soluble and, for the long-chain perfluoroalkyl group, inclusion of this polar head group generates surface acting agent (i.e., surfactant) properties.

Fully fluorinated perfluoroalkyl carbon chains (between 2 and 18 carbons) with either carboxyl, sulfonate, or phosphate functional groups and perfluoroalkyl ethers (such as GenX and ADONA¹) are commonly referred to as perfluoroalkyl acids (PFAA). This class of compound includes PFOS and PFOA and mainly speciates in aquifer systems as anionic, soluble compounds with sorption potential increasing with perfluoroalkyl chain

length and negligible volatility (Pancras et al. 2016). Researchers have further subdivided PFAA into long- and short-chain classifications depending on the bioconcentration/bioaccumulation of the PFAA, with long-chain PFAA being more bioaccumulative than short-chain PFAA. Despite less-adsorptive bioaccumulation, short-chain PFAA typically have higher solubilities, may bioconcentrate in fruits and vegetables (Blaine et al. 2014a; Blaine et al. 2014b), and are less amenable to adsorption-based removal techniques suggesting a need for understanding short-chain PFAAassociated toxicology (Danish Ministry of the Environment 2015).

When a perfluoroalkyl group is a component of a compound (such as a fluorotelomer alcohol), containing one or more carbon-hydrogen bonds, the entire compound is referred to as polyfluorinated. Polyfluorinated compounds represent a significantly more diverse group of PFAS with differing physical and chemical properties which are based on the nature of the functional groups attached to the perfluoroalkyl chain. Polyfluorinated compounds include a broad range of functional groups which complicates aquifer speciation because these compounds can have variable charges (neutral, anionic, cationic, zwitterionic [i.e., both cationic and anionic]), low to high sorption potential, and potential volatility (Backe et al. 2013). With respect to fate and transport in aquifers, biological, and chemical oxidation in situ reactive zones, and metabolism in higher organisms, polyfluorinated compounds and PFAA are interrelated because polyfluorinated compounds transform into PFAA (Wang et al. 2005; Vestergren et al. 2008; Dasu et al. 2012; Weiner et al. 2013; Harding-Marjanovic et al. 2015; Gebbink et al. 2015).

In this way, polyfluorinated compounds are often referred to as "precursors" of eventual persistent PFAA. Although polyfluorinated compounds have been demonstrated to biotransform into PFAA via other transient intermediates, the resultant PFAA are not amenable to further biodegradation (Colosi et al. 2009; Liu and Mejia Avendano 2013; Ochoa-Herrera et al. 2016; Ross et al. 2018). Therefore, as regulatory standards evolve for various PFAA and some individual polyfluorinated compounds (i.e., precursors), the full loading of precursors are of concern from a water treatment perspective because they will eventually form PFAA. Refinements in commercial laboratory analytical procedures (US Department of Defense/US Department of Energy 2017) and affordable advanced quantification techniques, such as the total oxidizable precursor (TOP) assay (Houtz and Sedlak 2012), are invaluable to a holistic understanding of the nature and extent of PFAS requiring treatment. As new PFAS are continually identified using forensic analysisinuniversitylaboratories(Barzen-Hanson et al. 2017), considering both precursor polyfluorinated compounds and PFAA maximizes the value of water treatment resources.

Addressing PFAS with Currently Available Water Treatment Technology

Currently, there are no widely accepted in situ groundwater treatment technologies that address the full range of PFAS. Permeable reactive barriers that enable active replacement of spent adsorbents in shallow groundwater are conceivable, but commercial available adsorbents are not generally applicable to all PFAS. Conventional in situ groundwater treatment technologies such as air sparging, enhanced aerobic bioremediation, and in situ chemical oxidation have been shown to facilitate transformation of precursors into PFAA (McGuire et al. 2014), but ultimately are ineffective at removing or destroying PFAA. Therefore, groundwater treatment strategies typically involve extraction and ex situ treatment. Upon extraction of groundwater from the aquifer, available treatment mechanisms include combinations of adsorption, separation, and destruction. The current state of the practice for ex situ treatment of water impacted by PFAS generally involves use of sorptive technologies which create a secondary waste as PFAS are not destroyed. There appears to be no single technology which is effective for removal and destruction of all shortand long-chain PFAA. It is noteworthy to acknowledge that municipal water

¹3H-perfluoro-3-[(3-methoxy-propoxy)propanoic acid]

treatment represents a similar treatment scenario for PFAS but at a significantly larger capacity and therefore as a result of the scale is not directly comparable to ex situ groundwater treatment.

Adsorption technologies, such as granular activated carbon (GAC) and ion exchange (IX), are currently the most commonly encountered interim response measures to achieve immediate compliance with drinking water standards and serve as the benchmark of practicality and effectiveness for other treatment technologies. For example, both GAC and IX resins can remove PFOA and PFOS from water at concentrations in the parts per billion range (ppb) to the ppt range with greater than 90% efficiency (Du et al. 2014). Comparative isotherms presented in the literature are often marred by different influent concentrations or geochemical conditions, and site-specific rapid small-scale column testing is recommended for specifying these adsorbents. As more interim water treatment applications are installed, inherent inefficiencies with treating large volumes of water with low concentrations of PFAA will become apparent. Conventional adsorption technologies provide an immediate decrease to PFAA detected in public water supplies; however, due to the comparatively low treatment goals, breakthrough is inevitable and can be at relatively low bed volumes if removal of short-chain PFAA is required.

Continued Research and Development (R&D) on conventional adsorbents highlights new information on inefficient/ineffective removal of shortchain PFAA and precursors (Xiao et al. 2017) raising questions as to the future-proofing of these interim water treatment applications. Engineering IX resins with PFAA-specific functional groups may improve PFAA-specific removal (Zaggia et al. 2016), but it is unclear how effective they might be toward a much broader cross section of the greater than 3000 PFAS. As is typical with adsorption-based technologies, in situations with comparatively higher concentrations of PFAS (100s to 1000s of ppb), pretreatment ahead of adsorption-based removal is often helpful to extend the operational

lifetime of the adsorbent but requires an effective separation technology to lower the concentration in the primary water feed such that it can be effectively polished with adsorption. Conventional separation technologies, such as reverse osmosis (RO) and/ or nanofiltration (NF), can be highly effective for PFAS removal (>99%), but have other fundamental limitations such as significant capital cost for larger systems, moderate efficiency of contaminant rejection (Higgins and Dickenson 2016), and low throughput flow rates that constrain the range of useful applications in point of entry treatment systems.

The Next Generation of Water Treatment Technology for PFAS

The next generation of viable PFAS water treatment technologies is continuing to build on the treatment mechanisms of adsorption, separation, and destruction. The following are examples:

- A next generation technology that is gaining attention in the area of adsorption-based removal involves organically modified silica adsorbents. As an example, in the Strategic Environmental Research and Development Program (SERDP) funding year 2018, 9 of the 11 R&D proposals focused on PFAS treatment that were accepted for funding were focused on either optimizing adsorption or regenerating spent adsorbents (SERDP 2018). One of these grants is focused on the evaluation of a modified silica adsorbent, which will be discussed in more detail here.
- A next generation technology that is gaining attention in the area of separation is referred to as "ozofractionation." This technology exploits the surfactant nature of PFAS. We will discuss work being done with the United States and Australian patent-holder for this technology (EVOCRA), including a field-scale demonstration in Australia.
- Several next generation technologies that are gaining attention as viable destruction-based technologies include sonolysis, electrochemical treatment, and a combination of advanced oxidation

and advanced reduction processes (AOP/ARP). Challenges in this area include comprehensive PFAS destruction as well as energy consumption limitations (Ross et al. 2018). While forms of combined AOP/ARP such as plasma (Stratton et al. 2017) and eBeam irradiation (Wang et al. 2016) are progressing rapidly at laboratory-scale assessments, this column will focus on the potential for practical-scale implementation of sonolysis and electrochemical treatment.

The following discussions dive deeper into the technologies highlighted above, in each of the three categories of treatment.

Adsorption: Organically Modified Silica

The adsorption of PFAS is influenced by many variables including perfluoroalkyl chain length, and concentration, the hydrophilic functional group (e.g., sulfonate vs. carboxylate), the solution pH and ionic strength, the concentration of natural organic matter (NOM) and other cocontaminants, and stearic hindrance and molecular size as it relates to the available surface binding sites. As the perfluoroalkyl chain length increases so does the hydrophobicity of the PFAA, and literature reports suggest the partitioning coefficient can increase 0.5 to 0.75 log units per moiety (Higgins and Luthy 2006; Ahrens et al. 2010). This provides a plausible explanation as to why conventional adsorbents relying on hydrophobic adsorption struggle to remove short chain PFAA. Furthermore, an increase of approximately 0.23 to 0.76 log units has been postulated for sulfonates compared to carboxylates, and more efficient adsorption removal of PFOS has been observed in the literature compared to PFOA (Higgins and Dickenson 2016). Adsorption of precursors will be determined by individual chemical structure as these species may be cationic, anionic, zwitterionic, or neutral. PFAA exist as anionic species and thus can bind through both electrostatic interactions with charged groups on the adsorbent surface and hydrophobic interactions between the perfluoroalkyl chain and the uncharged surface. Acidic pH in an aquifer presents a greater percentage of positively charged surface area, which may enhance PFAA adsorption. High concentrations of NOM/ cocontaminants may adversely influence PFAA adsorption through competition for adsorption sites (Hong et al. 2013). A historical optimization to GAC was the achievement of a higher iodine number, or an increase in the percentage of microporosity, which improved the removal effectiveness for smaller molecules, such as volatile organic compounds. Due to the competition for adsorption sites between PFAA and NOM, a greater percentage of mesoporosity (i.e., slightly larger porosity) has been observed to be more favorable for GAC-based PFAA removal (Nassi et al. 2014; Nowack 2017).

The developing technology of swellable organically modified silica or mesoporous organosilica (MPOS) targets exploiting a purely hydrophobic adsorption mechanism to remove PFAS from water in a potentially reversible manner. The viability of MPOS to adsorb both pure-phase and dissolvedphase organic compounds has been investigated for the past 15 years and is well documented throughout the literature (Edmiston and Underwood 2009; Edmiston and Jolly 2014; Yang and Spoonamore 2012). The MPOS adsorbent is a silica-based polymeric structure developed by ABS Materials and consists of cross-linked alkoxysilanes (Edmiston 2013) and has shown exceptional capability to interact with the perfluoroalkyl moiety common to all PFAS rather than the polar functional groups. MPOS adsorbents generally have a greater percentage of mesoporosity than other commercially available materials, which is expected to be more favorable for adsorption of PFAS. An area of current R&D focus is understanding the adsorption mechanism of PFAS to the MPOS adsorbent. Various forensic analytical techniques are proposed as part of an ongoing SERDP research grant to determine the mode of interaction between PFAS and the MPOS adsorbent. The hypothesis of MPOS adsorbents is that the PFAS adsorbs strongly via purely hydrophobic interactions related to the fluorinated carbon chain. This would imply that regardless of the electrostatic charge of the polyfluorinated precursor

or PFAA (long chain or short chain), removal should be broadly applicable. One of the most intriguing features of this adsorbent is that subtle changes during MPOS synthesis that can meaningfully alter surface hydrophobicity and pore size. Another objective of ongoing R&D is if copolymerization of variable organosilane precursors during the synthesis of MPOS adsorbents can be engineered to create a flexible pore space, which may have direct influence on mitigating competitive adsorption between PFAA and cocontaminants (Edmiston 2017).

Due to the surfactant nature of PFAS, it is prudent to discuss the potential for micelle and hemimicelle formation as the concentrations of individual PFAS increases. In most instances for groundwater treatment, the individual critical micelle concentrations (CMC) for PFAS are not relevant when considering aquifer concentrations ranging from 100s of ppt to 1000s of ppb (i.e., CMC for PFOA is on the order of parts per thousand [Bhhatarai and Gramatica 2011]), and PFAS are expected to be dissolved in groundwater. However, in an adsorbent treatment vessel, interadsorbate interactions may occur such as the formation of hemimicelles (Deng et al. 2010). The synergistic benefit of hemimicelle formation within conventional adsorbents is also possible, but with MPOS adsorbents hemimicelle formation may be intentionally engineered through modifications of the synthesis process to establish optimal pore structures. The development of numerous adsorption isotherms under environmentally relevant conditions with varying ionic strength and surface chemistry conditions will inform optimal synthesis processes facilitating self-organization of PFAS to improve adsorption capacity (Edmiston 2017).

The specially formulated MPOS adsorbents discussed herein are commercially available from ABS Materials in two forms: (1) as a bulk adsorbent under the commercial name Osorb® and (2) deposited as a film through covalent attachment to sand filtration media and porous silica media under the commercial name PuraSorbTM. The Osorb is described in the literature as "swellable" because the flexible pore structure expands to more than 2.5

times its size when placed in solvents and is more appropriate for higher influent concentrations of PFAS. PuraSorb does not swell as it is affixed to sand media and is more appropriate for lower influent concentrations of PFAS. Both media have been certified as filtration media that is safe for use in drinking water purification under the National Sanitation Foundation (NSF) and American National Standards Institute (ANSI) 42, 61, and 372 standards. As more innovative adsorbents become marketable, practitioners should ensure that suppliers have met NSF and relevant ANSI certifications as this can be a critical factor in regulatory decision-making at the feasibility level.

Available analytical data as part of "proof-of-concept" testing indicates that MPOS adsorbents remove PFAS equally or better than commercially available adsorbents. Adsorption isotherms for PFOA, PFOS, and perfluorobutanoic acid (PFBA) removal using Osorb are presented in Figure 1 (Edmiston 2017). It is noteworthy to mention that of the commercially laboratory quantifiable PFAA, PFBA represents the most soluble and leastadsorptive PFAA (miscible solubility at 25 °C and comparatively low partitioning constant [Pancras et al. 2016]), and therefore presents the greatest current challenge to adsorption-based removal technologies. Because the isotherms in Figure 1 remain linear, the MPOS adsorbent did not become saturated from the initial PFAA concentration of 2000 ppb. Adsorption isotherms for PFOA and PFOS removal using PuraSorb are presented in Figure 2 (Edmiston 2017). A key difference between Osorb and PuraSorb is the amount of MPOS adsorbent present, as PuraSorb has only 0.5% by weight on the filtration media. The isotherms presented in Figure 2 demonstrate saturation of the MPOS adsorbent over the 30-min equilibration time, but the adsorption capacity is approximately 10 times less than that of Osorb with 200 times less MPOS adsorbent. This implies that PuraSorb may be more efficient with respect to removal and further supports its use for lower concentration PFAS removal from larger throughput volumes. While this data is "proof-of-concept" testing and will



Figure 1. Adsorption isotherms for (a) PFOA, (b) PFOS, and (c) PFBA on MPOS, commercially available as Osorb® (Edmiston 2017). Adsorption isotherms were measured by adding MPOS to deionized water containing either PFOA, PFOS, or PFBA at an initial concentration of 2000 ppb. The isotherms are linear, which imply that the MPOS was not saturated by the initial concentration. Noteworthy is that PFBA, the most difficult perfluoroalkyl acid to remove via adsorption, suggests the greatest adsorption. Lastly, Osorb has the ability to swell when in contact with a solvent, and the flexible pore space enables greater adsorption surface area as well as absorption based removal into the MPOS.



Figure 2. Adsorption isotherms for (a) PFOA, (b) PFOS, and (c) PFBA on MPOS, commercially available as PuraSorbTM (Edmiston 2017). Adsorption isotherms represent a 30-min equilibration time in deionized water, and show saturation. The PuraSorbTM adsorbent represents 0.5% by mass of the MPOS contained in Osorb (200 times less MPOS), yet shows only a 10 time less adsorption capacity. This suggests PuraSorbTM has high efficiency at low concentrations of poly and perfluoroalkyl substances, potentially attributable to greater access to porosity. Aside from an anomalous data point, PFBA shows similar to slightly greater affinity for PuraSorbTM than PFOA, which is consistent with Osorb isotherms

be further evaluated during the SERDP research, it is notable that PFBA shows greater adsorption to the MPOS adsorbent than PFOA—a long-chain PFAA.

Lastly, there is focus throughout the industry on the reusable nature of adsorbents. GAC can be reactivated under extreme thermal conditions that destroy adsorbed PFAS, disintegrates approximately 15% of the GAC, and returns a product that may be capable of improved PFAS removal. There are also commercial applications of IX resin onsite regeneration with solvent/ brine extractants (ECT2 2018). As MPOS adsorbents target purely hydrophobic adsorption, it is plausible that regeneration of the adsorbent could be achieved with a solvent rinse, but there are operational concerns of onsite management of a PFAS-enriched extract that needs further treatment. Optimizing throughput of IX resins and MPOS may make single use with incineration more economical than regeneration.

Separation: Ozofractionation

The ozofractionation technology leverages the surfactant nature of PFAS. While the head functional group is hydrophilic, the perfluoroalkyl chain is hydrophobic which makes the air-water interface of a bubble ideal for agglomeration of PFAS. Sparging ozone gas through tanks containing PFAS-impacted water creates a PFAS- enriched foam that can be collected (Figure 3). Therefore, the ozofractionation technology represents a new class of separation technology.

The ozofractionation technology is a patented process by the company EVOCRA and available commercially as Ozofractionative Catalyzed Reagent Addition (OCRA) (Dickson 2013, 2014). The OCRA system includes a series of water tanks in which micronsized (less than 200 µm) ozone gas bubbles are sparged through PFASimpacted water to facilitate air-water interface partitioning in foam, which is collected for further treatment. Smaller bubble size is intentional as it maximizes the surface area for collecting PFAS. From a groundwater treatment perspective, PFAS are commonly associated with cocontaminants (NOM and petroleum hydrocarbons) and the ozone can chemically mineralize these cocontaminants while separating PFAS from water. This is a distinct advantage as PFAS treatment effectiveness does not compete with cocontaminants. Because the mechanism of separation exploits the surfactant nature shared by short- and long-chain PFAA and polyfluorinated precursors, OCRA is broadly applicable to PFAS. Moreover, the sparged ozone may facilitate biological/chemical transformation of polyfluorinated precursors into PFAA.

With numerous polyfluorinated precursors potentially in waste streams that are (to date) unidentified (Houtz et al. 2016), the OCRA process provides a mechanism to remove a greater quantity of PFAS that other treatment technologies.

The process is multiphase and customizable as shown in Figure 4. Applicable waste streams include scum, sludge, and liquid with up to 20% solids, though the water quality of the influent directly influences the non-PFAS treatment considerations. For example, a high concentration of total suspended solids will require some form of sediment management, includ-



Figure 3. A photograph of the per- and polyfluorinated alkyl substance (PFAS)enriched foam fractionate emanating from ozone sparging hydrocylcones in Brisbane, Australia. PFASs partition at the air-water interface of the bubbles, leaving the water. The foam is then captured for subsequent destruction-based treatment (photo courtesy of Michael Dickson, 2017).

ing leach testing to evaluate PFAS potentially adsorbed to the sediment. Waste streams generated by the OCRA process can include sedimentation of coarse grained solids, the PFASenriched foam concentrate (potentially with fine-grained solids), and spent adsorbent media or filtered rejectate (if necessary). Depending on site-specific discharge criteria, polishing of water after ozone sparing may be necessary with secondary treatment (e.g., RO/ NF or commercial adsorbents). The OCRA system represents the state of the practice for PFAS-impacted water treatment in that it employs a treatment train to achieve discharge levels in the ppt range.

A summary of key OCRA demonstration data is presented in Table 1. The full data set represents performance on a suite of 28 PFAS and has been reported previously (Ross et al. 2017). For brevity, five specific PFAS are shown in Table 1, as well as a sum of all PFAS both pre and post TOP assay. The overall PFAS removal of the OCRA process for this specific dataset is notable: 28,800 to <2 ppt (>99.99% reduction). Throughout the ozofractionation step, an enrichment in short-chain PFAA perfluoropentanoic acid and perfluorohexanoic acid was observed, represented as a negative percent removal (data not shown). This enrichment in short-chain PFAA is indicative of biological/chemical transformation of the polyfluorinated precursors, some of which is attributed



Figure 4. Conceptual schematic of the OCRA process (Dickson 2013, 2014). The separation of poly and perfluoroalkyl substances (PFASs) occurs via the development of a foam fractionate captured for offsite destruction. Polishing treatments (adsorbents, filtration, etc.) may be added to achieve lower discharge limits as necessary.

Table 1

A Select Summary of a Larger Poly and Perfluoroalkyl Substances (PFASs) Data Set (Ross et al. 2017) Demonstrating the Separation Effectiveness of the Ozofractionative Catalyzed Reagent Addition (OCRA) Process

PFAS	Influent (ppt)	Ozofraction % Removal	Filtration % Removal	Treated Water (ppt)	Total % Removal
PFOS+PFHxS	535	98.13		<2	99.63
PFOA	341	97.07		<2	99.41
6:2 FtS	18,400	99.14	96.84	<5	99.97
PFPeA	1140	82.46	99.00	<2	99.82
PFHxA	1050	96.19	95.00	<2	99.81
Sum PFAS	7480	96.87	99.15	<2	99.97
Total PFAS, TOP assay	28,800	98.58	99.51	<2	99.99

The influent concentration of two long-chain perfluoroalkyl acids (PFAAs; PFOS and PFOA), two short-chain PFAAs (PFPeA and PFHxA), and one polyfluorinated precursor (6:2 FtS) are presented with subsequent removal during ozofractionation and polishing treatment. Noteworthy is the final entry in the table that presents an accurate reflection of total PFASs as confirmed by the total oxidizable precursor (TOP) assay showing >99.99% removal. OCRA is a patented process by EVOCRA (Dickson 2013, 2014). PFOS, Perfluorooctane sulfonate; PFPeA, Perfluoropentanoic acid; PFHxA, Perfluorohexanoic acid.

to 6:2 fluorotelomer sulfonate (6:2 FtS) but also to other polyfluorinated precursors. Although the ozofractionation process removed 98.58% of the total PFAS post TOP assay, residual PFAS were subsequently removed by an NF filtration to achieve >99.99% reduction, demonstrating the benefit of the treatment train concept of the OCRA process.

Despite the numerous advantages of the OCRA system (e.g., broad applicability to PFAS, customization, adaptive footprint, mitigation of competition with cocontaminants), the technology does represent a separation technology meaning it generates a PFAS-enriched waste stream that needs further treatment. In a recent full-scale commercial application of OCRA in Australia, approximately 40,000 cubic meters (m³) of PFAS-impacted water ranging in concentration from less than 1 to greater than 5000 ppb total PFAS (as confirmed by TOP assay) were reduced to approximately 800 m³ (98%) volume recovery) with approximately 1000-fold concentration of PFAS (Ross et al. 2017; M. D. Dickson, personal communication, 2018). This performance contrasted with RO/NF, which, depending on the background water quality, may exhibit recoveries ranging from roughly 50% to 80%, brings relevance to this next generation of separation technology and may pair well with more energy intensive, destruction-based technologies.

Destruction: Sonolysis and Electrochemical Treatment

The most experimental of the next generation of PFAS treatment technologies are the destruction-based technologies. Despite the resistance to biodegradation, there are reports throughout the literature of physical and chemical technologies achieving partial or complete destruction of some PFAS (Vecitis et al. 2009; Merino et al. 2016; Kucharzyk et al. 2017; Ross et al. 2018). There are several consistent questions associated with reported destruction-based technologies, such as effectiveness on short-chain PFAA and polyfluorinated precursors, legitimacy of the reported fluoride mass balances, energy requirements, and secondary water quality. The relevancy of destructive-based technologies for PFAS currently appears to be on smaller volume concentrated waste streams where treatment residence times can be manipulated via circulation to control energy requirements and secondary water quality.

With respect to physical destruction methods, incineration is being implemented for spent adsorbents and solid phase waste and represents a large energy demand with high cost. For chemical destruction methods, the known radicals associated with AOP (hydroxyl, sulfate, ozone) have demonstrated only marginal effectiveness on long-chain carboxylate PFAA under aggressive activation scenarios. The strong electronegativity of the fluorine atoms makes them more susceptible to reductive attack (Song et al. 2013), and numerous methods of ARP may have relevance. ARP theoretically has the potential to generate meaningful concentrations of the powerfully reducing solvated electron, with a standard reduction potential of -2.9 V (Buxton et al. 1988) but this species is rapidly scavenged by oxygen and some anions (i.e., nitrate) and its practical application for treating PFAS seems limited. One destructive-based technology that leverages ARP through direct electron transfer at the surface of an anode is electrochemical treatment.

Electrochemical treatment refers to direct electron transfer from an anode to a molecule within an electrochemical cell designed with an anode, cathode, and electrolyte. Electrochemical cells can be divided or undivided, have demonstrated effectiveness for PFOA/PFOS at current densities of 1 to 50 mA/cm², and use various custom-synthesized mixed metal oxide anodes (Schaefer et al. 2015; Urtiaga et al. 2015; Gomez-Ruiz et al. 2017; Schaefer et al. 2017). The materials of construction of an anode can have a meaningful influence on electrochemical treatment performance because the PFAS interaction at the surface of the anode constitutes the destructive mechanism. Tin, iron, and lead-based anodes have been found to be less effective for PFAS treatment, and, due to expected acidic conditions around the anode, are suspected of leaching heavy metals and facilitating PFAS adsorption-particularly for PFOS (Schaefer et al. 2015). Boron-doped diamond (BDD) anodes appear to be emerging as the most effective from an operational standpoint (Urtiaga et al. 2015), though R&D continues to identify new options such as the titanium suboxide anode (Huang 2017). Consistent with other literature on destruction of PFOS, PFOS is comparatively more challenging to destroy than PFOA under similar conditions. For example, electrochemical treatment was recently demonstrated to be approximately 90% effective for PFOA and 65% effective for PFOS at environmental relevant concentrations of 300 to 600 ppb using a BDD anode (Schaefer et al. 2017).

Indeed, electrochemical treatment of PFAS is an emerging technology, with the first known reporting (specific to PFOA and PFOS) in 2015 (Schaefer et al. 2015). Thus, while evidence has been reported throughout the literature recently on the potential for electrochemical treatment, this form of PFAS water treatment is under development. For example, the effectiveness of electrochemical treatment to mineralize long-chain PFAA requires further study as over typical residence times of 4 to 10 h the persistent generation of short-chain PFAA has been observed (Gomez-Ruiz et al. 2017). Further, as demonstrated in Figure 5, greater than 4000 mg per liter perchlorate was generated after 10 h of electrochemical treatment (Gomez-Ruiz et al. 2017). The production of toxic by-products (e.g., hydrogen fluoride, chlorine gas, bromate, perchlorate, and adsorbable organic halides) have been reported and may form when treating PFAS-contaminated wastewater with cocontaminants via electrochemical treatment (Trautmann et al. 2015). Water treatment techniques exist for perchlorate, such as IX resins and anaerobic bioreactors, but the additional cost of these technologies may limit the application of electrochemical treatment for PFAS destruction in many cases where waters contain, chloride or bromide.

Sonolysis for the destruction of PFAS in water has been demonstrated in the literature, though primarily in the laboratory (Cheng et al. 2008; Campbell et al. 2009; Vecitis et al. 2009). As it pertains to PFAS-impacted water treatment, sonolysis is the application of sound waves through water to facilitate cavitation of microbubbles ultimately pyrolyzing PFAS that have partitioned onto the surface of the bubbles. When the cavitation occurs, the temperature at the surface of the bubble can reach several thousand degrees Kelvin (quasi-adiabatically) which is well above the temperature threshold to destroy PFAS. This destruction mechanism is not reliant upon creating oxidizing or reducing radicals and therefore secondary water quality concerns are generally not an issue with sonolysis. Additionally, because thermal destruction is broadly applicable to the entire class of PFAS, sonolysis is expected to destroy short-chain PFAA and polyfluorinated precursors as well as more commonly studied PFAA (i.e., PFOA and PFOS).

The present limitation of both forms of destruction-based treatment is large-scale application. These technologies have a high energy demand and are generally not applicable to high volume, rapid flow rate applications common to ex situ treatment. Combining sonolysis or electrochemical treatment with other forms of adsorption-based



Figure 5. Changes in inorganic chlorine species chloride (Cl-), free chlorine (Cl2), chlorate (ClO3 -), and perchlorate (ClO4 -) over 10 h of electrochemical treatment at 50 milliAmps per centimeter squared (mA/cm²) in typical wastewater treatment effluent. Results are averages of duplicate data, and suggest a nearly 1:4 ratio of influence Cl- (1341.4 mg/L) to effluent ClO4 (4070.3 mg/L) (modified from Gomez-Ruiz et al. 2017).

and/or separation-based treatment as part of a treatment train has significant promise to offset the current reliance on expensive and unsustainable incineration. The sweet spot for these developing destruction-based technologies is low volume, concentrated waste streams where treatment residence time can be engineered to optimize energy consumption.

Staying Ahead of a Changing Landscape

The necessary reliance on conventional water treatment technologies to deal with PFAS has identified numerous opportunities for creative solutions, which will continue to evolve along with the stakeholder's collective understanding of the nature of the contaminants and the range of related compounds subject to regulation. For the time being, it is clear that PFAS-impacted water treatment will necessitate a treatment train approach that combines adsorption and separation technologies with destructive technologies.

On the bright side, exciting advancements, such as those described in this column, are paving the way for greater treatment efficiency, even in the more challenging area of PFAS destruction. Destructive technologies such as electrochemical treatment, plasma, and sonolysis show promise, but require careful attention to the challenges associated with large-scale application, such as secondary water quality effects, energy demand, and residence times. In addition, the development of technologies that support in situ treatment is often inspired by the solving of water treatment challenges in an ex situ setting. As practitioners, we can take this opportunity to work closely with leading industry and academic institutions to overcome the inherent challenges to these technologies and by doing so stay out ahead of the evolving landscape associated with PFAS!

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