

Review

Poly- and Perfluoroalkyl Substance (PFAS) Analysis in Environmental Matrices: An Overview of the Extraction and Chromatographic Detection Methods

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Abstract: Per- and polyfluoroalkyl substances (PFASs) are carbon–fluorine compounds with widespread industrial and domestic use, posing potential toxicological risks to humans and ecosystems. Several analytical methods have been developed to assess the occurrence of PFASs in the environment, but a standardized method, applicable to all matrices, is still lacking. This paper reviews the extraction and chromatographic detection methods for PFAS assessment in environmental samples, considering parameters such as the LOD, LOQ, and recoveries. Solid phase extraction (SPE) is commonly used, showing high recovery rates for water, soil, and sediment samples using HBL and WAX polymeric sorbents (85–100% and 93–111.5%, respectively). LC-MS has demonstrated low LODs and LOQs in seawater (0.01–0.08 ng L⁻¹; 0.03–0.24 ng L⁻¹), marine sediment (0.002–0.018 ng g⁻¹; 0.004–0.054 ng g⁻¹), and dust (0.08–0.68 pg g⁻¹; 0.26–2.25 pg g⁻¹), indicating its sensitivity when detecting trace PFAS levels. Evaluating PFASs is crucial for the development of future removal strategies and risk assessments. Potential solutions including the use of PFAS substitutes and innovative adsorption techniques for their adsorption could present promise in reducing their environmental presence.

Keywords: PFAS; environment; exposure; toxicological hazards; extraction approaches; detection methods



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1. Introduction

Per- and polyfluoroalkyl substances (PFASs) are emerging contaminants, largely present throughout the environment. Data suggested by the Organisation for Economic Cooperation and Development (OECD, 2018) report 4700 PFASs being currently available on the market [1]. Their chemical inertness, thermal stability, amphiphilicity linked to their highly polarity, and strong carbon–fluorine (C-F) bonds allow them to be used in manifold industrial applications (households and industrial products, retardant flame, waterproof clothing, food packaging, aqueous film-forming foams (AFFF), functional fluids for machinery, and anti-corrosion agents) and their following release into the environment [2–5].

The emission of PFASs can occur through direct or indirect routes. Industrial discharges, landfills, agricultural runoff, firefighting training sites, and wastewater treatment plants (WWTPs), as well as atmospheric deposition, are direct sources which promote the distribution of PFASs to remote areas [4,6]. On the other hand, they can be indirectly released through the degradation of neutral and volatile PFAS precursors [7]. The persistence and bioaccumulation of PFASs in the environment have become a serious concern for the potential toxic effects on human and wildlife [8].

Studies focused on the toxicological profiles of perfluorooctanoic acid (PFOA), perfluorooctanesulfonic acid (PFOS), and perfluorohexanesulfonic acid (PFHxS) from the Agency

for Toxic Substances and Disease Registry (ATSDR) have suggested ranges of human half-lives of 2.1–10.1 years, 3.1–27 years, and 4.7–35 years, respectively [9]. The same compounds have also been observed to have half-lives in ranging from 1.2 to 3.3 days for the northern leopard frogs (*Rana pipiens*), a bioindicator frequently used for ecosystem health monitoring [10]. Generally, PFASs are present in varying forms of anions, zwitterions, cations, or neutral compounds within the environment [11].

The transport of PFASs from the environment to the human body occurs through several environmental media, such as water, sediments, air, and dust [12]. Therefore, they can enter the human body through digestive, respiratory, and cutaneous pathways [13]. Liver and blood are the main accumulative pathways of PFASs [14–16]. Levels of PFHxS, PFOA, and perfluorononanoate (PFNA) have been observed in liver and blood tissues in the mean concentrations of 0.153, 0.301, and 0.244 ng g⁻¹, with the highest mean concentration of PFOS being in liver (6.277 ng g⁻¹) [17,18]. Moreover, a higher concentration of PFASs in tumor liver tissue (mean 64.3, range 5.70–303 ng g⁻¹) has been found when compared to non-tumor liver tissues, confirming the major exposure of patients with cancer to oxidative stresses [19]. Nonetheless, the investigations about the combined effects and toxicological interactions have also confirmed the increasing cytotoxicity of PFASs to the human cell liver line (HepG2) with increasing carbon chain lengths [20,21]. The fate of PFASs can reach long distant organs, such as lungs and their bronchial epithelial cells [22]. The pulmonary epithelium and the layer of lung surfactants (LS) covering the cells are the primary portal of inhaled pollutants, creating a physical barrier of entry between the outside environment and blood circulation [23]. In the study of Pye et al. [24], it was shown that environmental protein contained in dust particles inhaled can act as a transport carrier of PFASs into the respiratory system, modifying the lung surfactant function and inducing pro-inflammatory and allergic responses [25–27].

Considering the fate of PFASs in the environment and their potential negative effects, it is indispensable to develop an accurate and sensitive analytical method with which to control them. Among the extraction procedure of PFASs, solid phase extraction (SPE) is the main protocol used for the extraction, purification, and pre-concentration of PFASs in environmental media [7]. Moreover, it is crucial to understand the behavior of PFAS–sorbent interactions at different conditions in order to optimize the extraction procedure [28]. For example, the hydrophilic–lipophilic balanced (HBL) sorbent exhibits a low affinity for short-chain PFASs (perfluorosulfonic acid (PFSA)) and perfluoroalkyl carboxylic acid (PFCA)), and a greater affinity for the zwitterionic PFASs. Otherwise, the weak anion exchanger (WAX) polymeric cartridges (e.g., Oasis WAX and Strata-X) exhibits better recovery rates for short chain homologues; the presence of tertiary amine functional groups enhances the retention of anionic PFASs via electrostatic interactions [29,30]. For PFAS monitoring, liquid chromatography–mass spectrometry (LC-MS/MS) is the main analytical procedure applied. Specifically, high-performance liquid chromatography (HPLC) and ultra-high pressure liquid chromatography (UHPLC), coupled with triple quadrupole mass spectrometers (QqQ) or triple quadrupole tandem mass spectrometry (MS/MS), show a higher sensitivity for PFAS quantification [3,31]. However, gas chromatography (GC) coupled with a flame ionization detector (GC-FID), an electron capture detector (GC-ECD), and a mass spectrometer (GC-MS) has been employed as well [32]. The present review aims to summarize the current extraction and analytical approaches for the determination of PFASs in environmental matrices and details like water, soil, air, and dust in order to underline the absence of a standardized methodology for such analytes. Furthermore, a critical analysis of the current knowledge about both analytical methods, as well as the toxicological aspects, has been provided. This review also considered the regulation of force thus far in order to analyze its effectiveness on the protection of both human and wildlife health statuses.

2. Literature Research

The literature research was conducted, searching the most recent studies (from 2015 to 2023) on Scopus and Google Scholar databases. To this purpose, the keywords selected were “per- and polyfluoroalkyl substances”, “environment”, “toxicology”, “extraction techniques”, and “analytical methods of detection”. A total of 1188 studies emerged. The studies were first analyzed by reading their titles and abstracts, and 556 studies were excluded. Afterwards, only studies which met the aim of the present review (no = 632) were screened by reading the full text. Hence, 80 out of the 632 were selected for the review.

3. Toxicology and Risk Assessment of PFAS

Toxicity studies of PFASs have shown the detrimental effects on humans and on the environment [8,33,34]. Due to the amphiphilic properties, these compounds can attach to proteins and be transported in the blood, kidney, and liver, causing multiple adverse effects, such as perturbations in thyroid, liver, and kidney disease, reproductive toxicity, high cholesterol, and immunosuppression [35,36]. A work by Poothong et al. (2020) [37] showed an average concentration of 0.80 ng mL⁻¹ and 0.90 ng mL⁻¹ of PFASs in the blood serum of people <41 and >41 years old, respectively. Furthermore, a positive correlation was demonstrated between the PFAS concentration in blood serum and those found in indoor dust. The highest levels of PFASs were detected in the human whole blood, serum, and plasma of samples collected from people aged between 20 and 66 years old. The levels were higher in both the serum and plasma (10 ng mL⁻¹) as compared to the whole blood (6.3 ng mL⁻¹) [38]. The entering of PFASs into the blood circulation could lead to the transport of PFASs into other organs and their consequent accumulation within them. A concentration ranging between 0.04 and 0.19 ng g⁻¹ was found in human kidney tissue, likely due to environmental exposure [39]. The potential toxicity of PFASs (PFOA and PFOS) is likely correlated to the extended half-life that they could have due to the re-adsorption in the renal tubule; specifically, in the proximal convoluted tubule and the distal convoluted tubule [40]. The distribution and bioaccumulation of PFASs in different tissues can be affected by their physicochemical properties. Experiments conducted on rat models conducted by Cao et al. (2022) [41] suggested the key role of binding proteins, like ASBT and NTCP, involved in the enterohepatic circulation in the reuptake of PFASs from the intestinal lumen and the accumulation in blood and the liver. Concentrations of PFASs in the liver are rarely reported. A work by Baumert et al. (2023) [42] showed for the first time the concentration of PFASs (PFOA and PFOS) measured simultaneously in the plasma and livers of 64 individual Teen-LABS participants. The highest concentration of PFOA and PFOS were detected in plasma (1–10 ng mL⁻¹). Likewise, a high PFOS concentration (1–10 ng g⁻¹) was observed in the liver. The accumulation of PFOS and PFOA in the liver and plasma tissues, as studied using the intratissue Mass Balance Model (MBM), was explained by the role of membrane lipids as carriers of hydrophobic, long-chain PFASs in the liver and by the albumin-binding hydrophobic PFASs, which are strongly present in plasma. Moreover, the strong correlation between plasma and liver concentrations ($r^2 = 0.991$), found by means of the multilinear regression (MLR) model, suggested the ability to predict the concentration of PFASs in liver from those found in plasma. Basing on the epidemiological evidence of PFAS toxicity, deleterious impacts on liver metabolome, specifically bile acid metabolism, have been shown following environmental exposure to PFASs [43]. It was observed that the increase in circulating cholesterol levels, as well as the alteration of bile acid and fatty acid metabolites, could affect the enterohepatic circulation and energy metabolism [44], and consequently dysregulate the lipid metabolism and sterol homeostasis, leading to the development of non-alcoholic fatty liver disease (NAFLD) [45] and its more severe form, non-alcoholic steatohepatitis (NASH). The first potential exposure to PFASs begins in utero, specifically for people who transfer the fetus through the placental passage [46,47]. Trophoblast cells form the outermost layer of the blood—placental barrier, which vehicles the PFAS transfer. In particular, the cell line JEG-3

is relevant for the presence of multinucleated trophoblasts, which simplify the exchange of nutrients, waste, and gases between maternal and fetus compartments in the placenta [48].

However, the concentration and ability of PFASs to cross the placental barrier depends both on passive and active diffusion and on their binding affinity to proteins in human serum. A work conducted by Lu et al. (2021) [49] reported the highest level of PFOSs and PFOAs (mean: 0.457 and 0.242 ng g⁻¹ ww, respectively) in placental human samples. They observed that the binding affinities of PFASs to human serum albumin (HAS) increased with the chain length. However, due to the difficult transfer of PFAS–protein complexes through the placental barrier, the concentration of free PFASs in the blood is an important factor for the placental passive diffusion [50]. As for the active transport process, PFASs act as a vehicle for the organic anion transporters (OAT) placed on the placenta membrane. Although the binding behavior of PFASs as placental transporters is still unknown, an increase in binding affinities to OAT4 was observed with the chain length of PFASs [49]. Prenatal exposure to PFASs may be harmful for the child's health. Studies have suggested negative outcomes regarding cognitive and neurobehavioral development [51,52], the risk of asthma [53], allergies, and infectious diseases [54]. However, although the toxicity of PFASs at high doses of exposure (acute toxicity) is well documented, there are few studies focusing on chronic effects on health. A work from Kristensen et al. (2013) [55] investigated the possible long-term effects of in utero exposure to PFOAs and PFOSs on female reproductive functions. A significant association between prenatal exposure to PFOAs and menarches occurring at a later age was observed, with a difference of 5.3 months between the lowest and highest tertiles of the PFOA concentration. This delay could have psychological consequences on human health, but no significant association between PFAS exposure and the menstrual cycle length, as well as the reproductive hormone levels or the number of antral follicles in young adulthood, was observed. Moreover, the only PFOA association with the age at the time of the menarche occurring could be explained via the easily transfer of PFOAs through the placenta barrier. Epidemiological studies on mice exposed to PFASs for 12 weeks showed elevated serum cholesterol levels, sterol metabolites, and bile acids, with some sexual dimorphic effects [44]. Moreover, a recent work by Maxwell et al. (2024) [55] on male mice exposed to PFASs for 18 weeks showed the influence on sperm methylation, which interfered with the epigenetic regulation of the gene expression of offspring liver and fat. This result could be significant in predicting the long-term offspring gene expression and the link between environmental PFAS exposure and the health of future generations. Dust particles inhaled, as well as dermal contact with household cleaning products have also been indicated as a potential source of PFAS exposure [56].

Emerging evidence has suggested associations between air pollutants, like PFASs, and lung damage [23,57,58]. It was observed that fine particulate matter with an aerodynamic diameter of less than 2.5 µm (PM 2.5) can readily enter the lower airways, causing respiratory and cardiovascular diseases, including asthma, chronic obstructive pulmonary disease (COPD), pulmonary fibrosis, cancer, type-2 diabetes, neurodegenerative diseases, and even obesity [59]. However, these pollutants are also spread in indoor environments (home and public spaces) and inhaled via air or dust. Animal studies conducted by Gustafsson et al. (2022) [60] revealed that the concentration of PFOAs on lung tissue was almost 2.5 times higher when compared to the liver and kidney following the inhalation of and oral exposure to PFOA-coated house dust. In addition, data obtained via US EPA 2017 suggested dust ingestion rates of 50–100 mg day⁻¹ (toddlers) and 20–60 mg day⁻¹ (adult), with daily PFOA-equivalent intakes exceeding the daily threshold fixed by the European Food Safety Authority (EFSA) (1.3–1.5 ng kg⁻¹ body weight (bw) d vs. 0.63 ng kg⁻¹ bw d⁻¹) [61].

4. Regulation

Ecological and potential human health effects of PFASs have caused regulations and restrictions regarding their use to be implemented. In 2018, the “Global PFC Group”, led by the Organisation for Economic Co-Operation and Development (OECD) and the

United Nations Environment Programme (UNEP), published a list of over 4700 PFASs that contain C_nF_{2n} ($n \geq 3$) or $\text{C}_n\text{F}_{2n}\text{OC}_m\text{F}_{2m}$ (n and $m \geq 1$) moieties [1]. Among these, PFOSs and PFOAs are the most known and frequently used PFAS, listed as persistent organic pollutants (POPs) under the Stockholm Convention, together with their related salts [62]. In January 2023, the National Authorities of Germany, Denmark, the Netherlands, Norway, and Sweden submitted a proposal of the reduced application of PFASs to the European Chemicals Agency (ECHA), with the aim to reduce PFAS emissions into the environment, and to make products and processes safer for people (ECHA/NR/23/01) [63]. This objective is in line with the ambitions of the EU Chemicals Strategy and the Zero Pollution Action Plan, which assumes the reduction levels of pollutants in air, water, and soil, and creates a toxic-free environment (European Commission, 2021) [64]. Despite the emergency of PFASs being a common focus of investigation for scientific and political communities, their toxicity, their potential risks on health, as well the exposure levels of humans are still uncertain. The lack of evidence between public health adverse impacts and their behavior has led governments to set different guidelines for some PFASs. Moreover, the lack of standardized analytical methods and stable isotope-labeled internal standards, the interdependence of programs, legislative support, and funding represent important challenges, which challenge setting uniform PFAS values among different countries. Therefore, with the aim of enforcing the effective measures of the protection of both health and the environment, it could be necessary to adopt integrated approaches, including the prevention, monitoring, and control of PFASs, the use of alternative chemicals, which should be less toxic and should not persist in the environment, and a strong cooperation among governments.

5. PFAS Analysis on Environmental Matrices

5.1. Surface Water, Underground Water, Wastewater

Consistent levels of PFASs have been identified in several environmental matrices. Predominantly, the aquatic environment is the most polluted [65]. Water is an essential element for the entire terrestrial ecosystem, and its pollution is a threat for all living creatures [66]. For this reason, an accurate evaluation of the concentrations of perfluorinated compounds in various aqueous matrices is necessary. The procedure of PFAS analysis for water samples usually consists of SPE, followed by LC-MS [67–72]. Table 1 summarizes the analytical procedures of samples from some recent studies on the analysis of PFASs in surface water, wastewater, and groundwater, and its recoveries, LOD, and LOQ. The comparison shows differences in the types of cartridges, extraction solvents, and analytical parameters used to determine different recovery, LOD, and LOQ rates. Aqueous samples usually contain a low level of PFASs (ng L^{-1} or pg L^{-1}). Therefore, greater attention must be paid to the sample preparation, focusing on the clean-up of the sample matrix background and the pre-concentration of PFASs [65]. Brumovsky et al. (2018) [28] assessed the influence of the type of sorbent, the matrix pH, the salinity, and the eluent on PFAS recovery. Firstly, they used three different SPE sorbents for the study: StrataTM-X, Oasis[®] hydrophilic-lipophilic balance (HLB), and Oasis[®] weak anion exchange (WAX). Thereafter, they prepared three matrices with different salinities: tap water, artificial seawater, and a 1:1 mixture of the two. They obtained the highest recovery of the studied PFASs using the Oasis[®] HLB sorbent, with methanol (MeOH) as the eluent, at a pH = 8, and with 50% (brackish water) or 100% (marine water) seawater content (82.8% and 78.3%, respectively). They found slightly lower values using the StrataTM-X sorbent at the same pH and salinity values (75.6% and 71.3%, respectively). On the other hand, the Oasis[®] WAX sorbent provided lower recoveries. This sorbent allows for the washing of the adsorbed analytes using MeOH, as PFCAs and PFASs are reluctant to be eluted under these conditions, representing an advantage when the removal of interfering organics is required. Similarly, Janda et al. (2019) [73] evaluated two sorbents with weak anion exchange properties (Oasis WAX and Strata X-AW) for the optimization of the SPE recoveries at different pHs, and two stationary phases for LC separation for the determination of PFCAs with chains from C₂ to C₈, as discussed later.

The obtained PFCA recoveries from C₄ to C₈ were between 76 and 133% for both SPE materials, regardless of the sample pH, whereas the extraction efficiencies of trifluoroacetate (TFA) and perfluoropropanoate (PFPrA) depended on the sample pH. They obtained PFPrA recoveries between 93 and 103% for all pH values using the Oasis WAX sorbent, while the recoveries with Strata X-AW were acceptable in the pH range from 3.0 to 5.0 (from 94 to 114%), and then decreased at a pH of 6.0 (36%). They achieved the quantitative extraction of TFA between pH 3.0 and 5.0 (95 to 103%) using the Oasis WAX sorbent, while the Strata X-AW sorbent recovered TFA at 99% (pH 3.0) with reduced extraction recoveries at higher sample pH levels. They selected the Oasis WAX sorbent for the SPE of surface, groundwater, and drinking water samples, and adjusted the sample pH to 3.9 ± 0.1 before extraction. They performed the analysis using a liquid chromatography–tandem mass spectrometry (LC-MS/MS) system with a core–shell C₁₈ column and an acidified eluent. On the other hand, Lockwood et al. (2019) [68] demonstrated the use of μSPE as an alternative to conventional SPE for PFAS extraction. The authors compared the efficiency of four sorbents for the extraction of 13 PFASs from ultrapure water. The sorbents studied were C₁₈, aminopropyl silica (APS) phase, pentafluorophenyl (PFP), and diol sorbent. Among them, C₁₈ provided good retention for most compounds, while APS was the only phase which was capable of retaining the compound in the chain short perfluorobutanoic (PFBA). For these reasons, the combination of the reversed-phase C₁₈ sorbent and APS (as a 50:50 mixture) was chosen to pack the μSPE cartridge. This method reduced the extraction times to 5 min compared to conventional SPE methods, and achieved similar results while using a smaller sample volume. Additionally, the automation of the extraction method was demonstrated using an eXact³ digital syringe and an ePrep[®] sample preparation workstation. Regarding the separation and detection of PFASs in water samples, LC-MS is the most used technique [65,67–74]. In the study reported above, Janda et al. (2019) [73] also studied two stationary phases for LC. The authors evaluated a mixed-mode phase, offering both ion exchange and HILIC (Obelisc N) and a core–shell RP column (Kinetex C₁₈) for chromatographic separation. Using the Obelisc N column, it was possible to obtain a separation of the studied PFCAs in ultrapure water; however, in the environmental samples, the separation was disturbed by matrix effects. For this reason, the study continued using the Kinetex C₁₈ column. In the final method, a binary gradient was applied (eluent A: 2 mM ammonium formate (AF) and 0.2% formic acid (FA) in H₂O_{mQ}/MeOH (4:1, *v/v*); eluent B: 2 mM AF in MeOH). FA was added to eluent A, as it was observed to promote the retention of TFA and PFPrA. Mulabagal et al. (2018) [69] investigated the presence of PFASs in estuarine water using ultra-high performance liquid chromatography–triple quadrupole mass spectrometry (UHPLC-QqQ-MS). Several parameters were examined to optimize the analysis as follows: column type, solvents in the mobile phase, flow rate, and column temperature. Best results were obtained with an Agilent ZORBAX RRHD Eclipse Plus C₁₈ column, thermostated at a T of 40 °C, and a binary solvent gradient consisting of 5 mM AF in water and MeOH. In addition, a guard column was used to achieve reproducible retention times. Following method optimization, recovery experiments were performed via adding 23 PFAS analytes and the internal standard (ISTD) to ambient water samples. Zhu et al. (2020) [75] investigated a simple method of the derivatization of PFOAs, which achieves the enabling of the separation of its isomers/enantiomers via the use of gas chromatography–electron capture negative ionization mass spectrometry (GC-ECNI-MS) detection. (S)-1-phenethyl chloride (PhEtCl) was used as a derivatizing agent; this reacts with PFOA enantiomers to form PFOA (R)-1-phenylethyl esters. Derivatization was performed via adding 100 mL of 5% PhEtCl in acetone (ACE) to a standard extract. The GC analysis was carried out by means of a non-chiral GC column (HP-5MS column). The method was subsequently applied to river water samples for measuring PFOA isomer/enantiomer concentrations. Samples were preliminary treated with an Oasis WAX plus SPE cartridge, and then were subjected to a derivatization step. Various PFOA isomers/enantiomers, mainly 3m-PFOA, 4m-PFOA, 6m-PFOA, and L-PFOA, were detected in the river water sample, with concentrations of 5.2, 3.8, 41, and 3400 ng L⁻¹, respectively.

Table 1. Summary of the analytical procedure for the analysis of PFASs in surface water, wastewater, and groundwater and their recoveries (%), LOD (ng L⁻¹), and LOQ (ng L⁻¹). N/A = not available, ^a = pg/injection.

Matrix	Extraction and Pretreatment	Analysis	Recoveries	LOD	LOQ	Ref.
Surface and underground water	Oasis WAX cartridge SPE online	High pressure liquid chromatography–mass spectrometry (HPLC-MS)	80–120	N/A	0.2–5	[65]
Seawater	Oasis WAX cartridges SPE	Ultra-high performance liquid chromatography (UHPLC-MS)	Above 50 except PFBA in seawater (<10)	0.001–0.331	N/A	[67]
Surface water	μSPE cartridge	UHPLC-MS	86–111	0.29–6.6	N/A	[68]
Estuarine water	Waters Oasis PRiME HLB cartridges SPE	Ultra-high performance liquid chromatography tandem mass spectrometry (UHPLC-MS/MS)	78.5–112.6	0.48–1.68 ^a	1.7–5.4 ^a	[69]
Surface water and rainwater	Waters Oasis HLB cartridges SPE	Ultra-performance liquid chromatography–tandem mass spectrometry (UPLC-MS/MS)	72–98	0.02–0.03	0.04–0.06	[70]
Surface water	WAX SPE cartridges	LC-MS/MS	68.5–118	0.5–10	2–20	[71]
Groundwater	Oasis WAX cartridges SPE	LC-MS/MS	73–137	0.03–0.06	0.03–1.27	[72]
Surface water and groundwater	Oasis WAX cartridges SPE	LC-MS/MS	83–107	0.1–5.5	0.6–26	[73]
Seawater	Oasis [®] cartridge SPE and Envi-Carb TM cartridge	LC-MS/MS	63.2–112.9	0.01–0.08	0.03–0.24	[74]
Surface water	Derivatization using (S)-1-PhEtCl	GC-MS	92	0.050–0.15	N/A	[75]
Surface water	Strata-X cartridge SPE	Liquid chromatography triple quadrupole mass spectrometer (LC-QqQ-MS)	44–100	N/A	0.01–2.00	[76]
Groundwater	Water Oasis HLB Plus Short cartridges SPE	UPLC-MS/MS	70–130	0.02–0.03	0.04–0.06	[77]

5.2. Soil and Sediments

Soil contains over 40 classes of PFASs, usually originating from human activities, such as industrial operations and waste disposal [78]. Table 2 summarizes the extraction, pre-concentration, and analysis techniques of some recent studies, along with their respective recoveries, LOD, and LOQ. The main differences among the studies are in the types of cartridges and analysis techniques used. Sammut et al. (2019) [77] conducted the extraction procedure using MeOH as the solvent and a short Waters Oasis HLB Plus cartridge for the SPE. They used UPLC-MS/MS with an ACQUITY UPLC BEH C₁₈ column for the separation. Zhong et al. (2021) [79] used MeOH as the extraction solvent, and performed a clean-up using the SPE with Supelco[®] ENVI-Carb cartridges. They performed the analysis and quantification via a LC-QqQ-MS system, using an ACQUITY UPLC BEH C₁₈ column for the separation. They obtained recoveries ranging from 96 to 127.1%, while the LOD and LOQ were 0.002–0.018 ng g⁻¹ dry weight (dw) and 0.004–0.054 ng g⁻¹ dw, respectively. Simon et al. (2022) [69] optimized a fast and simple SPE-free extraction method, based on liquid–solid extraction (LSE) using acidified MeOH. They compared the method with an additional SPE purification step, and showed that the clean-up step was unnecessary, as it

resulted in an underestimation of the organically bound extractable fluorine (EOF) concentrations. They carried out the analysis via by high resolution-continuum source-graphite furnace molecular absorption spectrometry (HR-CS-GFMAS). Peng et al. (2013) [80] developed a different technique to detect fluorotelomer alcohols (FTOH), using a derivatization reaction and liquid chromatography–electrospray mass spectrometry (LC-ESI-MS) in combination with low-energy collision-induced dissociation tandem mass spectrometry (CID-MS/MS). They used acetonitrile (ACN) as the solvent, and a WAX cartridge for the SPE for the extraction. They performed the derivatization by adding dansyl chloride (DNS) in acetonitrile and 4-(dimethylamino)-pyridine (DMAP) in dichloromethane (DCM) as the catalyst. They used ultra-performance liquid chromatography collision-induced dissociation tandem mass spectrometry (UPLC-CID-MS/MS) with a Waters ACQUITY UPLC BEH phenyl column for the analysis. Overall, the method recoveries ranged from 67 to 83%. The LOQ for all FTOHs were 0.017–0.060 ng g⁻¹. They applied the method to analyze six marine sediment samples. All FTOHs, except for 10:1 FTOH, were detected, and the total concentrations of FTOHs were 0.19–0.52 ng g⁻¹ dw. The developed method provides a new method to sensitively determine FTOHs in environmental matrices.

Table 2. Summary of the analytical procedure for the analysis of PFASs in soil and sediment and their recoveries (%), LOD (ng g⁻¹), and LOQ (ng g⁻¹). N/A = not available, ^a = ng mL⁻¹, ^b = method quantification limit (MQL, ng g⁻¹), ^c = method detection limit (MDL, ng g⁻¹).

Matrix	Extraction and Pretreatment	Analysis	Recoveries	LOD	LOQ	Ref.
Sea and river sediment and soil	Oasis® cartridge SPE and ENVI-Carb™ cartridge	LC-MS/MS	78.9–120	0.002–0.042 ^c	0.006–0.1 ^b	[74]
Sediment	Extraction with acetic acid (AcOH) and MeOH; cleaning up by Strata-X cartridge SPE	LC-QqQ-MS	44–100	N/A	0.04–8.00	[76]
Soils	Water Oasis HLB Plus Short cartridges SPE	UPLC-MS/MS	70–130	0.02–0.50	0.04–0.60	[77]
Soils	Oasis WAX cartridges, MeOH as solvent	LC-MS	52–167	0.100–400 ^c	0.01–0.30 ^b	[78]
Marine sediments	ENVI-Carb cartridges SPE, MeOH as solvent	LC-MS	96–127.1	0.002–0.018	0.004–0.054	[79]
Marine sediment	Derivatization dansyl chloride (DNS) in ACN under catalysis of 4-(dimethylamino)-pyridine (DMAP) and WAX and silica cartridges clean-up	Liquid chromatography–electrospray ionization–mass spectrometry (LC-ESI-MS) combined with CID-MS/MS	67–83	0.006–0.016	0.017–0.060	[80]
Soils	LLE using acidified MeOH	HR-CS-GFMAS	79–117	3.43	10.30	[81]
Sediment	MeOH-based extraction, Oasis WAX SPE cartridges	High performance liquid chromatography–electrospray ionization tandem mass spectrometry (HPLC-ESI-MS/MS)	86.7–113	0.013–0.089 ^a	N/A	[82]
Sediment	Pre-treatment with Oasis-HLB SPE and MeOH solvent	HPLC-ESI(-)-MS/MS	72–101.9	N/A	0.01–0.08 ^b	[83]
Sediment	ACN and MeOH as the extraction solvent	UPLC-MS/MS	80–115	0.02–1.11 ^a	N/A	[84]

5.3. Air and Dust

As reported above, many studies have focused on the measurement of PFAS concentrations in surface water, groundwater, soil, and sediments, while much less is known about atmospheric concentrations. The determination of volatile PFASs is crucial for studying their sources and fate. Volatile PFASs can be present in air and can be transformed into highly persistent PFCAs [85]. Dimzon et al. (2017) [86] investigated and validated a method for determining volatile PFASs in the air and water of industrial and municipal wastewater treatment plants (WWTP) in the Netherlands and Germany. During the study, a sampling and enrichment method was developed for volatile PFASs in air samples using Oasis HLB™ SPE cartridges. They used a low-volume air sampling device for air sampling. They spiked the cartridge with 20 µL of an enrichment control standard solution, attached it to a membrane pump, and eluted the analytes with 2 mL of n-pentane (C₅H₁₂). Furthermore, 1 µL of the sample was analyzed using GC-MS. They performed the mass analysis in a programmed selected ion monitoring (SIM) mode. They used different temperature ramp rates in the oven program for the GC separation of the different PFASs. They also chose a splitless injection to avoid system losses. They obtained an approximate MDL of 1 ng m⁻³ in the air, except for fluorotelomer acrylates (FTACs), which they could detect at >40 ng m⁻³. They obtained recoveries between 60 and 120% for all analytes using the volatilization and direct addition methods. Humans might be exposed to PFASs via indoor air and dust. Padilla-Sánchez et al. (2016) [87] developed and validated a fast and sensitive analytical method for the analysis of a large number of PFASs in indoor dust. They collected dust samples from vacuum cleaner bags, extracted them by adding MeOH, and then filtered them with a syringe coupled to a 0.45 µm PP filter. They mixed the extract with 0.25 mg of activated charcoal. They performed the analyses using ultra-high performance liquid chromatography–time-of-flight mass spectrometry (UHPLC-TOF-MS), equipped with an Oasis SPE column that allowed on-line SPE cleaning prior to analysis. In this study, the detection performed using ESI-MS was used, operating in sensitivity mode and negative ionization mode (NIM). They obtained MDLs and MQLs in ranges from 0.008 to 0.846 ng g⁻¹ and 0.027 to 2.820 ng g⁻¹, respectively, and recoveries in ranging from 58 to 114%. Similarly, Ao et al. (2019) [88] collected dust from carpet and floor surfaces using a domestic vacuum. They used accelerated solvent extraction (ASE) for the extraction and SPE for the clean-up of the samples. They used an Oasis WAX cartridge for the SPE process. The analysis was conducted using an UPLC-MS/MS. They obtained LODs and LOQs in the range of 0.08–0.68 pg g⁻¹ and 0.26–2.25 pg g⁻¹, respectively, for the target analytes. They obtained recoveries from 80.2 to 95.2%. PFASs have also been detected in remote regions, such as the Atlantic and Antarctic regions. Wang et al. (2015) [89] investigated the PFAS presence in the atmosphere across these regions. They conducted the air sampling onboard a research vessel. They collected a total of 33 air samples using a high-volume air sampler. They used a cartridge containing polyurethane foam and polystyrene divinyl benzene copolymer (PUF/XAD®-2) for sampling. They carried out the extraction in a Soxhlet extractor using DCM for 16 h. The analysis was performed using GC-MS in SIM mode with a positive chemical ionization (PCI) mode. They used a SUPELCO WAX 10 column for the separation, MeOH as the reagent gas, and helium as the carrier gas. They obtained an MDL in the sampled air, ranging from 0.01 to 0.10 pg m⁻³. They determined the total gas phase concentration of 12 PFAS, which ranged from 2.8 to 68.8 pg m⁻³, with an average value of 23.5 pg m⁻³. Camoiras González et al. (2021) [90] also used the PUF in their study, where they monitored the air in developing countries with a passive sampler. They exposed the PUFs for 3 months and then the extracted the analytes using a Soxhlet with a 60:40 methyl tert-butyl ether (MTBE)/MeOH mixture for 24 h. They performed the clean-up of the samples in the first period of the study using an ENVI-Carb SPE tube, and collected them with MeOH. In the second period of the study, they stacked the ENVI-Carb SPE tubes under a SPE-WAX cartridge. All the sample were analyzed with a LC-MS/MS, with ESI operating in NIM. Recoveries were in the range of 30–150% for all target analytes, and LOQs for PFSAs and PFAAs were between 11 pg/PUF and 14 pg/PUF. Wild birds have

also proven useful in the biomonitoring of environmental contaminants. For these reasons, in a recent study, Groffen et al. (2021) [91] developed an analytical method to determine a broad range of PFASs, using bird feathers as indicators of PFAS contamination. The feathers were collected from the ground, having fallen from free-range domestic chickens near a fluorochemical plant in Belgium. Simultaneously, non-contaminated feathers were collected at a site with low PFAS contamination. The samples were washed to remove soil and dust, cut into small pieces, and, for extraction, 10 mL of 100% MeOH and 10 ng of bulk labeled ISTD solution were added. Subsequently, the samples were vortexed for 1 min and left in the dark for 24 h at room temperature. After centrifugation, the supernatant was dried, and then reconstituted with 2 mL of a 2% ammonium hydroxide (AH) solution in ACN, vortexed for at least 1 min, and filtered through a 0.2 μm Supor polyethersulfone ion chromatography Acrodisc 13 mm syringe filter (PES). For the analysis, UPLC-MS/MS with a negative electrospray ionization mode (ESI-) was employed. An ACQUITY BEH C₁₈ column was used for the separation. Different gradients of FA and water were used during the analysis. Target analytes were quantified using the multiple reaction monitoring (MRM) of two diagnostic transitions per analyte. The extraction recovery ranged from 67.3% to 112%, while the LOD and LOQ values for all the analytes were between 0.09 and 25.8 ng g⁻¹ wet weight (ww) and 0.29 and 86.0 ng g⁻¹ ww, respectively.

6. Discussion

Numerous studies focusing on the extraction and analysis of PFASs suggest SPE as the best procedure for extracting PFASs from environmental samples. This technique shows high versatility as it is used to extract different categories of contaminants (PAHs, phthalates, PCB, pesticides), including PFASs from a wide variety of samples (biological, food, and environmental). Moreover, it is useful as a preconcentration and clean-up approach for the isolation and separation of the analyte, removing all the interference that could affect the chromatographic analysis. The concentration of the analyte in a small volume solvent enhances the accuracy and reproducibility of the analysis. Therefore, many parameters such as temperature, matrix pH, salinity, and the choice of specific adsorbent need to be optimized in order to achieve a better extraction efficiency. Despite the versatility of SPE being seen as a benefit, it can also be a drawback due to the potential lack of selectivity.

A few sorbents like Oasis HLB and WAX Oasis are used for the extraction of numerous target compounds, which are simultaneously retained [92].

Regarding the water samples, the best recoveries of PFASs were achieved using Oasis WAX (73–137%) and Water Oasis HLB Plus Short SPE (70–130%) cartridges at pH values between 6 and 8; on the other hand, slightly lower recoveries (44–100%) at pH values between 3 and 5 are shown with Strata-X cartridge. However, the μSPE procedure, when applied as an alternative to traditional SPE, bypassed the limitations of SPE via the use of smaller adsorbent sizes (<5 μm), leading to an increase in the extraction efficiency and lower solvent volumes (2 mL) and extraction times (5 min). Hence, the optimized mixture of C18:APS adsorbents (50:50) has allowed us to obtain satisfying recoveries for short- and medium-chain PFASs (C4-C10) and sulfonated fluorotelomers (96–105%) with RDS < 14%, excepting for PFOs (recovery: 129%; RSD < 29%). In addition, the derivatization procedure based on the identification of PFAS isomers/enantiomers has proven to be particularly useful for the evaluation of the potential toxicity of PFASs. Specifically, isomers/enantiomers of PFOAs, such as 3m-PFOA, 4m-PFOA, 6m-PFOA, and L-PFOA, are detected in river water samples at concentrations of 5.3, 3.8, 41, and 3400 ng L⁻¹, respectively. Derivatives of 5m-PFOA, 4.5 dm-PFOA, and 2m-PFOA are not detected, whereas the recovery of 3C4-PFOA was 92%. Although the derivatization process allows us to reduce the persistence of PFASs, attention should be paid to the production of by-products (i.e., PFHpS, PFHxS, PFPeS, PFBS, PFOA, PFHxA, and PFBA), which could have unknown effects on health and the environment. Furthermore, difficulties that could be found are a loss of analytes in traces during the pretreatment and derivatization steps, matrix effects with detection difficulties resulting in false positives, and the evaporation

and loss of volatile compounds such as PFAS esters when transferred from the aqueous to the organic phase. In addition, the analysis can be limited since only a particular class of PFASs with the same fractional group can be analyzed via derivatization [93].

For soil and sediment samples, the derivatization of fluorotelomeric alcohols (FTOH) with dansyl chloride (DNS) in acetonitrile and 4-(dimethylamino)-pyridine (DMAP) in dichloromethane (DCM) has demonstrated satisfactory recoveries (67 ± 6.0 – $83 \pm 9.4\%$) and matrix effects $< 15\%$. Methanol is the solvent mostly used for PFAS extraction from soil and marine sediments, showing interesting recoveries in combination with Oasis HLB 70–130%), ENVICARB (96–127.1%) and Oasis WAX (52–167%) cartridges.

Chromatographic analysis conducted using UHPLC-MS and LC-QqQ-MS are proved to be the most sensitive, achieving low LOD and LOQ values in water (0.001 – 0.331 ng L^{-1} ; 0.01 – 2.00 ng L^{-1}) and sediment (0.002 – 0.18 ng g^{-1} ; 0.004 – 0.054 ng g^{-1}). Combined with the conventional SPE procedures, they have been shown to be reliable and reproducible by identifying the most relevant PFASs, such as PFOS ($< \text{LOD}$ - 5.91 ng g^{-1}), PFOA (LOD - 0.58 ng g^{-1}), and PFDA ($< \text{LOD}$ - 1.05 ng g^{-1}) in real sediment, soil, and water samples. Moreover, although these analytical techniques are widely developed, they are only effective for the determination of a limited number of analytes. When compared to water and soil matrices, the number of studies focused on determining PFASs in the atmosphere are still limited. Analytical methodologies based on sampling with air samplers and enrichment using HLB and Oasis WAX cartridges have reported good recoveries of PFASs in air (60–120%) and dust (58–114%). Moreover, the UHPLC-TOF-MS analysis demonstrated a good sensitivity ($\text{MDL} = 0.008$ – 0.846 ng g^{-1} ; $\text{MQL} = 0.027$ – 2.820 ng g^{-1}), finding PFAS concentrations in real indoor dust samples in the range from 0.02 (PFHxS, NIPH 1) to 132 ng g^{-1} (6:2-diPAP, NIPH 4).

7. Conclusions

The relevant use of PFASs in various industrial sectors has raised considerable concerns about their harmful effects on the environment, humans, and the entire ecosystem. The ingestion and inhalation of contaminated air particles form the pathways of environmental PFAS exposure. Toxicological and detrimental hazards on respiratory, digestion, cardiovascular, and neurological systems have been proven, thus highlighting the necessity of developing a sensitive analytical method for their detection and removal. However, the widespread occurrence at a low level concentration (pg L^{-1} to mg L^{-1}) into the environment has enforced governments to set limits and restrictions regarding their applications. In this review, several analytical approaches for the extraction and following chromatographic separation of PFASs in environmental matrices have been discussed. SPE procedures followed by LC-MS analysis are the main approaches applied. The average recoveries using Oasis[®]s HLB (85–100%) and Oasis[®] WAX (93–111.5%) cartridges have been confirmed as providing the best performance for the purification and PFAS extraction in water, sediment, and soils samples. In addition, air and dust samples have also showed recoveries of PFASs between 60 and 120% and 80.2 and 95.2% using HLBTM SPE and Oasis WAX cartridges. The results obtained could be explained via the flexibility of the Oasis[®] HLB (N-vinylpyrrolidone-divinylbenzene copolymer) sorbent for the extraction of a large variety of compounds of different polarities, as well as by the similar structure to the Oasis[®] HLB of the Oasis[®] WAX sorbent (styrene-divinylbenzene polymer modified by a weak anion-exchange group piperazine), highly affine to anionic short-chain compounds at high pH levels. Very low LODs and LOQs were observed in seawater (0.01 – 0.08 ng L^{-1} ; 0.03 – 0.24 ng L^{-1}), marine sediment (0.002 – 0.018 ng g^{-1} ; 0.004 – 0.054 ng g^{-1}), and dust (0.08 to 0.68 pg g^{-1} ; and 0.26 to 2.25 pg g^{-1}) through LC-MS analysis. There are many advancements and innovative approaches being developed to improve the extraction, analysis, monitoring, and remediation of PFASs in the environment. These include the use of passive sampling techniques and alternative analytical techniques, which can help in identifying and monitoring PFASs in the environment. For example, the use of the NTA-HRMS (non-target analysis-high-resolution mass spectrometry) approach allows

us to identify the structures of unknown PFASs, distinguishing ions which are slightly different in their mass/charge ratio with high mass resolution, selectivity, and sensitivity. Advanced water and wastewater treatment technologies are effective for PFAS removal, but may require high operating costs and generate toxic waste. Therefore, PFAS removal and degradation technologies (electrocoagulation, foam fractionation, bioremediation, chemical oxidation/reduction, advanced oxidation/reduction processes), as well as PFAS adsorption on various materials (porous organic polymers (POPs) or low-cost carbon-derived materials) are being explored for an efficient PFAS remediation from both water and soil. In addition, the use of PFAS substitutes, such as carboxylic acid fluorotelomer 6:2 (6:2-FTCA), perfluoroalkyl carboxylates (PFECAs), GenX, ADONA, PFPiA, perfluoroalkyl ether carboxylic acid (PFESA), perfluorobutanesulfonic acid (PFBS), and perfluorohexanoic acid (PFHxA), which are less harmful than some specific PFASs (PFOA and PFOS), can help reduce the toxicological risks associated with them.

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