

Research Progresses of Determination of Perfluorinated Compounds in Environmental Water and Solid Samples



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Abstract: Perfluorinated compounds (PFCs), a group of persistent organic pollutants, have been widely detected in environmental media and posed great threats to human health. The researches on environmental pollution and health risk of PFCs have become the hot subject in recent years. The advanced and reliable analytical methods for determination of PFCs are urgently needed since various homologs and isomers of PFCs are detected at trace levels (ng g^{-1} or $\mu\text{g L}^{-1}$) in environment. At present, studies on analytical methods of trace PFCs in environmental samples have been extensively carried out in China and abroad. However, reviews which systematically discuss the sample pretreatment, analytical method, and matrix effect of determination of PFCs in complex environmental matrixes are hardly available. Therefore, the present review highlights the pretreatment methods, matrix effects, and detection techniques (especially isomers) of PFCs in environmental samples (water, sediment, sludge, soil and plant). It hopes that this review will provide valuable references for the environmental researches on PFCs.

Key Words: Perfluorinated compound; Environment sample; Sample pretreatment; Matrix effects; Isomers; Review

1 Introduction

Perfluorinated compounds (PFCs) are a kind of organic contaminants whose hydrogen atoms linked to carbon atoms are replaced by fluorine atoms. PFCs can be divided into four main types of compounds, i.e., perfluoroalkyl carboxylic acids (PFCAs), perfluoroalkyl sulfonic acids (PFASAs), fluorotelomer alcohols (FTOHs), and perfluoroalkyl sulfonamides (FOSAs) according to the types of their functional groups^[1–3]. Because of their excellent properties such as thermostability, chemical stability and surface activity, PFCs are widely used as adhesives, surfactants, lubricants, and so on in industrial, agricultural and commercial applications^[4,5]. Consequently, large amounts of PFCs entered environment^[6–9]

and have been widely detected in various environmental media, e.g. drinking water^[10,11], surface water^[6,12,13], ground water^[11,14], sediment^[6,15,16], soil^[6,17], and so on.

With high bond energy of carbon-fluorine bonds, PFCs are highly stable and difficult to be biologically degraded, especially for perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS) which are hardly degraded in natural environment unless artificial chemical techniques such as ultrasonic irradiation and ultraviolet photocatalytic oxidation are applied^[15,18,19]. PFCs can transfer and diffuse among different environmental media and thus lead to their occurrence in the areas far away from the discharge points, even in the remote Northern and Southern poles^[20]. Meanwhile, PFCs are readily absorbed and bio-accumulated

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in animals and crops, and then pose a serious threat to human health via food chain^[15,21,22]. Nowadays, PFCs have been universally detected in human blood and milk^[15,23–25]. PFCs accumulated in human exhibit various toxicities such as hepatotoxicity, embryotoxicity, reproductive toxicity, neurotoxicity, endocrine toxicity, and even carcinogenicity^[26–28]. Accordingly, the environmental pollution and health concern of PFCs have been becoming one of the hot subjects in environmental science and toxicology^[15,29].

Because PFCs contain lots of homologs and isomers (e.g. dozens of PFOA or PFOS isomers have been observed in environmental media) which are commonly detected at trace levels (ng g⁻¹ or µg L⁻¹ levels) in environment, advanced and reliable analytical methods for determination of PFCs in environment are urgently needed. At present, the studies on analytical methods of trace PFCs in environmental samples have been intensively carried out in China and abroad^[7,30–34]. However, reviews which systematically discuss the sample pretreatment, analytical method, and matrix effect of determination of PFCs in complex environmental matrixes are hardly available. The present review highlights pretreatment methods, matrix effects, and detection techniques (especially isomers) of PFCs in environmental samples (water, sediment, sludge, soil, and plant), which is expected to provide valuable references for the environmental researches on PFCs.

2 Sample pretreatment

2.1 Sample extraction

2.1.1 Water sample extraction

Solid-phase extraction (SPE) has several advantages including short extraction time, less consumption of organic solvents, and combined extraction and clean-up procedure^[35,36], and thus is commonly used for extraction of PFCs in environmental water. The frequently-used SPE columns include weak anion exchange cartridge (WAX), hydrophile-lipophile balance cartridge (HLB), and C₁₈ cartridge, among which WAX cartridge provides the best extraction effect in the extraction of most of PFCs including PFCAs, PFSA and FOSAs. While HLB cartridge provides satisfactory extraction effect only for PFCAs and PFSA with long carbon chains^[37]. In addition, C₁₈ cartridge is less used in recent years because its packing materials contain small amounts of PFCs (46 pg L⁻¹ PFOA and 12 pg L⁻¹ PFOS)^[38], which can remarkably interfere with determination of trace PFCs. Solid phase microextraction (SPME) is a new extraction technique based on SPE which shows several advantages such as solvent-free extraction process and high PFC recoveries. But it is mainly used to determine volatile or semi-volatile organic compounds. Accordingly, when SPME is used to determine PFCs

especially for PFCAs and PFSA, derivatization of these compounds was required^[20,39]. For instance, Monteleone *et al.*^[40] obtained a satisfactory extraction effect (recoveries 84.4%–116.8%, RSD 0.4%–14.5%) of PFCAs in environmental water samples by SPME with headspace analysis and PFCAs derivatization by a mixed solvent of propyl-chloroformate/propanol.

2.1.2 Soil sample extraction

Nowadays ultrasonic extraction (UAE) is a main method for extraction of PFCs in solid samples (soil, plant sediment, and sludge), which exhibits many advantages including high-efficiency extraction, short extraction time and attainable extraction equipment^[41–45]. Pan *et al.*^[45] obtained desirable extraction effects of PFCs in soils (recoveries 81.5%–127.2%) using UAE method with methanol as extractant. Besides UAE method, accelerated solvent extraction (ASE) has been applied for extraction of PFCs in solid samples, which can provide auto and rapid extraction with less organic solvent under high temperature and high pressure conditions. Wang *et al.*^[42] used ASE method to extract PFCAs (C6–C12, C14) and PFSA (C4, C6, C8) in soil samples and obtained a satisfactory detection result (recoveries of 71.2%–119.2% and RSD < 10%). However, ASE method is less used in common laboratories for regular monitoring due to its relatively expensive equipments.

2.1.3 Plant sample extraction

Extraction of PFCs in plant samples usually involves mixed solvents, because single solvent cannot provide satisfactory extraction efficiency. This is attributed to the much more complex matrix components (pigment, cellulose, sugar, and so on) in plant samples compared with soil samples, which can adsorb PFCs or form complexes via hydrogen bond, hydrophobic force, dispersion force, etc.^[46]. Mixed solvents of dichloromethane-methanol (50:50, *V/V*)/ammonium acetate-methanol (99:1, *V/V*) could provide satisfactory extraction effects (average recovery 85%) of PFCAs (C4–C10) and PFSA (C4, C6–C8, C10) in each part (root, shoot, and leaf) of various vegetables (lettuce, tomato) using UAE method^[47]. Furthermore, mixed solvents of acetonitrile and water could also efficiently extract PFCAs (C4–C12) and PFSA (C4, C6, C8, C10) from various vegetables (potato, kidney bean, lettuce and spinach), with the recoveries ranging from 70% to 105% and RSD between 1% and 21%^[33]. PFCs can dissociate into anions in alkali solution, and then react with positively charged tetrabutyl ammonium hydrogen sulfate (TBA) to form ion-pairing, which can be readily extracted by low polar solvent methyl tertbutyl ether (MTBE). At present, the ion-pair extraction (IPE) with NaOH as dissociation agent,

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