EDITORIAL

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Themed issues on per- and polyfluoroalkyl substances

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Per- and polyfluoroalkyl substances (PFASs) are a large family of chemicals manufactured since the 1940s and researched with increasing intensity since the turn of the century.[1] Contamination associated with PFASs poses a grand challenge to environmental scientists due to their unique environmental chemistry, bioaccumulation behaviour and (eco)toxicology. Over the last 15+ years, research into the unique properties of PFASs and the risks these chemicals pose to the environment and human health has led to innovative approaches for chemical analysis, exposure and effects assessment, modelling, and remediation. In recognition of the large interest in research into PFASs, the Royal Society of Chemistry (RSC) decided to generate a pair of Themed Issues on PFASs. These themed issues appear respectively in the RSC sister journals: *Environmental Science: Processes & Impacts* (ESPI) and *Environmental Science: Water Research & Technology* (ESWRT). The ESPI Themed Issue focuses on

sources, occurrence, fate and transport, bioaccumulation, human exposure and effects of PFASs; whereas the ESWRT issue focuses on treatment, remediation, and management of PFASs.

In this editorial we, the guest editors, briefly summarize current PFAS research while referring to the novel contributions in these two themed issues. We are delighted that the contributing articles cover a broad spectrum of topics, as summarized below.

- Sources of PFASs.
- Emerging analytical methods for addressing the number and diversity of PFASs.
- Fate and transport of PFASs.
- Bioaccumulation of PFASs in wildlife.
- Human exposure pathways of PFASs.
- Effects of PFASs.
- Regulation of PFASs.
- Treatment technologies for PFASs.

Sources

As awareness of human and ecological exposures to PFASs grows, there is an increasing desire to determine all the various sources of PFASs to the environment and evaluate the exposure scenarios they create. While high concentration point-sources such as industrial sites[2] or sites where aqueous film-forming foam (AFFF) has been released[3] have been the focus of much research, given the developing regulatory environment focusing on low ng L^{-1} levels of many PFASs, the

more diffuse, non-point sources of PFASs are receiving increasing attention. This is particularly true in cities, where stormwater is increasingly viewed as a potential water source for thirsty cities facing greater climate uncertainties.[4] Thus, the work of Janousek *et al.* (DOI: 10.1039/C9EM00091G) documents the presence of PFASs, including long-chain perfluoroalkyl acids (PFAAs) such as perfluorooctanoic acid (PFOA) and its precursors, in building materials, fabrics, and coatings often present in cities and marine environments. Importantly, this research shows that not only are some of these long-chain PFAAs still being used in products, but they can leach from these products into an aqueous matrix. Clearly, this has important ramifications with respect to efforts to manage or eliminate releases of long-chain PFAAs to aquatic environments. Of course, even if these products do not release PFASs during their useful lives in the marketplace, they still may potentially leach PFASs when disposed in landfills. Interestingly, Knutsen *et al.*'s efforts (DOI: 10.1039/C9EM00170K) to characterize PFAS emissions from Norwegian landfills and compare their profiles to historic landfill leachates suggest that the general shift to shorter-chain PFAS chemistries may already be evident in landfills. In light of the difficulty in removing short-chain PFAAs from water (see discussion below) and the general difficulty in treating

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landfill leachates,[5–7] this study highlights the potential growing challenge of controlling current and future emissions of PFASs from landfills.

Emerging analytical methods for addressing the number and diversity of PFASs

The Organisation for Economic Co-operation and Development (OECD) has estimated that over 4700 PFAS-related CAS numbers exist worldwide. This list covers a wide variety of monomeric and polymeric substances, including cationic, anionic and neutral PFASs.[1] The large number and diversity of structures has necessitated development of novel analytical approaches such as the total oxidizable precursor (TOP) assay,[8] total and extractable organofluorine approaches,[9–12] and non-target/suspect screening methodologies.[2,13,14] These techniques are being increasingly applied by the scientific community to generate new knowledge about PFAS contamination and behavior in the environment, and the themed issues of ESPI and ESWRT highlight several applications. For example, Koch *et al.* (DOI: 10.1039/C9EM00281B) demonstrated that the use of isomer profiles and extractable organofluorine (EOF) can improve our understanding and characterization of PFAS point sources. Consistent with other studies,[9,15] a large fraction (>91%) of EOF in water from one site was not accounted for by target PFASs. Questions remain surrounding the identity of this missing fraction. Short-chain PFAAs such as trifluoroacetic acid (TFAA), trifluoromethane sulfonic acid, and hexafluoropropylene oxide dimer acid (HFPO-DA; Gen-X) are one possibility, but these substances are challenging to measure using conventional analytical approaches. Two further studies address this issue by using novel, stand-alone methods specifically designed for short-chain PFAAs. Vughs *et al.* (DOI: 10.1039/C9EM00393B) use a zwitterionic chromatography column together with orbi-trap mass spectrometry with optimized source conditions to capture trifluoromethane sulfonic acid, while Janda *et al.* (DOI: 10.1039/C9EM00169G) applied ion chromatography-mass spectrometry to

measure TFAA, perfluoropropionic acid (PFPrA) and perfluorobutanoic acid (PFBA). Collectively these studies highlight the need for multi-platform analytical approaches to capture the full range of PFASs which exist in the environment.

The TOP assay has also become increasingly popular due to its potential to convert both known and unknown PFAA-precursors to PFAAs. The application of the TOP assay to a soil core by Janda *et al.* (DOI: 10.1039/C9EM00169G) demonstrated that precursors to long-chain PFAAs such as legacy fluorinated paper chemicals or their breakdown products persist in the upper soil horizons (*i.e.* >40 cm) over long time periods, while ultra short-chain breakdown products (*i.e.* C2- and C3-perfluoroalkyl carboxylic acids (PFCAs)) can be transported to deeper layers. In another application, Simonnet-Laprade *et al.* (DOI: 10.1039/C9EM00322C) applied the TOP assay for the first time to sediment and biota, revealing the presence of substantial proportions of extractable unknown PFAA-precursors in all samples (*i.e.* 15–80% of PFASs upon oxidation). The authors highlighted the need to identify the structure of the unattributed PFAA precursors using techniques such as high-resolution mass spectrometry.

As target lists increase, it is important to consider the analytical challenges associated with PFAS analysis. As highlighted by Point *et al.* (DOI: 10.1039/C9EW00765B), PFASs may be subject to significant sorption during sample preparation due to the choice of solvent and container. Clearly, the development of new authentic and isotopically labelled standards is required to mitigate these analytical artifacts.

Finally, with the discovery of an ever-growing number of PFASs, naming of individual PFASs must be revisited. Indeed, the naming system developed by Buck *et al.*[16] was only designed for simple molecular structures. In an attempt to tackle the growing number of diverse PFASs, Sha *et al.* (DOI: 10.1039/C9EM00321E) explore how a combination of expert knowledge and chem-informatic approaches could help address this PFAS nomenclature in a systematic manner. Two chem-informatics approaches (splitPFAS and

ClassyFire) were evaluated to explore the potential of using such automated, open tools to enable stakeholders to systematically categorize and name PFASs.

Fate and transport of PFASs

While the global circulation of PFASs in the oceans[17] and atmosphere[18] has been long-recognized, the ESPI themed issue includes an important effort to evaluate the vertical transport of PFASs in the open ocean. By evaluating surface samples and samples from the deep chlorophyll maximum (DCM) in the Atlantic, Indian and Pacific oceans, González-Gaya *et al.* (DOI: 10.1039/C9EM00266A) ultimately estimated that mean residence times of perfluorooctane sulfonate (PFOS) and PFOA in the mixed layer are 360 and 32 years, respectively, though they note additional sinking flux processes not evaluated may contribute to additional vertical flux of these compounds.

Bioaccumulation of PFASs in wildlife

PFASs have been ubiquitously detected in terrestrial, freshwater and marine wildlife on a global scale.[19–21] The bioaccumulation of PFASs is compound-specific and depends on their perfluoroalkyl chain length and their functional group[22] as shown by Remucal (DOI: 10.1039/C9EM00265K) for herring gull (*Larus argentatus*) eggs from the Great Lakes. The highest bioaccumulation potentials have been found for long-chain PFAAs such as PFOS as evaluated in several studies in the themed issue (DOI: 10.1039/C9EM00322C; DOI: 10.1039/C9EM00265K). The themed issue contains a study on the biomagnification of PFAAs in the freshwater food web in the Orge River, France, and showed the biotransformation of PFAA precursors to PFAAs through the food web (DOI: 10.1039/C9EM00322C). Furthermore, the PFAS isomer profile can change during the bioaccumulation process due to the general higher excretion rate of branched PFAAs.[23] Another study (DOI: 10.1039/C9EM00202B) examined the spatiotemporal variability of PFAS concentrations in crustaceans (Eastern School Prawn, *Metapenaeus macleayi*) in a river estuary in Australia. Taylor (DOI: 10.1039/

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C9EM00202B) demonstrated how complex biological, ecological and physical factors influence tissue concentrations in aquatic species. They also applied a model to demonstrate that migration patterns of the crustaceans could explain the contaminant levels. In a companion study, Taylor et al. (DOI: 10.1039/C9EM00394K) additionally investigated the relationship between PFAA concentrations and endogenous metabolites in the same crustacean species. PFAA profiles were mirrored by significant differences in the profiles of both fatty acids and amino acids, indicating a potential association between PFAA concentrations and the composition of metabolites in the crustaceans.

Most studies have focused on the bioaccumulation and biomagnification of PFASs in freshwater and marine wildlife, whereas few studies have reported the accumulation of PFASs in the terrestrial environment.[21] For example, the highest PFAS concentrations were found in marine mammals such as polar bears (*Ursus maritimus*).[21] In general, PFASs are not accumulating in fat tissue as classical hydrophobic persistent organic pollutants (POPs), but mainly in liver, blood and other organs.[24] This can be explained by the fact that PFASs bind to the transport protein serum albumin, as highlighted in one study in the themed issue by Allendorf et al. (DOI: 10.1039/C9EM00290A) on the albumin/water partitioning coefficient for PFAAs and four alternative PFASs.

Human exposure pathways of PFASs

Humans in the general population are continuously exposed to PFAAs and their precursors *via* several pathways: the inhalation of air; the ingestion of dust, beverages, drinking water and food; and although essentially unexamined, through dermal absorption (*i.e.*, after contact with consumer products).[25] Whereas the exposure of adults to PFAAs and their precursors is becoming better understood,[26] childhood exposure studies are lacking. Understanding exposure in early life is important for refining risk assessments in light of the reported associations between health outcomes and prenatal and childhood

exposure.[27] In the ESPI themed issue, Balk et al. (DOI: 10.1039/C9EM00323A) aimed to reconstruct the serum concentrations of PFAAs in a cohort of children using a pharmacokinetic modelling approach. In early life, children receive prenatal exposure from their mother *via* placental transfer and then following birth receive a further maternal exposure through breastfeeding. Balk et al. (DOI: 10.1039/C9EM00323A) demonstrate that children's exposure to PFOA and PFOS after breastfeeding and with increasing age resembles the exposure of adults and occurs mainly through dietary ingestion. The modelling work also revealed that perfluorohexane sulfonate (PFHxS) exposure is poorly understood and there appears to be some unknown pathways of human exposure for this substance.

Effects of PFASs

The aquatic ecotoxicity of PFASs has been well-studied.[28–31] There is relatively little information on the terrestrial ecotoxicity of PFASs or on mixture toxicity, although research is ongoing. One interesting aspect of mixture toxicity is how PFASs can alter the response of organisms to other co-contaminants present in the environment. This issue is particularly relevant at sites that have been contaminated with AFFF given that in addition to many different PFASs, AFFF contains solvents, corrosion inhibitors, and hydrocarbon-based surfactants. Fitzgerald et al. (DOI: 10.1039/C9EM00241C) showed that PFASs and AFFF can alter the toxicity of a co-contaminant 2,4-dichlorophenol (DCP) that is known to partition into cell membranes and cause uncoupling. Furthermore, PFASs and AFFF were shown to inhibit degradation, decrease the number of methanogens present, and change the microbial community structure.

Owing to the structural similarity of PFASs to fatty acids, both are activators of peroxisome proliferator-activated receptors (PPARs). Activation of PPARs by PFASs provides a mechanism for some of the observed effects in many organisms. Information on one subtype of PPARs, PPARβ/δ, is relatively limited, although PPARβ/δ are known to play an essential role in many biological functions, such as

the regulation of lipid metabolism, cell proliferation, cell differentiation and cell inflammation (DOI: 10.1039/C9EM00218A). PFOS has been shown to exhibit agonistic activity towards human PPARβ/δ,[32] whereas two other studies showed that PFOS and PFOA had agonistic activity to mouse PPARβ/δ, but not human PPARβ/δ.[33,34] To further understand the toxicity mechanism, study of the binding and activity of PFASs toward PPARβ/δ is needed. In the ESPI themed issue, Li et al. (DOI: 10.1039/C9EM00218A) investigated the direct binding and transcriptional activity of PFASs towards human PPARβ/δ, and further revealed the structure–binding and structure–activity relationship between PFASs and PPARβ/δ. They showed that PFASs could bind to the PPARβ/δ receptor directly, with a chain length dependence, and showed an agonistic effect towards the PPARβ/δ signaling pathway.

Regulation of PFASs

A few PFASs have been investigated extensively and regulated, but for many other PFASs there is a lack of knowledge about uses and hazards. A group of more than 50 international scientists and regulators recently outlined new approaches for assessing and managing PFASs in the Zurich Statement on Future Actions on PFASs.[35] One policy initiative recommended in the Zurich Statement was to reduce, and eventually phase out, non-essential uses of PFASs. The ESPI themed issue includes an important critical review that addresses this initiative by Cousins et al. (DOI: 10.1039/C9EM00163H), who are a group of European and North American scientists and policy-makers. The critical review applies the concept of "essentiality" developed in the Montreal Protocol to PFASs. By articulating the sorts of uses that could be considered non-essential, substitutable, and essential, the authors developed a framework for regular assessment of whether the use of PFASs in many products is needed. Their effort to inform and encourage retailers to consider essentiality when considering PFASs for use in products and enable policy-makers to undertake data-driven assessments of

essentiality will be critical in regulatory efforts aimed to reduce releases of PFASs and/or exposures to PFASs. This timely work is reflective of the growing efforts by many retailers to seriously reconsider the use of PFASs in their products.

Treatment technologies for PFASs

As regulation and concerns about PFASs have grown, a host of technologies have emerged, and continue to emerge, to treat PFAS-contaminated drinking water, groundwater, wastewater, soil, and sediment. There is a particularly high demand of remediation technologies for PFASs at fire training facilities due to the use of PFAS-containing AFFF, landfills, and PFAS manufacturing and processing facilities. Furthermore, treatment technologies for PFASs are needed for PFAS-contaminated wastewater, landfill leachate, and drinking water. Treatment approaches using immobilization (e.g. granular activated carbon (GAC), anion exchange (AIX), stabilization and solidification), separation (e.g. nanofiltration, reverse osmosis), and destruction (e.g. electrochemical, sonochemical) of PFAS are described in some detail in recent reviews.[36-38] In the ESWRT issue, Wei et al. (DOI: 10.1039/C9EW00645A) add to these reviews with a focus on treatment of landfill leachate.

In the ESWRT themed issue, Liu et al. (DOI: 10.1039/C9EW00349E) provide an example of efforts to improve efficiencies of separation and immobilization through the use of novel media or modifications to commonly used media. Their study focused on improved efficiency through pilot-scale study and modeling work, particularly evaluating GAC's chain-length dependent breakthrough. In particular, the short-chain PFAAs are challenging to treat with GAC due to their high mobility. In another example in this themed issue, Aly et al. (DOI: 10.1039/C9EW00426B) introduce commercially available coagulants to soil/water systems to enhance absorption of PFASs by soil, thus reducing their impact on groundwater. In their initial batch studies, they were able to enhance PFAS sorption capacity of the soil by up to six times, which was then validated in flow-through column tests. Stebel et al. (DOI: 10.1039/

C9EW00364A) introduce a swellable organically modified silica which significantly outperformed GAC in PFAS sorption evaluations. The column experiments showed a high sorption capacity for even the short-chain PFAAs.

An example of a destructive approach for treating PFASs is highlighted in Franke et al. in the ESWRT issue. Franke et al. (DOI: 10.1039/C9EW00339H) describe treatment of PFASs using ozonation coupled with persulfate with removal efficiency dependent on PFAS chain length. For this approach, the shorter chain compounds (< C7) had the slowest/least extensive treatment.

Other approaches to improving treatment of PFASs involve combining technologies toward greater efficiency. Franke et al. (DOI: 10.1039/C9EW00286C) provide an example of such an approach to improve the efficiency of GAC and AIX treatment by integrating nanofiltration. Nanofiltration was first used to treat drinking water plant feedwater, then GAC and AIX were used, and compared, to address the concentrated retentate, resulting in increased overall efficiency in the use of GAC and AIX resin. Another combined technology is demonstrated by Dai et al. (DOI: 10.1039/C9EW00701F), who combined a separation approach (foam fractionation) with a destructive treatment approach (UV/ozonation). This combined approach outperformed each individual approach, but the removal of short-chain PFAAs was less efficient in comparison to the long-chain PFAAs, similar to the findings of Franke et al. (DOI: 10.1039/C9EW00339H).

The themed issues in ESPI and ESWRT highlight the broad scope of research and state-of-the-art tools used to tackle the challenging problem of PFAS contamination in the environment. While these advances show great promise, there remains an ongoing need for improved oversight of the PFAS manufacturing industry, in particular with regards to replacement substances. This includes regulation of both current and newly developed PFAS, but also safe management of PFASs over the course of their use. We hope that readers enjoy these articles and we would like to thank all contributing authors, peer reviewers,

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