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REVIEW

Chemistry, abundance, detection and treatment of per- and polyfluoroalkyl substances in water: a review

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Abstract

Per- and polyfluoroalkyl substances (PFAS) encompass a wide range of compounds containing carbon–fluorine bonds. Due the strength of this bond and the high electronegativity of fluorine atoms, PFAS display stability, wettability and other characteristics that are unique for industrial applications and products. However, PFAS induce adverse effects on the environment and human health. Here we review the chemistry, synthesis, properties, analysis, occurrence in water, filtration, removal and oxydation of PFAS. We highlight emerging hybrid treatments to remove PFAS from water.

Keywords PFAS · Synthesis · Analytical · Hybrid treatment · Health · Environment

Introduction

Per- and polyfluoroalkyl substances (PFAS) compounds and their salts have a broad variety of applications in both consumer products and industrial processes (Lewis et al. 2020; Pourehie and Saien 2020). In many cases, PFAS are still being applied as polymerization agents in fluoropolymer materials production (Ateia et al. 2019). Furthermore, PFAS are being used as surface treatment agents, surfactants, and for the fabrication of side-chain fluorinated compounds. These materials are common in carpet, textiles, fire-fighting foams, and paper industries (Dai et al. 2019; Fang et al. 2019). However, extensive evidence suggests their adverse environmental impact and health hazards (Kallenborn 2004; Dong et al. 2017; Holmquist et al. 2018). Reports indicate that the consumption of these materials can cause damage to the endocrine system (change of thyroid hormones, exposure > 0.01 g/L) (Wielsøe et al. 2015), liver (exposure > 5 mg/l) (Crebelli et al. 2019), kidney (exposure > 4.8 µg/L) (Blake et al. 2018), and fetal exposure can lead to growth stunting and IQ loss (exposure > 3.5 ng/mL) (Gabbert 2018), etc. These are only a few damaging effects that PFAS can cause which is why they have been included on the registration, evaluation, authorization, and restriction of chemicals (REACH) agency candidate list as substances of very high concern (Rosenmai et al. 2016).

Although PFAS and their adverse effects are often referred to in broad terms, it is crucial to note that there are nearly 5000 different PFAS in use by numerous manufacturing facilities, confirmed by the United States Food and Drug Administration (Ginsberg et al. 2019; Levine 2019). Most research and regulatory attention have been focused on two PFAS family members: perfluorooctanesulfonic acid (PFOS)

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and perfluorooctanoic acid (PFOA). In 2016, the United States environmental protection agency released the lifetime health advisory levels of 70 ng/L for the total concentration of PFOS and PFOA in drinking water; such a low threshold indicates substantial public health concerns (Pontius 2019). However, some states in the USA like California, Vermont, and Minnesota adopted levels of 13, 20, 27 ng/L for PFOS and 14, 20, 35 ng/L for PFOA, respectively (Pontius 2019). While regulatory guidelines and limits vary across countries, published values for PFOA and PFOS concentration are consistently among the lowest for any chemical compound (Pontius 2019). Since the start of the twenty-first century, many manufacturers in North America and Europe were forced by regulators to decrease the production of several long-chain PFAS (Wang et al. 2014; Land et al. 2018). While PFAS production in wealthy countries has declined, the consistent increase in the production of these compounds in developing countries and limited accessible data still pose great concern (PFASS and QUO; Zolghadr 2016).

The goal of regulatory measures and risk management strategies is to ensure that the problems associated with the uptake of PFAS can be controlled in the short and long term. However, the social and economic benefits and costs of PFAS must be evaluated fairly. Figure 1 illustrates the economic impacts, the assessment of substitution and the usage of PFAS. For example, the economic (direct and indirect) cost of low birth weight of infants caused by the PFAS was assessed to be $13.7 billion (US dollars) for the period 2003–2014 (Malits et al. 2018). This assessment includes the costs of hospitalization for medical care and concerns, the indirect cost of IQ loss, and the out-of-pocket expenditures of parental lost workdays (Malits et al. 2018). Figure 1 shows some estimates of the financial aspects of removing PFAS from consumer products and supply chains; these estimates imply that substituting or removing PFAS from all manufacturer production chains may not be feasible, at least in the short term. Therefore, it is clear that improved understanding of the legacy and outcomes of PFAS in our consumer products and in the environment is essential for preventing exposure and protecting human health. Nonetheless, as outlined herein, the patterns in the publication of scientific literature on PFAS treatment and remediation ("Hybrid treatment of per- and polyfluoroalkyl substances" section) in water resources reveals some gaps in our understanding of mitigation approaches for PFAS in water and little or no consideration of the extent to which economical PFAS substitutes may be available for particular applications.

While many reviews on the adverse environmental and health effects of PFAS have been published (Pelch et al. 2019; Sunderland et al. 2019; Fenton et al. 2020; Garg et al. 2020), this article is an attempt to fill a number of gaps in the understanding of environmental engineers, scientists, regulators and other stakeholders, working around issues associated with PFAS and seeking potential approaches to mitigate their adverse environmental and health effects. Our goal is to provide a nuanced perspective on the family of compounds that make up PFAS, their structure and physicochemical properties, the uses and economic impacts, and outline how the properties of PFAS make it unlikely that this family of compounds will be replaced in some industrial processes. We then address the strengths and weaknesses of the suite of analytical methods available for detecting PFAS in water samples, and emerging options for treatment with a particular focus on hybrid treatment approaches, including discussion of how not all PFAS compounds are equally susceptible to all treatment approaches.

Chemistry and synthesis of per- and polyfluoroalkyl substances

Perfluorinated compounds were first synthesized about 50 years ago (Fromme et al. 2010). PFAS are a class of anionic perfluorinated compounds, characterized by a perfluoroalkyl chain and a sulfonate or carboxylate solubilizing group. Several perfluorinated materials are utilized as precursor substances in preparing large molecular weight fluorinated polymers such as perfluoropolyethers (Carbone and Reinert 2015). PFAS are a class of perfluorinated compounds characterized as non-biodegradable, non-reactive, non-photolytic, and hydrolysis-resistant, making these materials recalcitrant in the environment (Ritscher et al. 2018). The fluoro-carbon portion of PFAS molecules is nonpolar while its tail segment is polar (Zeng et al. 2019). The carbon chain of perfluoroalkyl compounds are fully fluorinated and these tend to be more resistant in the environment in comparison to polyfluoroalkyl material which have carbon chains with some C-H bond between C-F bonds (Buck et al. 2011).

Thousands of PFAS produced for a variety of industrial applications (Fig. 2 illustrates the most common PFAS). Due to the large industrial demands, PFOS and PFOA are among the predominant chemicals in the PFAS family; they have physicochemical properties such as chemical and thermal inertness, low surface energy, low volatility and active surface sites that make them useful for numerous industrial applications (Vecitis et al. 2009; Sungur 2018). The existence of 17 and 15 C–F bonds in PFOS and PFOA, respectively, make them especially resistant to hydrolysis, photolysis, and microbial degradation (Parsons et al. 2008). Across the diverse range of PFAS, two synthesis methods are by far the most commonly used: electrochemical fluorination and telomerization (Pelch et al. 2019).

Electrochemical fluorination was the most common fabrication method for PFAS through the end of the 20th-century, but it has since been overtaken in popularity by

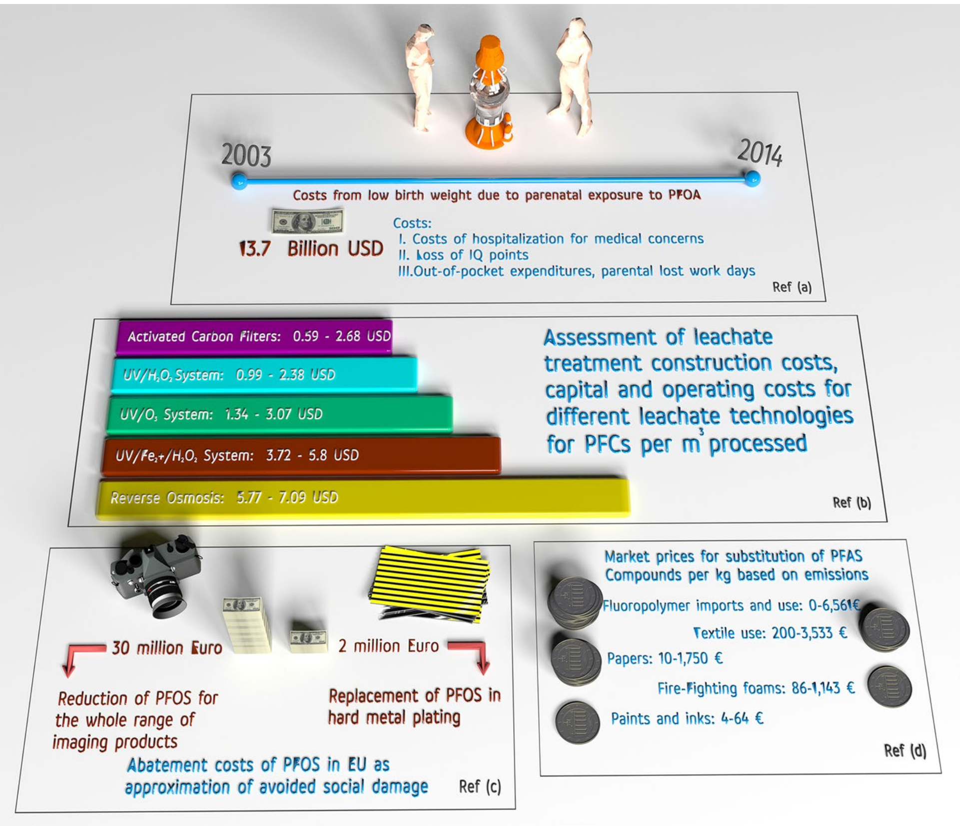

Fig. 1 Economic impacts of PFAS substitution and usage (PFAS: per- and polyfluoroalkyl substancesPer- and polyfluoroalkyl substances). **a** This panel shows the economic impacts of PFAS on a low birth weight as a result of PFAS consumption by parents. This includes the direct and indirect costs which sums up to total 13.7 billion US dollars cost between 2003 and 2014, **b** this panel shows different technologies costs for handing perfluorinated compound leachate. Among these technologies, reverse osmosis costs more than the others with 5.77–7.09 USD and activated carbon filter costs less with 0.59–2.68 USD per m^3. **c** This panel demonstrates the annual expenditure needed for the replacement of PFAS in hard metal plating (2 million Euro) and imaging products (30 million Euro). **d** This panel shows the potential increase in ongoing costs per kg of various industrial products were they to substitute alternative compounds for PFAS. The data source for Ref (a) (Malits et al. 2018), Ref (b) (Bernier 2014), Ref (c) (Greßmann et al. 2014), and Ref (d) (2014) are inserted next to them. Abbreviations are PFAS: Per- and polyfluoroalkyl substances, PFOA: Perfluorooctanoic acid, PFCs: perfluorochemicals, PFOS: Perfluorooctanesulfonic acid, UV: Ultraviolet–visible, EU: European Union, IQ: Intelligence quotient, and USD: United states dollar

telomerization (Järnberg et al. 2007). The electrochemical fluorination process is suitable particularly for carboxylic, ethers, and sulfonic acid derivatives. This is due to the low solubility of alkanes at the 0 °C temperature at which electrochemical fluorination takes place; electrochemical fluorination requires functionalized groups while the telomerization method involves free radical reactions so there is no need for functional groups (Grottenmuller 2002). The linear fraction of PFAS in the electrochemical fluorination methods usually falls between 70 and 0% of the compound while telomerization processes yield higher percentages. Also, in telomerization methods, PFAS have a non-fluorinated signature as a result of termination with alcohols (Eschauzier et al. 2012).

Abundance and detection of per- and polyfluoroalkyl substances

PFAS are produced in many industries and are relatively soluble in water, causing them to be widespread and common in many water bodies (Fig. 3). The largest pollution of PFAS in the environment happens due to their discharge by manufacturing facilities that produce aqueous film-forming foams (AFFF) (Hu et al. 2016; Dorrance et al. 2017). AFFFs need to be highly resistant to heat and grease, and therefore PFAS have typically been used to impart these properties (Dorrance et al. 2017). Their presence in both natural waters and wastewater and their recalcitrance to many conventional drinking water and wastewater treatment technologies provide opportunities for human exposure through drinking water, recreational water, irrigated

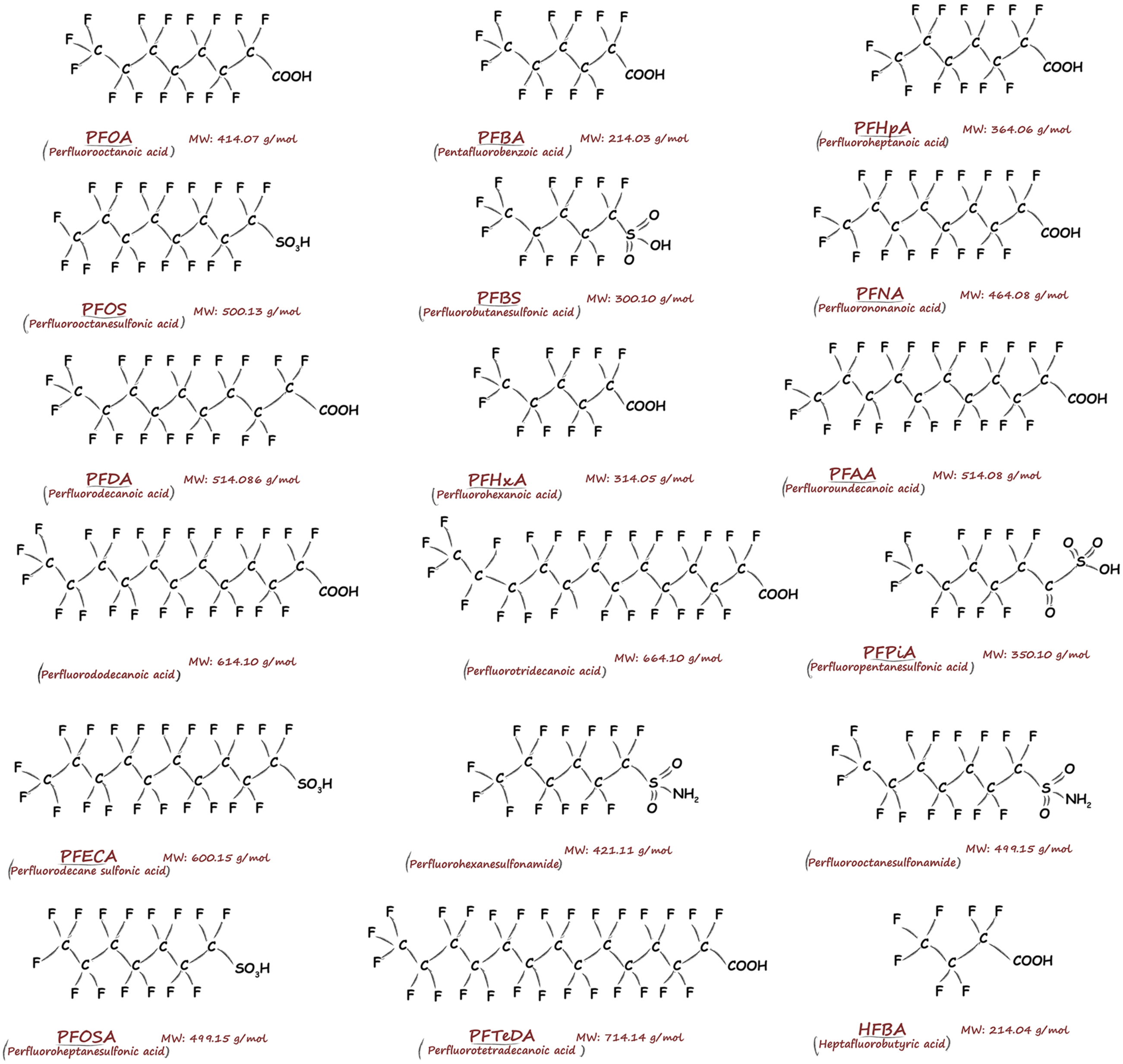

Fig. 2 Typical per- and polyfluoroalkyl substances (PFAS) and their molecular weight. Most of the PFAS molecular weight falls between 300 and 700 g/ mol. Among all these compounds, PFOA and PFOS are much more widely used and thus much more prone to be a potential contaminant in water. Abbreviations in the figure are PFOA: Perfluorooctanoic acid, PFBA: Pentafluorobenzoic acid, PFHpA: Perfluoroheptanoic acid, PFNA: Perfluorononanoic acid, PFAA: Perfluoroundecanoic acid, PFPiA: Perfluoropentanesulfonic acid, HFBA: Heptafluorobutyric acid, PFTeDA: Perfluorotetradecanoic acid, PFHxA: Perfluorohexanoic acid, PFBS: Perfluorobutanesulfonic acid, PFOS: Perfluorooctanesulfonic acid, PFDA: Perfluorodecanoic acid, PFECA: Perfluorodecane sulfonic acid, and PFOSA: Perfluoroheptanesulfonic acid

crops, etc. (Tittlemier et al. 2007; Landsteiner et al. 2014; Domingo and Nadal 2019; Ghisi et al. 2019). To evaluate potential human exposure to particular PFAS species and the associated health risks, it is necessary to choose an appropriate analytical method that can identify their abundance in surface water, coastal waters, groundwater, and drinking water (Sinclair and Kannan 2006; Hepburn et al. 2019).

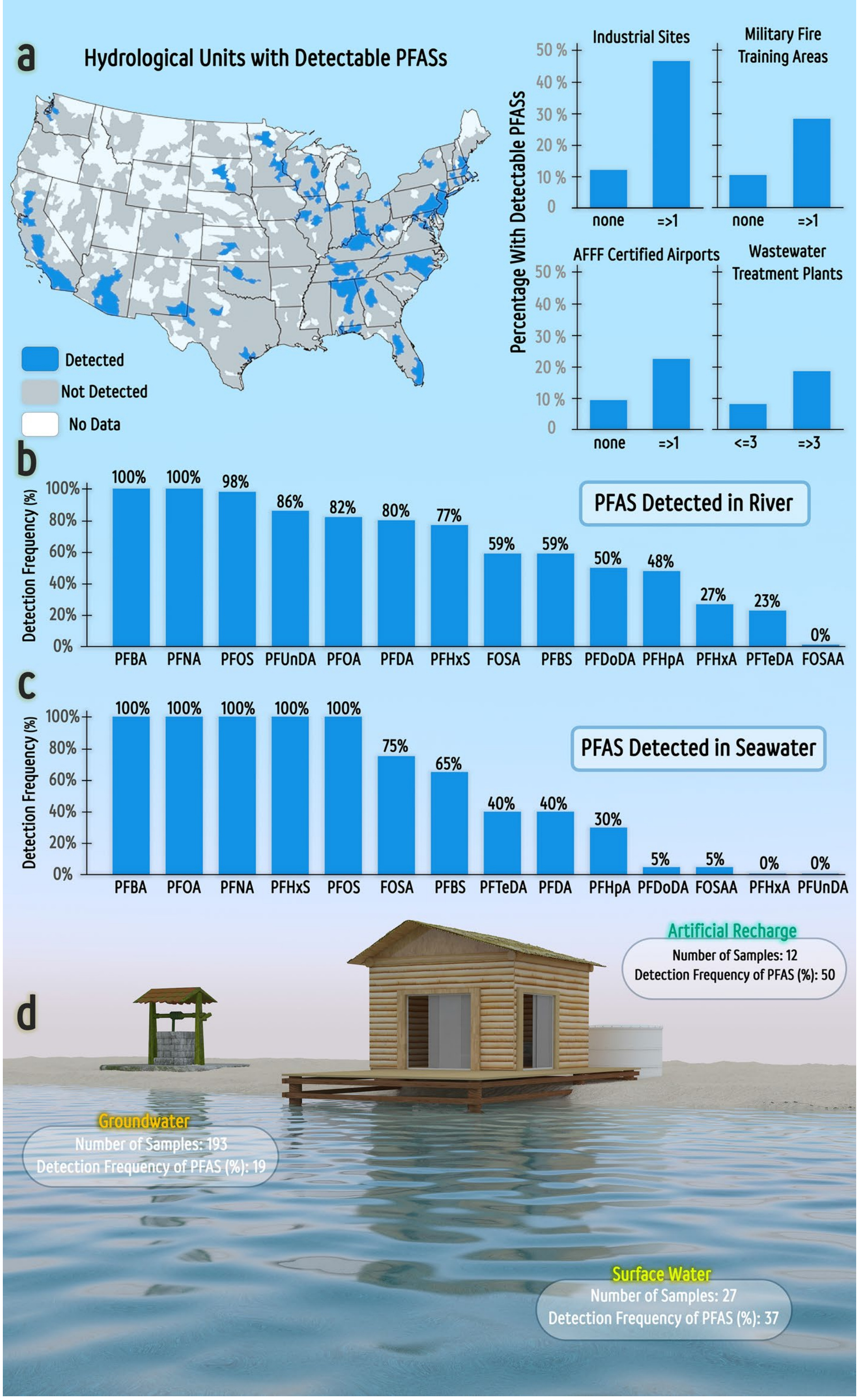

Fig. 3 **a** Locations inside the United States where PFAS detection has (blue) or has not (gray) been observed in water supplies (PFAS: per- and polyfluoroalkyl substancesPer- and polyfluoroalkyl substances); areas where no data are available are represented by white color (reproduced with permission from ACS (Hu et al. 2016)), **b** The detection frequency in different water sources, including surface water, groundwater, and artificial recharge are also shown (data source (Banzhaf et al. 2017)), **c** Graph bars show PFAS detection frequency in river and seawater samples, based on 40 samples for river and 18 samples for seawater (data source (Nguyen et al. 2017)). **d** Schematic graphic of three different PFAS abundant locations. Here, conventional wells represent groundwater, surface water along with river/sea and artificial recharge containers which are contaminated with PFAS (data source (Banzhaf et al. 2017)). Abbreviations are AFFF: Aqueous film-forming foams, PFAS: Per- and Polyfluoroalkyl substances, PFBA: Pentafluorobenzoic acid, PFNA: Perfluorononanoic acid, PFOS: Perfluorooctanesulfonic acid, PFUnDA: Perfluoroundecanoic acid, PFOA: Perfluorooctanoic acid, PFDA: Perfluorodecanoic acid, PFHxS: Perfluorohexanesulfonic acid, FOSA: Perfluoroheptanesulfonic acid, PFBS: Perfluorobutanesulfonic acid, PFDoDA: Perfluorododecanoic acid, PFHpA: Perfluoroheptanoic acid, PFHxA: Perfluorohexanoic acid, PFTeDA: Perfluorotetradecanoic acid, and FOSAA: Perfluorinated sulfonamidoacetic acid. Reprinted with permission of American Chemical Society from (Hu et al. 2016)

Abundance of per- and polyfluoroalkyl substances in water

Since the addition of PFAS to the unregulated contaminant monitoring rules (UCMR), water supplies for nearly 6 million U.S. residents have been flagged for concentrations of PFAS in these resources higher than the 70 ppt threshold established by the environmental protection agency (Hu et al. 2016; Dorrance et al. 2017). Because PFAS have unique molecular fingerprints, chemical fingerprinting is reported to be a suitable forensic method for the detection of these compounds (Dorrance et al. 2017). Chemical fingerprinting is a methodology that applies analytical chemistry to identify the origins of complex environmental contaminants; it is based on the fact that particular chemical sources have characteristic distributions of individual segments of the chemical structure (Boehm et al. 1997; Douglas et al. 2007; Headley et al. 2013). Point sources are where the PFAS contamination originates, primarily discharges from manufacturing facilities. There are also reports(Hu et al. 2011) of the existence of PFOS (532 ng/L) and PFOA (1060 ng/L) in effluent from wastewater treatment plants (WWTPs) that discharge into urban water resources or facilities that have been closed but their contaminants were washed or infiltrated in groundwater. The detection of a sudden or gradual increase in the concentration of PFAS-related compounds suggests a potential PFAS point source or multiple sources discharging to surface water or the arrival/mobilization of previously sequestered PFAS compounds (e.g., from disturbed sediments or in groundwater base flow) (Sinclair and Kannan 2006; Dorrance et al. 2017). Because PFAS are being used in industry and military, it may be feasible to utilize chemical fingerprinting of the wastewater of these sectors to determine the initial point source discharge of PFAS that make their way to WWTPs (Houtz et al. 2016).

A number of large surveys of PFAS contamination in water resources of the US and Europe have been undertaken in recent years. A study (Hu et al. 2016) conducted on the point source analysis of PFAS, reports on more than 8500 WWTPs, 16 industrial sites, nearly 300 military fire training areas, and more than 500 civilian airports (AFFF-certified airports). It found that production of PFAS varies widely among different sites and the statistical analysis (Hu et al. 2016) also yielded insight into how the higher concentration of PFAS in drinking water is correlated with the number of point sources within a watershed. Figure 3a shows locations within the US where PFAS abundance has been investigated (Hu et al. 2016; Dorrance et al. 2017). These studies (Hu et al. 2016; Dorrance et al. 2017) demonstrate that 4% of public water supplies contain above or close to the environmental protection agency acceptable levels of PFAS concentration. These resources serve 6 million residents in more than 30 US states, and three American territories. The reported concentration values are as high as 1800 ng/L and 349 ng/L for PFOS and PFOA, respectively (Hu et al. 2016). Furthermore, a number of studies have shown that the long-chain PFAS, including PFOA and PFOS, are detected more often in groundwater, and the short-chain PFAS are detected more often in surface water (Taniyasu et al. 2008; Li et al. 2010; Buck et al. 2011; Hu et al. 2016). A similar study on PFAS contamination on potable water resources, accounting for the water consumption of ~ 4 million in Sweden, shows that 22% of the collected samples contain detectable amounts of PFAS (Holmström et al. 2014; Banzhaf et al. 2017). In contrast to what has been observed in the US in the aforementioned study (Hu et al. 2016), this dataset from Sweden shows that the detection frequency in samples collected from groundwater is less than those in surface water. But similarly, the most frequently detected PFAS are PFOS and PFOA. Overall, around 50% of targeted PFAS were found in samples; this includes 13 and 12 out of 26 detectable PFAS in river and seawater samples, respectively (Nguyen et al. 2017). The corresponding numbers are presented in Fig. 3b (Nguyen et al. 2017). Figure 3c also shows the statistics associated with the detection frequency in different water sources (Holmström et al. 2014; Banzhaf et al. 2017). Another study targeted 26 PFAS in northern Europe, investigating samples from the Baltic Sea, Kattegat, and several Swedish rivers (Nguyen et al. 2017). The results indicate that 10 rivers contain more than 4 ng/L of PFAS (Nguyen et al. 2017). It is also shown that there is a correlation between two broadly used PFAS species (PFOS and PFOA) and population density (Nguyen et al. 2017).

Analytical methods for detection of per- and polyfluoroalkyl substances in water

To fully characterize the water samples and confirm the existence and concentration of the PFAS, many analytical techniques have been employed (Nakayama et al. 2019). High-performance liquid chromatography (HPLC) coupled with tandem mass spectrometry (MS/MS) is known as a primary detection and concentration determination technique for PFAS and has been standardized by US environmental protection agency (Hansen et al. 2002; Higgins et al. 2005; Poothong et al. 2012; Ciccotelli et al. 2016; Schaefer et al. 2017). In HPLC, a 250 mL water sample is fortified with surrogates and passed through a solid-phase extraction cartridge. The solid-phase extraction cartridge contains polystyrene-divinylbenzene to better separate the PFAS compound from the media. Usually, a small amount of methanol is utilized to desorb PFAS from the existing solid-phase sorbent in the sample to accurately measure all present PFAS (Lein et al. 2008; EPA 2018). Using internal standard techniques, the concentration of PFAS can be determined.

Particle-induced gamma-ray emission (PIGE) spectroscopy is a method developed for the quantification of elemental fluorine (Ritter et al. 2017). PIGE is a non-destructive and quick surface analysis technique (Langton et al. 2020). PIGE has been widely used for medical and biological applications, but only recently it has been suggested for the measurements of PFAS-containing samples (Butzen et al. 2020). In this technique, the surface of the sample is struck by an accelerated beam of protons which leads to the excitation of fluorine nuclei. Subsequently, due to the de-excitation, gamma rays will be emitted. These gamma rays will produce a specific intensity proportional to the number of fluorine atoms on the surface. For measuring total fluorine, PIGE is advantageous because it has good sensitivity in the range of 13–45 nmols of fluorine per cm^2 (Ritter et al. 2017; McDonough et al. 2019). Figure 4a, b presents typical PIGE spectra of PFAS polluted water, and Fig. 4c shows a typical schematic of PFAS sample preparation. PIGE has a number of disadvantages for use in water samples: it has limited depth of penetration, with the maximum of 250 µm, depending on the energy of bombarding protons (Ritter et al. 2017; McDonough et al. 2019) and the detection resolution is on the order of 1.0 µg/L, whereas the regulatory standards resolution are often set with precision of 1.0 ng/L (Kotthoff and Bücking 2018; Reade et al. 2019a). Perhaps most important for analysis of water samples in the U.S., PIGE only measures the concentration of atomic fluorine in water; therefore, it cannot be used with drinking water that is fluoridated.

The adsorbable organic fluorine assay is another technique that can measure organic fluorine concentration based on combustion ion chromatography (Wagner et al. 2013). In this method, organic fluorine, in addition to any residual inorganic fluorine in the sample, is converted to hydrofluoric acid by combustion between 900 and 1000 °C (Miyake et al. 2007a). Hydrofluoric acid will be absorbed into a solution of sodium hydroxide, then the total concentration of fluoride ions can be measured through ion chromatography (Itota et al. 2004). The extractable organic fluorine assay, also referred to as total organo-fluorine-combustion ion chromatography (TOF-CIC), is similar to absorbable organic fluorine. The terms extractable organic fluorine and TOF-CIC refer broadly to techniques in which the organic fluorine content is isolated by ion-pairing methods and total fluorine is measured by combustion ion chromatography (McDonough et al. 2019). Usually, for water, fluoride and other impurities are removed by weak anion exchange solid phase extraction before starting combustion ion chromatography analysis to distinguish between fluoride and organic fluorine (Miyake et al. 2007a). Absorbable organic fluorine and extractable organic fluorine do not offer any structural details about the detected PFAS (Cousins et al. 2020). Another disadvantage of absorbable organic fluorine/ extractable organic fluorine is that the detection limits are on the order of 1.0 µg/L (Willach et al. 2016; Kotthoff and Bücking 2018).

The total oxidizable precursor assay is a useful method for the detection of PFAS when the target PFAS are known (Casson and Chiang 2018). This method is applicable only for compounds that can be oxidized to form targeted PFAAs. (Zhang et al. 2019) A major advantage of total oxidizable precursor for PFOS and PFOA is a detection limit on the order of 1.0 ng/L (Houtz and Sedlak 2012; Kotthoff and Bücking 2018). However, the total oxidizable precursor approach can only detect PFAS species that are already

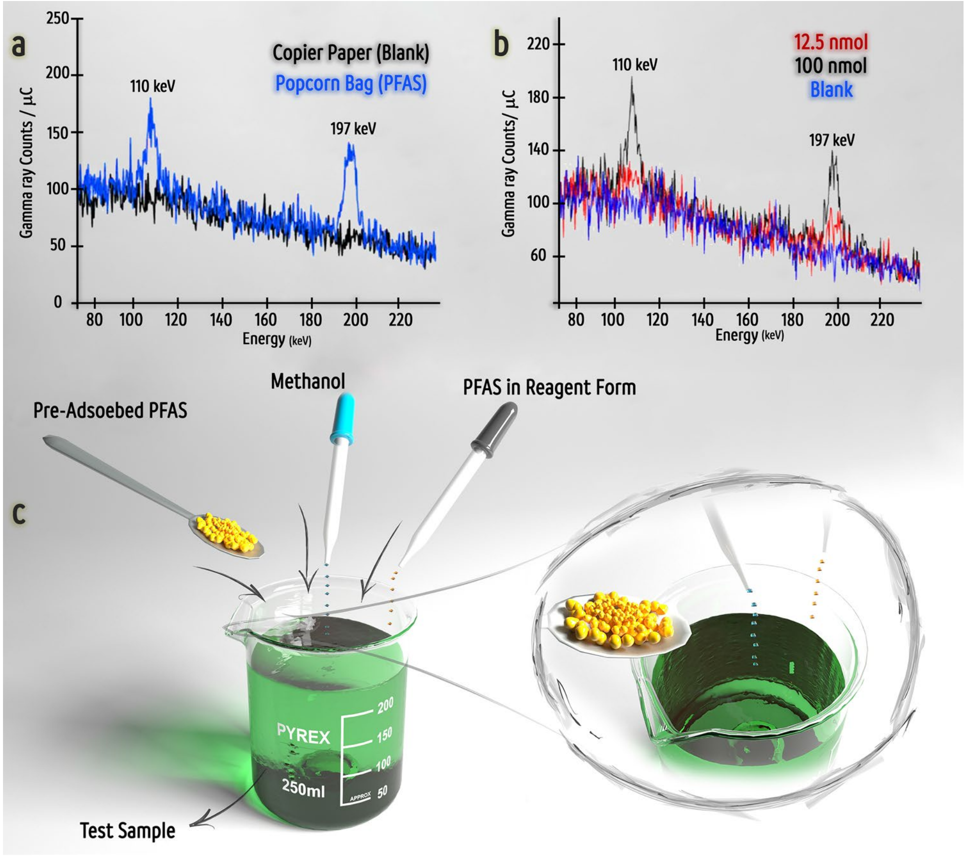

Fig. 4 **a** Comparing the gamma-ray spectra of a PFAS-treated popcorn bag (blue line) with blank copier paper (black line) resulted from a typical PIGE measurement. Fluorine gamma-rays at 110 keV and 197 keV are detected for popcorn bag, **b** PIGE spectra of PFOA extracted on the surface of WAX cartridges. Three samples are 0 (blue line), 12.5 (red line), and 100 nmol (black line) PFOA on WAX cartridges. The two gamma-rays are observed at 110 keV and 197 keV due to excitations of fluorine nuclei (data source (Peaslee et al. 2019)). **c** A typical PFAS sample preparation. PFAS, per- and polyfluoroalkyl substances. Reprinted with permission of Elsevier from (Ritter et al. 2017). PFAS: per- and polyfluoroalkyl substances. PIGE: Particle-induced gamma-ray emissionParticle-induced gamma-ray emission

assumed to exist in the water sample (Martin et al. 2019). Therefore, any other precursors which oxidize to an unselected PFAS would be missed (Martin et al. 2019).

In conclusion, a variety of analytical methods have been applied for detecting PFAS, but they must be selected carefully based on the application, the medium, and the desired detection limit. Many of these methods are currently applicable not only to water but also to sediment and soil including total oxidizable precursor, (Michigan 2018) extractable organic fluorine, (Yeung et al. 2013) and nuclear magnetic resonance; (Oliver et al. 2020) some have been used for biological media (e.g., blood and tissue) including LC-MS/MS, (Oliver et al. 2020) PIGE, (Falandysz et al. 2012) and extractable organic fluorine; (Miyake et al. 2007b) some are also applicable to paper and textile including PIGE (Ritter et al. 2017) and total oxidizable precursor (Falandysz et al. 2012). However, absorbable organic fluorine is applicable only in water (Wagner et al. 2013). Additionally, methods that do not differentiate between atomic, organic and inorganic fluorine (e.g., PIGE, absorbable organic fluorine, and extractable organic fluorine) cannot be used in fluoridated drinking water, wastewaters from areas with fluoridation, or natural waters that may have contributions from the public water supply. Moreover, some methods' detection limits preclude their use for drinking water regulatory applications (including PIGE, nuclear magnetic resonance, absorbable organic fluorine, and extractable organic fluorine). Many known PFAS including PFOS and PFOA can be detected by LC-MS/MS (Kucharzyk et al. 2017).

Detection and quantification of specific PFAS compounds requires that analytical standards be available for that compound; to date, these standards are available for less than 100 relevant PFAS compounds out of ~ 5000 different PFAS (Liu et al. 2019b; McDonough et al. 2019). Several methods for measuring/characterizing organo-fluorine structures have been developed to quantify the unidentified PFAS in environmental samples. The techniques that are too inclusive, PIGE as an example, are not capable of distinguishing between organic and inorganic fluorine, and therefore are not practical for measuring PFAS-related organo-fluorines (Langton et al. 2020). Additionally, PIGE should not be used for any water samples that could be influenced by either fluoridated drinking water or municipal wastewater in areas with drinking water fluoridation. Typical municipal piped water is fluoridated at 1 mg/L, so even a small proportion of municipal wastewater or drinking water in a sample will cause a large systematic bias in measurements. Fluoridated municipal drinking water makes up most of the municipal wastewater in that case, and piped water supplies are typically used for watering lawns, washing cars and other activities that lead to runoff. In contrast, extractable organic fluorine is more exclusive toward detection of specific PFAS (McDonough et al. 2019). In general, regarding all characterization methods, another challenge is reaching sufficient detection limits, especially when working on natural water and drinking water samples. Total oxidizable precursor is a more sensitive technique than extractable organic fluorine and absorbable organic fluorine (Houtz and Sedlak 2012; Kotthoff and Bücking 2018). Meanwhile, extractable organic fluorine and absorbable organic fluorine are the most suited among the more inclusive surrogate methods (Miyake et al. 2007b; Kotthoff and Bücking 2018). PIGE typically requires preconcentration of large samples to achieve suitable detection limits (McDonough et al. 2019). To summarize, choosing between different techniques is a trade-off between selectivity, sensitivity and other practical and financial considerations. Table 1 summarizes the major advantages and limitations of some available analytical approaches to quantify PFAS (Michigan 2018; Reade et al. 2019b).

Hybrid treatment of per- and polyfluoroalkyl substances

In the last 20 years, various treatment methods for PFAS remediation have been tested including adsorption (Lin et al. 2015a; Milinovic et al. 2015; Zhang et al. 2021), oxidation (Niu et al. 2016; Gomez-Ruiz et al. 2017), filtration(Niu et al. 2016), thermal(Gu et al. 2016, 2020), and biological treatments (Vierke et al. 2012; Garg et al. 2021). Many of these technologies suffer from the need for substantial energy and chemical usage, costly operating conditions, and immobile treatment facilities (Stanifer et al. 2018; Sunderland et al. 2019). The ideal treatment approach for a particular situation can vary substantially depending on variables including cost, starting PFAS concentration, target concentration, treatment/residence time available, design life of the treatment facility, and the mix of particular PFAS species (e.g., short-chain or long-chain) that are present in the water to be treated (Ahmed et al. 2020). With the goal of understanding current and emerging treatment approaches for PFAS in water, Fig. 5 is prepared after surveying over 70 published studies on the remediation of PFAS from 2010 to 2020 (the resources for creating Fig. 5 can be found in supporting information Table S1). As shown in Fig. 5a, although there was an increase in the number of publications on PFAS remediation and treatment from 2010 to 2015, the rate of publication seems to have leveled off. Notably, publications on photocatalysis of PFAS were the most common (Fig. 5b), but most of these publications were between 2010 and 2013. Figure 5c–e illustrates the different treatment parameters of each mentioned process. Although the process efficiency of most of these methods is in an acceptable range, there are substantial differences (approximately 100-fold) between the treatment time and the values of PFAS initial concentration across techniques. Therefore, a cursory reading of the

Table 1 The comparison of different analytical methods for detecting per- and polyfluoroalkyl substances (PFAS) (Bartell et al. 2018)

Methods	Advantages	Disadvantages
Liquid Chromatography–Tandem Mass Spectrometry (LC-MS/MS) Method 537 V 1.1	Commercially available Quality control extensive Quantifies individual PFAS UCMR3/Method 537/SW-846 8327 and 8328/ ASTM based on instrument Differentiates branched/linear	Expensive Limited number of PFAS Value for forensics depends on number of PFAS evaluated
Total oxidizable precursor	Commercially available Quality control improving Some chain length and branched and linear isomer information Reveals the presence of significant precursors in AFFF-contaminated water, sediment, soil, and wastewater Data sets obtained by this methodology are comparable between sites and across states	Twice as expensive as LC-MS/MS No information on individual PFAS Conservative (lower) estimate Limited comparative data at this time Caution at low levels Limited value for forensics
LC-HRMS	Unlimited number of PFAS Stored data can be searched in future Value as a forensics tool	Instruments available but PFAS analysis by LC-HRMS not commercially available in the US (research tool) Expensive No standards for the other PFAS Data are ‘screening’ level or semi-quantitative Limited comparable data—data obtained on different instruments, rationing to various internal standards may not be comparable between sites and across states (generates laboratory-specific data until standardized)
PIGE	Quantifies Fluorine Currently, captures anionic PFAS, being adapted for cationic/zwitterionic PFAS Less expensive Available through only one academic laboratory that may have a commercial partner	Only quantifies total Fluorine (the atom) Cannot be used with fluoridated drinking water No information on individual PFAS Small database (few comparative data) Not as sensitive (yet) as LC-MS/MS or LC-HRMS Limited value for forensics
Absorbable organic fluorine	Total adsorbable Fluorine (what the title says) captures a broad spectrum of PFAS Can be compared to individual PFAS analysis to determine the presence of other PFAS (e.g., precursors)	Measures total Fluorine (the atom) Cannot be used with fluoridated drinking water No information on individual PFAS Not commercially available in the US (or elsewhere) Must convert total Fluorine in units of molar F to equivalents, assuming a specific PFAS to compare measurements Few comparable data

literature may give the false impression that these treatment technologies would yield similar PFAS removal performance in the context of a treatment train with a known residence time. Additionally, simply reporting treatment results for PFAS as a class of compounds provides inadequate detail on the removal of particular compounds; some treatment technologies are more effective for PFAS with smaller or larger chains. Another major factor that may not be apparent from an overview of removal performance is the need with many technologies to dispose of media, retentate or other waste in which the removed PFAS have been concentrated.

The performance of individual treatment technologies for PFAS have been covered at depth in the literature (Kucharzyk et al. 2017; Crone et al. 2019; Nzeribe et al. 2019; Mahinroosta and Senevirathna 2020). Notably, since 2015, a new trend has been followed for performing PFAS remediation by combining multiple treatments in one systematic protocol, called “hybrid treatments.” As discussed earlier in this section, for a system that could be able to fully address the removal of the PFAS, many factors are playing a role. For instance, one treatment in a short period of time may not be able to remove a desired amount of PFAS but it can be used to pre-treat the feed so that it is suitable for a subsequent treatment step that can achieve the target concentration. Combining treatment technologies using a hybrid approach can yield the

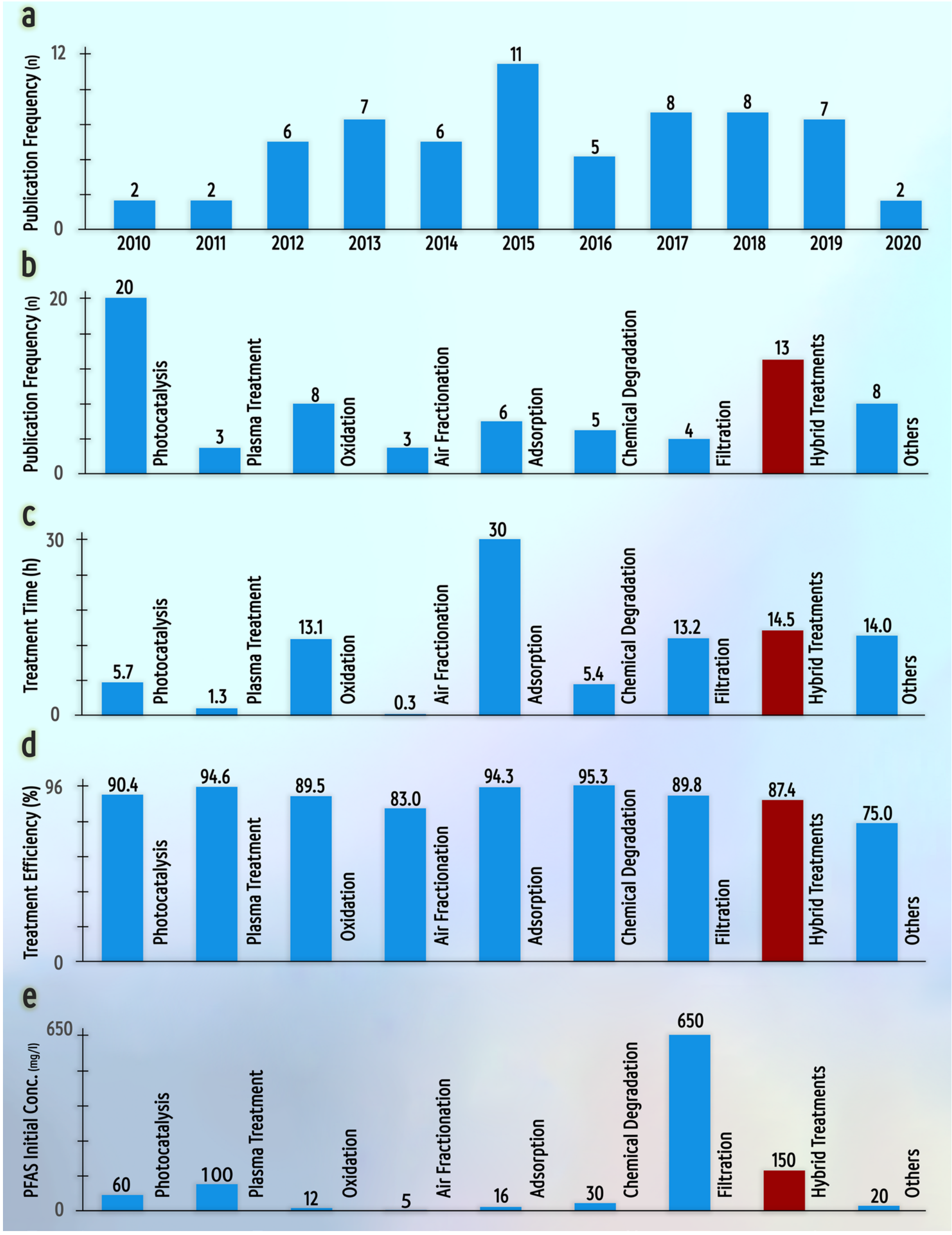

Fig. 5 **a** Publication trend over the last decade related to mitigation of per- and polyfluoroalkyl substancesPer- and polyfluoroalkyl substances (PFAS), **b** different treatments contribution in PFAS removal, **c** the average treatment time in selected studies, **d** the average process efficiency in selected studies, and **e** the average PFAS initial concentration in selected studies. Observed papers in "others" section have been referenced here (Lee et al. 2010, 2016; Vecitis et al. 2010; Appleman et al. 2014; Hori et al. 2015; Lin et al. 2015b; Park et al. 2016; Pourehie and Saien 2020). The resources for creating Fig. 5 can be found in supporting information Table S1. PFAS, per- and polyfluoroalkyl substances

desired removal while controlling cost, treatment time, and other relevant variables. Additionally, some treatment technologies are better for either short-chain or long-chain PFAS species. For these reasons, hybrid treatments have attracted much more attention in recent years; the rest of this section covers and discusses more about them. Hybrid treatments can be performed in a series of the treatments, or simultaneously.

Adsorption-based hybrid treatments

Adsorption processes are popular and widely used for treatment of various contaminants in water; advantages include its simplicity and low cost. Therefore, adsorption is one of the processes that is able to be combined with other processes in a hybrid treatment system. Adsorption usually takes place first in series in hybrid treatment to collect most of the PFAS and prepare it for second process in the treatment train. The main challenge with removal of PFAS with adsorption is the inability of the media to degrade PFAS. Adsorption processes typically concentrate the PFAS on a solid media that then requires another process to reactivate the adsorbents and degrade the collected PFAS, making PFAS adsorption a hybrid process. Adsorption processes are categorized into chemical and physical adsorptions. The main mechanism of chemical adsorption is chemical bonding with the acidic tail of the PFAS. This needs a proper population of functional groups on adsorbent surface to react with the PFAS (Firouzjaei et al. 2020a). On the other hand, physical adsorption is based on the electrostatic interaction between PFAS and adsorbent. However, because of large molecular size of the PFAS, physical adsorption is less prominent than chemical adsorption.

Adsorption–reduction is one of the noteworthy technologies taking a part in the removal of PFAS (Gagliano et al. 2020). The treatment takes place in a column packed with a series of adsorbents and reductants, with two processes working in tandem (Kucharzyk et al. 2017). For both of the adsorbents (e.g., biochar, ion exchange resins) and reductants (e.g., zero-valent iron), the temperature of the experiment is a vital feature impacting the treatment process (Guo et al. 2017). Also, the length of the PFAS carbon chain is an important factor for adsorption efficiency (Gagliano et al. 2020); the shorter the carbon chain is, the higher hydrophilicity and the lower adsorption efficiency will be (Gagliano et al. 2020). Media characteristics also have a strong effect on performance, media generated at higher pyrolytic temperatures typically have higher pore density, surface area, and adsorption–reduction rate, which can improve adsorption (Kupryianchyk et al. 2016). Also, the reduction in particle size is another important factor that can increase the removal rate (Zhou et al. 2010). As a downside, most of these adsorptive-reductive approaches suffer from (1) low potential for regeneration after saturation and/or (2) require a large amount of salt and organic solvents for regeneration at high temperatures (Vecitis et al. 2008; Zhang et al. 2016). Therefore, these types of adsorbents and reductants need another oxidation process for degradation of PFAS/PFOA for the regeneration of their active sites which can be assumed as a adsorption–reduction–oxidation hybrid method (Punyapalakul et al. 2013). The oxidation part can be accomplished through photocatalysis, high alkaline pH, heat, and ultrasound (Tsitonaki et al. 2010). Reactions 1 and 2 describe the regeneration of hydroxyl and sulfate radical sites:

$$S_2O_8^{-2} + 2e^{-} \rightarrow 2SO_4^{\cdot} \tag{1}$$

$$SO_4^{\cdot} + H_2O \rightarrow HO^{\cdot} + SO_4^{-2} + H^{+} \tag{2}$$

Another alternative for the oxidation step of this hybrid method is thermal mineralization. Thermal treatment of the pre-adsorbed PFAS usually happens at – 700 °C (Watanabe et al. 2018). While oxidation is typically much less expensive than thermal-mineralization, for some PFAS species, like PFOS, the degradation rate is as low as 20% due to the ineffectiveness of oxidation process (Park et al. 2016; Crimi et al. 2017). However, one downside of thermal mineralization is the potential for PFAS evaporation and escape through thermal detachment from the adsorbent (Kucharzyk et al. 2017; Watanabe et al. 2018). The main mechanism of the separation here is the difference in the boiling point of the pre-adsorbed-PFAS and solvent in column.

Along with conventional oxidation process, recently, electrochemical anodic oxidation attracted attention for the regeneration of PFAS-saturated adsorbents (Merino et al. 2016; Liang et al. 2018). For some adsorbents (especially ion exchange resins), the adsorption process can produce a highly concentrated layer of PFAS called "still bottom" (Liang et al. 2018). For the remediation of this highly saturated layer, electrochemical anodic oxidation can be effective when anode material possesses high conductivity, potential scalability, and durability (Schaefer et al. 2020). In this process, – 80% defluorination of PFOA and PFOS can be achieved (Schaefer et al. 2020). The defluorination occurs when the C–F bond breaks which is a signal of PFOA/PFOS complete mineralization but this still yields lots of organic solvents existing in the still bottom layer that needs to be extracted (Lu et al. 2020). The extraction of these compounds requires multiple distillation processes which increase the treatment time and energy consumption (Lu et al. 2020).

Filtration-based hybrid treatments

As mentioned above, it is generally infeasible to achieve high removal and degradation efficiency using only one method, so it is vital to treat the contaminants using a series of processes (Lu et al. 2020). As a suitable candidate for coupling, filtration is a fast and cost-effective method (Firouzjaei et al. 2018, 2020b; Rahimpour et al. 2018). Filtration would be able to increase the adsorption capacity of the whole treatment cycle, at the beginning or in the end (Horst et al. 2018). Membranes are widely used for size-based separation of unwanted species from the liquid phase (Pejman et al. 2020a, b, 2021; Seyedpour et al. 2020). The important factors for each membrane-based filtration are the PFAS structure and molecular size (Murray et al. 2019). Changes in solution pH can increase the repulsion between the membrane and PFAS by altering the electrical charge of the membrane surface which can affect the rejection efficiency (Murray et al. 2019). Using a nanofiltration (NF) set-up for the removal and concentration of PFOA can result in removal of more than 99% of PFOA, and can double the PFOA concentration in the feed tank (Boonya-atichart et al. 2018). Furthermore, a photocatalytic oxidation step can be used to treat retentate by UV source. The oxidation step can degrade up to 60% of PFOA in the retentate tank (Dombrowski et al. 2018). But another process like ultrafiltration (UF) may be necessary to remove the remaining PFOA before disposal. UF not only can take a part in the removal of the remaining PFOA but also will remove the oxidant agent in the second step. UF can be appropriate for groundwater treatment, particularly when a fast treatment time/short residence time is required. It is capable to remove up to 70% of PFOA in only one cycle. It is also feasible to treat the residual PFOA by multiple similar cycles due to the rapid nature of UF (Boonya-atichart et al. 2018).

In situ hybrid treatments

In situ hybrid treatments utilize multiple simultaneous treatment processes to improve the degradation and removal efficiency of the PFAS. For in situ hybrid system, a combination of technologies is employed to activate and use free radical reactions for PFAS degradation. These in situ treatments include sonochemical oxidation, electrochemical oxidation, and plasma-based degradation. Unlike the other treatment protocols, in situ treatments has no need for extreme operation conditions, has a lower need for chemical and energy consumption, and it is not suffering from secondary by-product creation while it keeps the removal and degradation efficiency high (Trojanowicz et al. 2018; Singh et al. 2019).

The use of simultaneous electrochemical and electro-Fenton oxidation at the same time is one way for activation of radical-based treatment (Lu et al. 2020). The availability of hydroxyl radical groups is the main feature of the electro-Fenton process (Lu et al. 2020). PFAS will be degraded in the electro-Fenton process and the electrochemical process provides the in situ hydroxyl radicals for the electro-Fenton process. The higher rate of oxygen reduction in the electrochemical process increases the selectivity of the electro-Fenton process (Wang et al. 2019). Reactions 3–5 simplify the electrochemically generated hydroxyl radical and Fig. 6 depicts a schematic shape of process (Diaw et al. 2017). The combination of thermolysis and photolysis processes for degradation of PFAS is another in situ method. Using these two technologies at the same time shortens the reaction time and there is no need for a highly acidic environment to accelerate the experiments. Here, the main mechanism for PFAS degradation is homolytic decarboxylation which results in C–C bonds breakage (Reaction 6) (Liu et al. 2017).

$$O_2 + 2H^+ + 2e^- \rightarrow H_2O_2 \tag{3}$$

$$Fe^{3+} + e^- \rightarrow Fe^{2+} \tag{4}$$

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO^{\cdot} + OH^- \tag{5}$$

$$C_7F_{15}COOH \longrightarrow C_7F_{15}^{\cdot} + HOOC^{\cdot} \tag{6}$$

Using an electron beam is another emerging approach for PFAS remediation (Jiang et al. 2016). By inducing the ionizing radiation to the water, the electron beam can produce extreme reducing and oxidizing radicals in the water (Singh et al. 2019). But, to fully decompose the contaminant using this method, a high level of energy is needed for enough radical production. So, it is necessary to couple this method with another to achieve more cost-effective treatment (Han et al. 2012). One way is to couple electron beam with activated persulfate (Fig. 7). This combination can cause a synergistic effect in three different mechanisms for PFOS degradation: (1) electron beam radiation will activate the persulfate (Reaction 7), (2) electron beam will directly break the chemical bonds in PFOS (Reaction 8) and (3) chemical bond in PFOS will be broken by pre-activated persulfate (Reaction 9). Although this method can effectively increase the degradation efficiency for PFOS treatment, it is still not applicable for full-scale water treatment plants due to the high capital costs for the electron beam source (Han et al. 2012; Kim et al. 2019).

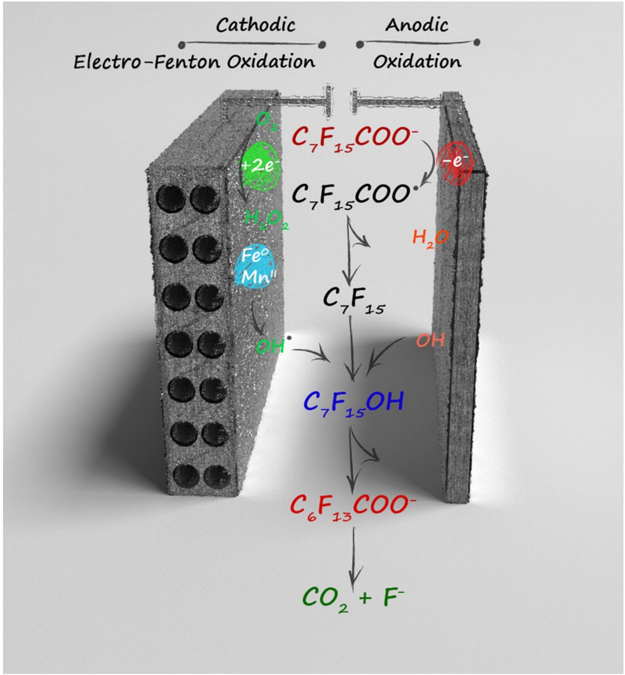

Fig. 6 Cathodic electro-Fenton and electrochemical anodic oxidation system, the reaction sequences and final product

$$S_2O_8^{2-} + e_{aq}^- \rightarrow O_4^{\cdot-} + SO_4^{2-} \tag{7}$$

$$C_8F_{17}SO_3^- + e_{aq}^- \rightarrow C_8F_{17}SO_3^{2-\cdot} \tag{8}$$

$$C_8F_{17}SO_3^- + SO_4^{\cdot-} \rightarrow C_8F_{17}SO_3^{\cdot} + SO_4^{2-} \tag{9}$$

Biological treatment is another method that has been used in an in situ hybrid treatment system. Although environmentally friendly, biological treatment is not solely sufficient for removal of PFAS from the water (Gonzalez et al. 2020). However, as a subdivision of in situ treatments,

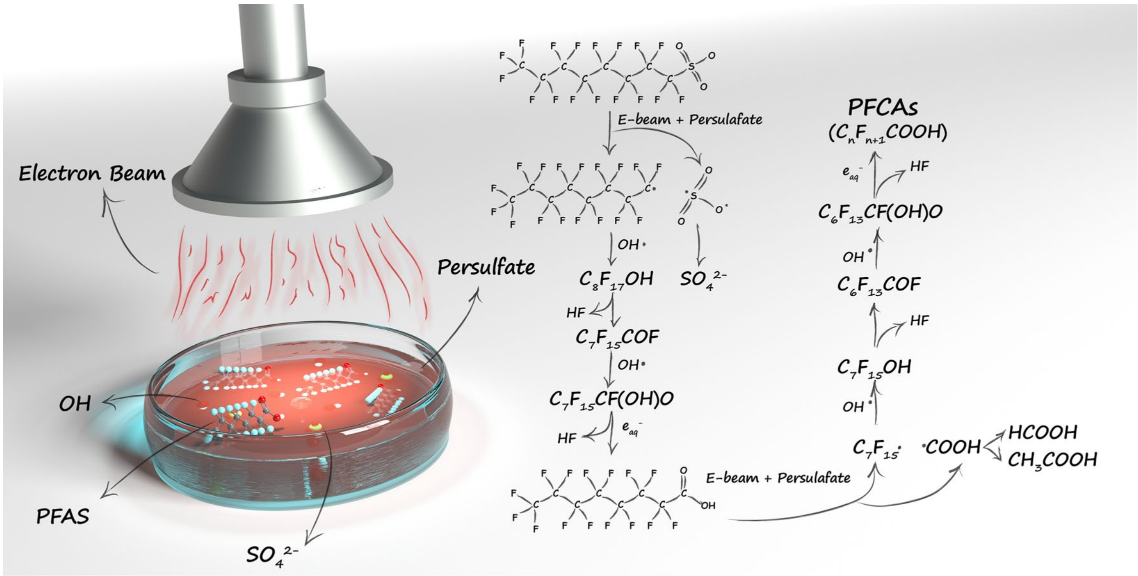

Fig. 7 Electron beam system coupled with persulfate and proposed PFAS decomposition chart. PFAS: per- and polyfluoroalkyl substances and PFCA: perfluorinated carboxylic acid

there is a chance for biodegradation of PFAS. This type of treatment is based on the cooperation of oxidant–reductant substances and microbes (Gonzalez et al. 2020). The living species can provide oxygen for reductants like zero-valent iron under a stressed condition (Hameed and Rahman 2008). Notably, living diatoms (like *Chaetoceros muelleri*) can bond strongly to the metallic parts of the reductant and then use the released carbon dioxide from PFAS degradation for photosynthesis inside the water (Albert et al. 2020). This technology is still challenging due to the difficulty of its scale-up. Also, it is hard to harvest the living diatom after each treatment cycle and there is a large volume of hydrogen peroxide in the disposal that needs to be removed (Bourgeois et al. 2015; Ochoa-Herrera et al. 2016).

Figure 8 compares three different variables for six various effective treatment protocols. Based on this short review, it can be concluded that adsorption–reduction and/or filtration processes can result in a high rate of PFAS treatment efficiency, but these methods will typically need an extra step to treat the concentrated retentate or regenerate the adsorbent. All the oxidation-based processes require large amounts of acidifier or alkane materials, and high operating temperatures. In some cases, these degradations create hazardous by-products which increase the cost of the whole operation.

Based on the articles included in this review, the combination of the electro-Fenton and the electrochemical processes appears to be the most promising treatment protocol for PFAS degradation. This system is based on the two processes which have the same oxidation-based mechanism with no need for multiple energy sources. These two processes have a high rate of electrical to chemical energy conversion. Both the electro-Fenton and the electrochemical processes produce in situ hydrogen peroxide; therefore there is no need for the external input of chemicals. Overall, the whole system lies in the cathodic–anodic cycle, has a high removal rate, and short residence time so it has the potential for relatively simple scale up for large-scale treatment plants.

Conclusion

PFAS have been utilized globally in many products (e.g., cookware, firefighting materials, carpet, textile, paper, leather, etc.) since 1940 (Cui et al. 2020). Their physicochemical properties make them attractive across many industries such as semiconductors, etching, metal plating, building and photolithography. However, PFAS are associated with many adverse effects on the environment and human health. There are numerous reports on their bioaccumulation, and their prevalence and persistence in the environment. PFAS do not partition from water to air because of their low vapor pressure and their high solubility in water (Place and Field 2012) but PFAS can transfer from water to soil and enter the food chain (Liu et al. 2019a). Many PFAS with long half-lives have been found present in biological system. This can be an underlying reason contributing to obesity, cancer, immune system suppression, elevated cholesterol, and endocrine disruption (Pramanik et al. 2020).

PFAS (mostly PFOA and PFOS) have been frequently detected in groundwater, surface freshwater, landfill leachate, and drinking water; environmental protection agency has issued a lifetime health advisory of 70 ng/L

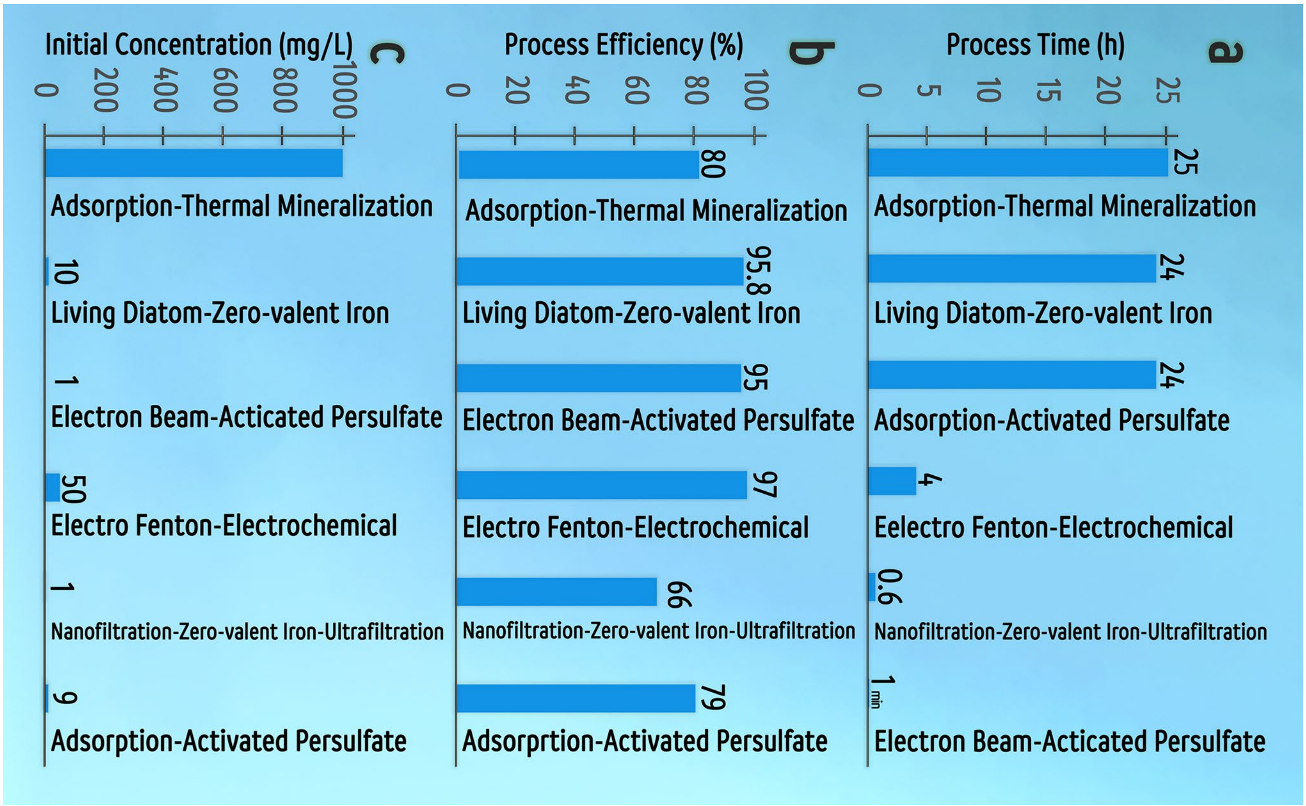

Fig. 8 Comparison of (**a**) process time (in hours), (**b**) process efficiency and (**c**) PFAS initial concentration (in mg/L) in different treatment protocols. The resources for creating Fig. 8 can be found in supporting information Table S1

for PFOS and PFOA in drinking water. Therefore, it is crucial to classify an effective characterization method for detection of PFAS. Chromatographic-based techniques coupled with mass spectroscopy are the most common characterization methods for PFAS detection. The official analytical method that environmental protection agency has established is based on solid-phase extraction LC-MS/MS. In this method, a PFAS mixture is simultaneously separated and detected. Furthermore, this method is more precise, accurate, sensitive, and selective than other methods. However, solid-phase extraction LC-MS/MS is not suitable for on-site monitoring application because of the need for professional operators, expensive instrumentations, and lengthy sample preparation. The major advantage of solid-phase extraction LC-MS/MS is that all competing methods lack precision or accuracy, especially in discriminating various types of PFAS. Furthermore, current characterization methods are mainly focused on PFOS and PFOA. There are around 5000 types of PFAS in the environment, so it is vital to develop characterization techniques that can analyze a wider range of PFAS and explore poorly studied PFAS.

Addressing PFAS will require a multi-faceted strategy including reducing PFAS production, decreasing discharge to the environment, remediation of past PFAS contamination and treatment of water resources to prevent adverse effects on health and environment. While eliminating PFAS from industrial processes completely would be desirable, PFAS compounds have chemical characteristics that are both rare and necessary for many processes and products; therefore, it is unlikely that they will be fully replaced by other chemicals in the foreseeable future. Additionally, past discharges of PFAS and their mobility and persistence in the environment will continue to pose a threat to humans through exposure and consumption of trace-level contaminated water. Along these lines, more strict regulatory limits are likely to be promulgated in the coming decades, and the treatment of contaminated water resources will likely remain an important societal need throughout the 21st-century.

Selecting treatment processes for a particular PFAS-contaminated water requires the consideration of many factors such as treatment effectiveness, cost, energy, carbon footprint, and the generated by-products. Given the huge number of PFAS compounds and their specifics, it is expected that we witness the growth and development of many hybrid treatment approaches and processes for treating water resources contaminated with complex or unknown mixtures of PFAS. In summary, hybrid treatments using multiple removal techniques can further improve the quality of effluent, lower the time and cost of experiments. Ultimately, designing an ideal treatment approach for PFAS requires proper analytical and reaction engineering knowledge along with a good understanding of the PFAS compounds present and their physicochemical properties. This knowledge can be leveraged to tailor the treatment approach to achieve the desired and permissible discharge concentration value in the effluent.

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Declarations

Conflict of interest The authors have no conflicts of interest to declare that are relevant to the content of this article.

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