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Review

A Review of Recent Advances in Detection and Treatment Technology for Perfluorinated Compounds

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Abstract: Perfluorinated compounds (PFCs) are a novel type of environmental pollutant with a specific structure. PFCs have become a global concern due to their environmental persistence and biotoxicity properties. In this paper, we review the hazardous effects, detection technologies, and treatment methods of PFCs. We present the current status of PFCs pollution in water, the atmosphere, soil, and organisms. Moreover, we show that PFCs have toxic effects, such as hepatotoxicity, neurotoxicity, immunotoxicity, endocrine disruption, and reproductive and developmental toxicity. Six sample pretreatment techniques and four assays for PFCs are listed in this paper. This review focuses on the analysis of the treatment methods for PFCs, such as physical adsorption, microbial degradation, photochemical oxidation, electrochemical oxidation, acoustic oxidation, Fenton oxidation, and so on. We systematically analyze the treatment effects, removal mechanisms, and future directions of various technologies to provide support and suggestions for PFCs pollution control technologies.

Keywords: perfluorinated compounds (PFCs); pretreatment; detection technology; adsorption; photochemical oxidation; electrochemical oxidation

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1. Introduction

Perfluorinated compounds (PFCs) are a special class of synthetic, organic compounds with a special structure. As we all know, PFCs refer to organic compounds in which all the hydrogen elements connected to carbon are replaced by fluorine elements. Fluorine is the most electronegative element, and bound fluorine is one of the most stable elements. Fluorine attracts electrons to itself by chemical bonds, giving polarity and strength to the C–F bond (~110 kcal/mol) [1]. The high-energy C–F bond makes PFCs resistant to hydrolysis, photolysis, and biodegradation, and makes them persist in the environment. In addition, the fluorine atom has three pairs of negatively charged electrons in its external electron shell which do not participate in bonding with other atoms. These unbonded electrons act as protective covers, forming a highly fluorinated system with excellent thermal and chemical stability [2]. Due to the unique structure of PFCs, they have unique physical, chemical, and biological properties, such as high surface activity, heat and acid resistance, hydrophobicity, and oil repellency. These properties make them highly resistant to environmental and biological decomposition [3]. Perfluoroalkyl carboxylic acids (PFCA), perfluoroalkyl sulfonic acid (PFSA), perfluoroalkyl sulfonamide, and other polyfluorinated compounds, such as fluorinated telomeric alcohols, are all examples of polyfluorinated compounds [4]. Among them, perfluorooctane sulfonic acid (PFOS) and perfluorooctanoic acid (PFOA) are the two most widely used PFCs. They can be released into the air directly from pollution sources or indirectly when other PFCs break down.

Since the 1960s, PFCs have been widely used in industrial production and daily life, including textiles, paper, pesticides, food packaging, cosmetics, and other fields. In the process of manufacture, use, and disposal of related chemicals, all kinds of PFCs will be discharged into the environment directly or indirectly. At present, PFCs have been detected in air [5], water [6–10], sediment [11,12], animals [1], and humans [13–15]. Tail water discharge from municipal sewage treatment plants is one of the main ways to introduce PFCs used in household, commercial, and industrial into the water bodies [16,17]. These compounds are distributed all over the world and have environmental persistence, bioaccumulation, and potential harmfulness [18]. Toxicological studies on PFCs showed that these compounds also have toxic effects, such as hepatotoxicity [19,20], neurotoxicity [21,22], immunotoxicity [23,24], endocrine disruption [25,26], and reproductive and developmental toxicity [27,28]. Therefore, in order to ensure environmental safety and protect human health, it is particularly imperative to establish a reliable analysis method and treatment technology for PFCs. This paper briefly describes the pollution status of PFCs, and summarizes research progress on the pretreatment technology, detection methods, and treatment technology of PFCs. This information gives directions for monitoring and controlling pollution, toxicity research, and environmental PFC exposure.

2. The Hazards of PFCs

2.1. Pollution Status of PFCs

Generally, PFCs pollute the environment in two ways. The first is direct entry: industrial production of PFCs and discharge directly into surface water, soil, air, and other environmental media. The production and transportation of PFCs are closely related to the production of PFC pollution sources in the environment. Secondly, indirect entry refers to PFCs precursors found in products or everyday necessities. These precursors enter the atmosphere through volatilization and diffusion into rivers, lakes, and other media, and react to form stable PFCs [29]. Currently, three diagnostic ratios can be used to analyze the main sources of PFCs in the environment, which are PFOS/PFOA, PFOA/PFNA, and PFHpA/PFOA. So et al. [30] described the ratio of PFOS to PFOA in the coastal waters of Hong Kong, South China, and South Korea. They found that only one sample had a value greater than 1, which may suggest that there is an independent potential point source of PFOS near the sampling site. The ratio of PFHpA to PFOA in surface water was proposed by Simcik and Dorweiler [31], which can be used as an indicator of atmospheric PFCs. A ratio of <1 indicates that there is a non-atmospheric source—surface water; and a ratio of >1 indicates the relative importance of atmospheric deposition. Armitage et al. [32] estimated that the PFOA/PFNA ratio of direct emissions from the manufacturing process is 7–15. In general, a ratio of less than seven means that there is no point-source emission of perfluorocarboxylic acid (PFCA) during the manufacturing process, but there are effects from secondary sources, such as the breakdown of volatile precursors [33,34].

In recent years, as people have paid more and more attention to PFCs, more and more studies have shown that PFCs already exist in water, the atmosphere, soil, and organisms. PFCs contain hydrophilic groups of carboxylic acid or sulfonic acid, which usually exist in water in the form of anions that can pollute surface water and groundwater. For example, the range of total PFC concentrations detected in surface water in the Pacific Northwest is 1.5–41 ng/L [35], and the concentration of PFCs in groundwater samples from a city in northern China ranged from undetectable to 64.8 ng/L [36]. In addition, it has been reported that perfluoroalkyl substances (PFAS) in the atmosphere of Northern Greenland have their maximum concentrations in summer; the average concentration range of total PFAS is 1.82–32.1 pg/m^3 [37]. PFCs can also be detected in the soil of fluorosis areas in southwest China. The concentration range is 0.508–6.83 ng/g—mainly perfluorononanoic acid (PFNA) and PFOA [38]. It can be seen that the environment has been polluted by PFCs to varying degrees. This pollution will gradually accumulate or magnify in various ways in organisms. Taniyasu et al. [39] analyzed fish samples in Japanese waters. The presence of PFCs was found in all 78 fish samples examined. PFOS were detected at a high recovery percentage of 100% and a maximum concentration of 7900 ng/mL. Kadar et al. [40] detected PFCs, mainly PFOS and PFOA, in French women's breast milk samples, with concentration ranges of 24–171 and 18–102 pg/mL, respectively. These data on exposure are in the same range as other values that have been reported in European countries.

2.2. Toxicity Analysis of PFCs

With the detection of a large number of PFCs around the world, people have started to worry about whether PFCs in the environment are harmful to human beings. Therefore, scholars from various countries have begun to study the toxicology of PFCs. According to the chemical structure of PFCs, they are highly stable, so it is often difficult for PFCs to be metabolized in organisms. In addition, PFCs are both water-soluble and fat-soluble, so they are easy to accumulate in different tissues of animals and interfere with the normal physiological processes, resulting in toxicity. PFOA serum concentrations reported in occupationally exposed humans were between 428 and 12,000 ng/mL, and between 145 and 3490 ng/mL for PFOS [41]. Terribly, higher concentrations of serum PFOA ($\geq$5.7 ng/mL) and PFOS ($\geq$36.8 ng/mL) are associated with current thyroid disease in the U.S. general adult population [25].

By controlling the cell cycle in vivo, changing the proliferation of peroxidase, and the expression of apoptotic genes, PFOS and PFOA may increase liver weight and even cause hepatocyte death [42]. Johansson et al. [43] found that PFOA and PFOS (1.4 or 21 μmol/kg body weight) can cause developmental neurotoxic defects in mice. These defects are characterized by persistent abnormalities in spontaneous behavior, habitual ability, learning, and memory; and changes in the cholinergic system. Furthermore, astrocytes play an important role in the neurotoxicity caused by PFOS [44,45]. In addition, DeWitt et al. [46] took 3.75 mg PFOA/kg as the lowest observed adverse effect level (LOAEL), proving that PFOA may have immunotoxicity and affect immune organs, immune cells, and immune factors. The mechanism of PFCs immunotoxicity may involve activation of peroxisome proliferator activated receptors-α (PPAR-α), nuclear factor-κB (NF-κB), and the mitochondrial apoptosis pathway [47]. Previous studies on the immunotoxicity mechanism of PFCs are still in the preliminary stage. Therefore, it will be necessary to further explore the immunotoxicity and molecular mechanisms of PFCs in the future. Long-term exposure to PFOS may cause abnormalities of new vivid objects in pregnant animals, such as systemic edema, delayed ossification, weight loss, attention deficit, and acute ventricular heart disease [48–51]. Wang et al. [27] found that 250 μg/L PFOS exposure also impaired the gonadal development of male zebrafish and decreased their sperm activity; the embryos conceived by female zebrafish showed severe deformities, and the final mortality was 100%. The endocrine disrupting effect caused by long-term exposure to low-dose PFCs should not be ignored. Lafuente et al. [52] demonstrated that 0.5 mg/kg PFOS can alter the activity of the hypothalamic–hypophysis–adrenal (HHA) axis. Additionally, PFCs have interference effects on androgen, estrogen, thyroxine, insulin, and so on [53]. In addition, it has been reported that PFC exposure would lead to attention deficit and hyperactivity disorder in American children aged 12–15 years [48]. Kranthi Kumar et al. [54] used computer biology tools to effectively express the toxicity of PFCs and their binding levels to steroid acute regulation (StAR) proteins. This revealed the molecular characteristics of PFC inhibition and the function of mitochondrial membrane destruction, which is helpful to understand other environmental pollutants and health risks. Due to the endocrine disrupting effect of PFCs, there are many potential targets and many health hazards. This is why it is important to do more research and find scientific laws in this area.

3. Detection Techniques for PFCs

3.1. Sample Pretreatment Techniques for PFCs

The accuracy of an established instrumental analysis method depends to a large extent on the pretreatment method used on the sample. The method of pretreatment determines the accuracy and repeatability of the test results, so it can be said that the sample pretreatment for the detection method plays a decisive role. The following introduces the pretreatment methods for PFCs analysis and detection, including liquid–liquid extraction (LLE), solid-phase extraction (SPE), solid–liquid extraction (SLE), ultrasonic extraction (USE), accelerated solvent extraction (ASE), and microwave-assisted extraction. The reported pre-processing and detection methods of PCFs are shown in Table 1.

Table 1. Pre-treatment and detection methods for PFCs.

Substrates	PFCs	Pre-Processing Methods	Detection Methods	Limit of Detection or Limit of Quantitation (LOD/LOQ)	Recovery (%)	Ref.
Sea water	PFOA, PFOS and PFOS precursors	Vortex assisted liquid-liquid microextraction	HPLC-LTQ Orbitrap HRMS	0.7–6 ng/L (LOQ)	95–100	[55]
Food contact material	PFOA, PFOS	Ionic liquid dispersion liquid-liquid extraction	UPLC-MS/MS	PFOA: 0.5 μg/L (LOD); 2 μg/L (LOQ) PFOS: 1 μg/L (LOD); 5 μg/L (LOQ)	86.4–100	[56]
Experiment water	PFBA, PFOA, PFOS and PFDA	Solid phase extraction	HPLC-MS/MS	0.04–0.05 ng/L (LOD) 0.15~0.20 ng/L (LOQ)	94.5–100	[57]
Animal Muscle	PFOA, PFOS and other 11 PFCs	Alkali digestion-Solid phase extraction	HPLC-ESI-MS/MS	0.002–0.032 μg/L (LOD)	80–100	[58]
Fish and shellfish	PFOA, PFOS and other 9 PFCs	Alkali digestion-Solid phase extraction	HPLC-MS/MS	0.16–2.0 ng/g (LOD)	97.6–100	[59]
Experiment water	PFOA, PFNA and other 7 PFCs	Headspace solid phase microextraction	GC-MS	0.08~6.6 ng/L (LOD) 0.17~14.3 ng/L (LOQ)	84.4–100	[60]
Leather	PFOA, PFOS	Soxhlet extraction	UPLC-MS/MS	1.0 μg/L (LOD)	PFOA: 91.6–100 PFOS: 90.0–99.4	[61]
Animal food	PFOA, PFNA and other 14 PFCs	Solid-liquid extraction	HPLC-MS/MS	1–30 pg/g (LOD) 2.5–60 pg/g (LOQ)	88–100	[62]
Sewage Sludge	PFOA, PFOS	Focused ultrasonic solid-liquid extraction	UPLC-MS/MS	0.2 ng/g (LOD)	69–100	[63]
Nonstick coating	PFOS	Accelerated solvent extraction	HPLC-MS/MS	0.4 μg/m^2 (LOD)	93.4–100	[64]
Textile	PFOA	Microwave ultrasonic synergistic extraction	GC-MS	0.088~0.132 μg/mL (LOD)	60–100	[65]
Milk	PFOA, PFNA and other 5 PFCs	Solid phase extraction	GC-MS/MS	4–18 ng/kg (LOQ)	81–100	[66]
Pork	PFOA, PFNA and Perfluoro decanoic acid (PFDA)	Solid phase extraction	LC-MS/MS	0.011–0.08 ng/g (LOD)	89.3–100	[67]
Mussel Fish Liver Fish meat	PFOA, PFOS and other 14 PFCs	Focused ultrasonic solid-liquid extraction	LC-MS/MS	0.1–3.8 ng/g (LOD) 0.1–2.7 ng/g (LOD) 0.2–3.1 ng/g (LOD)	59–100 65–100 67–100	[68]
Animal liver	PFOA, PFNA and other 20 PFCs	Solid-liquid extraction	HPLC-MS/MS	0.05–0.2 μg/kg (LOD) 0.4–0.5 μg/kg (LOQ)	70.3–100	[69]
Reservoir water	PFOA, PFOS and other 12 PFCs	Solid phase extraction	UPLC-MS/MS	0.03–0.48 ng/L (LOD)	51.1–100	[70]
River water	PFOA, PFOS and other 18 PFCs	Solid phase extraction	UPLC-MS/MS	0.002–4 ng/L (LOD) 0.006–12 ng/L (LOQ)	55–94	[71]
Leather	PFOA, PFOS	Ultrasonic extraction	UPLC-MS/MS	0.25 μg/g (LOD)	PFOA: 89.2–100 PFOS: 86.4–96.2	[72]

3.1.1. Liquid–Liquid Extraction (LLE)

Liquid–liquid extraction (LLE) is a classical sample pretreatment technique. The principle is to separate the tested substance from the matrix by using different components with different partition coefficients or solubility in two immiscible solvents [73]. LLE has the advantages of operating at room temperature, simple operating conditions, and convenient operation. Its disadvantages are that it requires more manpower and more organic solvents, it is easy to emulsify and easily causes secondary pollution, and it has low extraction efficiency [74].

To improve extraction efficiency, LLE is usually combined with ultrasonic extraction, solid-phase extraction, and other methods [75]. At present, LLE is mainly used to extract PFOS from solid and semi-solid biological samples (such as animal tissue) and liquid samples (such as water, breast milk, and serum). Concha-Grana et al. [55] extracted PFAS from seawater by vortex assisted liquid–liquid microextraction (VALLME), and then analyzed it directly by liquid chromatography (LC) and high-resolution mass spectrometry (HRMS).

This method only requires a sample of 35 mL and 100 µL of extraction solvent, and has the advantages of rapidity, accuracy, and sensitivity. In order to overcome the shortcomings of the traditional LLE method, Yan et al. [56] reported an ionic liquid dispersion liquid extraction method. In this method, a mere 220 µL of the ionic liquid can be used to extract PFOS and PFOA from a migration solution of food contact materials. Additionally, the use of vortex and centrifugal extraction significantly minimizes the number of organic solvents and reduces pretreatment time.

3.1.2. Solid-Phase Extraction (SPE)

At present, solid-phase extraction (SPE) is widely used in medicine, food, environmental testing, commodity inspection, chemical testing, and other fields [76]. SPE is based on the liquid chromatographic separation principle of selective adsorption and selective elution. Usually, the target compound solution is separated from the solvent by the solid adsorbent. It is then eluted from the solid adsorbent so that the target compound can be separated from the impurities and the analyte with higher purity and the concentration can be obtained [57,77]. The commonly used solid-phase extraction columns include weak anion exchange (WAX) columns, mixed anion exchange (MAX) columns, hydrophilic equilibrium (HLB) columns, and C18 columns. With respect to PFCA and perfluorosulfonic acid (PFSA) molecules with different chain lengths, the WAX column has the best extraction effect. However, the HLB column only provides satisfactory extraction results for PFCA and PFSA with long carbon chains [78,79]. When the carbon chain is short (C4, C5), PFCs have strong acidity, ion exchange is dominant, and the retention on the HLB column is weak. When the length of the carbon chain is long (>C14), the non-polarity of PFCs is enhanced, the ion exchange interaction with the WAX column is weakened, and the non-polar force is dominant. The extraction effect of the HLB column is slightly better than that of the WAX column [80].

Wang et al. [58] used ion-pair liquid–liquid extraction and alkali ablation extraction to treat animal viscera and muscle tissue samples, respectively, and combined them with a solid-phase extraction column for cleanup and then fed them into the sample for analysis. After comparing the extraction results of the WAX and MAX columns, it was found that the WAX column retained about 85% of all 11 PFCs and internal standards. In contrast, the WAX column had reasonable retention of only two types of regulated acids and no retention for the remaining nine PFCs. Pan et al. [59] reported that satisfactory recovery results were also obtained when solid-phase extraction was applied to fish and shellfish samples. The recoveries of the nine analytes were all above 82.0%. Compared with the traditional liquid–liquid extraction method, SPE has higher analyte recovery, a shorter extraction time, less organic solvent consumption, and better separation of analytes and interfering components [81–83]. A new solid-phase extraction method and solid-phase microextraction separation technology were developed based on the optimization of SPE technology. For example, Monteleone et al. [60] extracted PFCA from environmental water samples by headspace solid-phase microextraction after preliminary derivatization with a mixture of propyl chloroformate and propanol. The LOD and LOQ values were between 0.08–6.6 and 0.17–14.3 ng/L, respectively, which can be considered very satisfactory.

3.1.3. Solid–Liquid Extraction (SLE)

Solid–liquid extraction refers to the process in which the solvent enters the solid material and transfers the effective components from the solid phase to the liquid phase, so the solid–liquid extraction method is suitable for the pretreatment of solid samples. Soxhlet extraction is a type of solid–liquid extraction that uses a continuous reflux solvent and the siphon principle to extract samples. Chen et al. [61] compared the three extraction methods of soaking, ultrasonication, and Soxhlet to extract PFOS and PFOA from leather. The experimental results showed that Soxhlet extraction was the most efficient. Methanol is the most commonly used solvent in Soxhlet extraction because PFCs are highly soluble in it and it has a suitable boiling point. Ballesteros et al. [62] used a mixture of tetrahydrofuran

(THF) and water as an extractant to extract PFCs from animal food. After experimental optimization, it was determined that when the volume ratio of tetrahydrofuran to water is 75:25, the extraction effect of PFCs in the sample is the most effective, and the recovery rate is 88–110% after HPLC-MS/MS detection. Although this method has good extractability, it takes a long time and a large amount of solvent, so its efficiency is not high. Soxhlet extraction is not a reliable method for sample pretreatment of PFCs. As the glass instrument used in Soxhlet extraction has a certain adsorption effect on PFCs, it will affect the detection results of PFCs.

3.1.4. Ultrasonic Extraction (USE)

Ultrasonic extraction can make use of the characteristics of ultrasonic wave to increase the kinetic energy of medium molecules [84]. Ultrasonic extraction has been widely used in various detection technologies because of its advantages, such as low temperature functioning, short extraction time, low energy consumption, high extraction efficiency, and simple operation [85]. It is also widely used in PFC detection technology [85,86]. Begley et al. [87] used a mixture of ethanol and water with an equal volume ratio as the solvent for ultrasonic extraction for one hour when detecting the content of PFOA in the paper. The results showed that the recovery of PFOA was between 60 and 75%. Ultrasonic extraction cannot purify complex samples, and other pretreatment techniques need to be used to optimize the method when the samples are very complex. For example, when Chen et al. [88] determined the PFOS in foam fire extinguishing materials, the ultrasonic extraction method with water as solvent was used to extract the sample, and then the solid-phase extraction column was used to purify the sample. Finally, the standard recovery of the determination method reached 93.4%. Besides that, focused ultrasonic solid-liquid extraction (FUSLE) is a low-cost and efficient extraction technology [63]. Using 8 mL of acetonitrile as an extraction solvent and ultrasonic irradiation of 70 W power for 20 s, the two extraction cycles are sufficient to quantitatively extract PFCs from sewage sludge. The detection limit of the target analyte is lower than 0.2 ng/g dw, the relative standard deviation is less than 8%, and the recovery is between 69% and 104%.

3.1.5. Accelerated Solvent Extraction (ASE)

Ultrasonic methods are currently commonly used to extract the sampling membranes of PFCs, but they require a lengthy pretreatment process, making them unsuitable for large quantities of samples. Accelerated solvent extraction (ASE) can complete the extraction, filtration, and purification of compounds in solid and semi-solid samples in a short time. It uses step temperature control and pressure control extraction, which not only greatly shortens the extraction time but also reduces the solvent consumption, and has the advantages of a small matrix effect, high recovery, and reliable reproducibility [89–91]. The optimization of ASE parameters is only related to the selection of extraction solvent, temperature, and time of PFCs separation. ASE can be used to automatically separate analytes, but the main problem with this method is that the equipment is expensive [92].

Some studies have shown that when using the accelerated solvent method to extract PFOA from textiles, toluene should be used as a solvent, and methanol should be used to extract PFOS [93]. Cheng et al. [64] developed a rapid and quantitative method for the determination of PFOS in non-adhesive coatings based on high performance liquid chromatography–tandem mass spectrometry (HPLC-MS/MS) and ASE. The optimization of ASE was achieved by setting the extraction temperature to 100 °C, the pressure to 10.3 MPa, the equilibrium time to 7 min, and repeating the extraction twice with acetonitrile as the extraction solvent. The recovery of PFOS was in the range of 93.4–103%, the relative standard deviation was 0.48–3.59%, and the detection limit was 0.4 $\mu g/m^2$. The method has the advantages of simple sample treatment; rapid determination; and superior accuracy, precision, and recovery.

3.1.6. Microwave-Assisted Extraction

Microwave-assisted extraction is a separation method by which some organic components in solid or semi-solid materials can be effectively separated from the matrix by the action of an electromagnetic field, and the original compound state of the analytical object can be maintained [94]. This method does not need to use a lot of reagents for extraction, so it produces less waste liquid and causes less pollution to the environment [95]. In addition, it is very convenient to heat the extraction solvent. The temperature of the extraction solvent can be easily controlled, and the original solid sample will not be damaged in the extraction process. Therefore, it is a commonly used sample extraction method [96]. He et al. [65] compared the extraction results of PFOA by Soxhlet extraction, reflux, ultrasonic extraction, and microwave-assisted ultrasonic extraction. It was found that the fourth method was the best. Microwave-assisted ultrasonic extraction requires less solvent, the extraction time is the shortest, and the recovery ranges between 60% and 100%.

3.2. Detection Technologies for PFCs

In order to cope with the dynamic industrial production of various PFCs, researchers need to quickly adopt monitoring procedures and analytical techniques that should cover these emerging compounds. At present, the most widely used method for the detection of PFCs is a kind of chromatography with tandem mass spectrometry. Although the instruments required are more expensive, two-stage mass spectrometry can provide more structural information, more analysis modes, less background interference, high selectivity, and sensitivity, and simplify the pretreatment process [97,98]. It is often used in the detection of samples with low levels of harmful substances. This section is mostly about different chromatography–tandem mass spectrometry methods that can be used to analyze and track PFCs.

3.2.1. Gas Chromatography–Tandem Mass Spectrometry (GC-MS/MS)

Chromatography has a strong ability to separate mixtures but a poor ability to characterize compounds. In contrast, mass spectrometry itself has no ability to separate mixtures but can be used to determine the relative molecular masses and chemical structures of compounds, making it an excellent qualitative tool [96]. Therefore, GC-MS is playing an increasingly prominent role in many fields of analytical detection and research, especially in the routine monitoring of many organic compounds as a necessary tool [99]. Li et al. [66] used a nylon 66 membrane syringe to extract PFCA residues from milk, desorbed them, and derivatized them with acetyl chloride–methanol. Finally, GC-MS/MS was used to quantitatively monitor the extracted PFCA residues. Under the optimum conditions, the established method has good linearity (correlation coefficient $\geq$ 0.9996), precision (relative standard deviation $\leq$ 13%), accuracy (recovery > 81%), sensitivity (quantification limit: 4–18 ng/kg), and extraction performance (10 min). Compared with the previously reported methods, this method does not need additional extraction solvents and adsorbents for the extraction, purification, and concentration of trace PFCA. It also has the advantages of simple operation, low cost, fast extraction speed, accurate, and sensitive analysis. Since some PFCAs and PFSAs are not volatile, it is necessary to derive them when using gas chromatography-mass spectrometry (GC-MS). This increases the difficulty of the experiment, increases the detection cost, and produces toxic substances. Therefore, gas chromatography is not suitable for the detection of PFCs with a wide range of content [100].

3.2.2. Liquid Chromatography–Tandem Mass Spectrometry (LC-MS/MS)

The ion source of liquid chromatography—mass spectrometry (LC-MS) can be selected according to its own needs, which can make the analysis process easier [101]. LC-MS can also be used for the analysis and determination of non-volatile compounds, polar compounds, thermally unstable compounds, and high-molecular-weight compounds [102]. Zou et al. [67] prepared a molecularly imprinted phenolic resin adsorbent via a hydrothermal method and applied it to needle-filter extraction combined with LC-MS/MS for rapid extraction and determination of trace PFCs in pork. The method has the advantages of simple equipment (syringe filter), a low amount of adsorbent use (2 mg), a short operation time (6–8 min), a low detection limit (0.011–0.08 ng/g), a reasonable recovery (>89.3%), and satisfactory intra-day (≤4.0%) and inter-day (≤8.6%) precision. Zabaleta et al. [68] used LC-MS/MS to determine PFCs in muscle tissue, fish muscle tissue, and liver samples. For liver, mussel, and fish muscle samples, the apparent recoveries were 65–100%, 59–100%, and 67–100%, respectively; and the method detection limits (MDL) were 0.1–2.7 ng/g, 0.1–3.8 ng/g, and 0.2–3.1 ng/g, respectively.

3.2.3. High Performance Liquid Chromatography–Tandem Mass Spectrometry (HPLC-MS/MS)

High performance liquid chromatography–tandem mass spectrometry (HPLC-MS/MS) has high sensitivity and selectivity, can quantify the target, and is suitable for the detection and analysis of low-concentration toxic and harmful substances. Due to its simple pretreatment process, it has a significant advantage in the analysis of low content residues of harmful substances [103]. He et al. [69] developed a method for the simultaneous determination of 20 PFASs in animal liver using HPLC-MS/MS, and acceptable linearity was obtained for all 20 PFASs in the range of 0.1–10 μg/L with correlation coefficients greater than 0.9995. The LOD and LOQ were 0.05–0.2 and 0.4–0.5 μg/kg, respectively. The recoveries were greater than 70.3%. The method is simple, rapid, sensitive, and accurate; and it has the advantages of low solvent consumption and effective matrix effect correction. It can be widely used for the determination of PFAS in animal samples. He et al. [57] used fluorinated functionalized paper-based solid-phase extraction (Fp-SPE) coupled with HPLC-MS/MS to determine PFCs. Fluorinated functionalized paper (Fp) exhibited excellent extraction performance for PFCs due to fluorine affinity interactions and had the advantages of biodegradability, simplicity of operation, and low cost. The method can effectively reduce matrix effects and achieve good LOD (0.04–0.05 ng/L) and LOQ (0.15–0.20 ng/L) values. This means that it can be used to analyze PFCs in water with high reproducibility and sensitivity.

3.2.4. Ultra-Performance Liquid Chromatography–Tandem Mass Spectrometry (UPLC-MS/MS)

Ultra-performance liquid chromatography (UPLC) has higher sensitivity and better separation than high performance liquid chromatography (HPLC), and the overall design of the instrument is better [104]. Li et al. [70] extracted 12 anionic PFCs from reservoir water using a cationic F-TMU-66^{+}Cl^{-}/polyvinylidene fluoride metal–organic framework (MOF) mixed matrix membrane (F-TMU-66^{+}Cl^{-}/PVDF MMM) as a solid-phase extraction adsorbent. The extraction process of F-TMU-66^{+}Cl^{-}/PVDF MMM is simple and stable, and the detection scheme has high sensitivity, stability, and selectivity. The method is practical for the ultra-trace analysis of anionic PFCs in water. The ultra-low detection limit in the range of 0.03~0.48 ng/L is provided by combining it with UPLC-MS/MS. Onghena et al. [71] investigated the performance of analyzing 18 PFCs in water samples using UPLC-MS/MS. UPLC-MS/MS was performed using a Zorbax C18 column (50 mm × 2.1 mm, 1.8 m) with gradient elution using ammonium formate and methanol as mobile phases. UPLC-MS/MS is better than traditional LC-MS/MS for identifying PFCs in water samples. It has higher sensitivity and better resolution, and it also speeds up the analysis process.

4. Treatment Technologies for PFCs

4.1. Physical Methods

4.1.1. Adsorption

Physical adsorption technology is the focus of early scientific research, and the treatment of PFCs in water has the characteristics of simple operation, low cost, high efficiency, and so on. Table 2 summarizes the related studies on the removal of PFCs by adsorption. The commonly used adsorbents can be roughly divided into four categories: activated carbon, natural minerals, ion exchange resins, and carbon nanotubes. It can be seen that there are many studies on the adsorption of PFOS and PFOA by commercial activated carbon (AC), but the available data on other PFCs is limited. For activated carbon adsorbents, the smaller the particle size and pore size, the larger the specific surface area, and the more surface functional groups, the more obvious the adsorption effect. Therefore, compared with granular activated carbon (GAC), powdered activated carbon (PAC) shows superior adsorption performance [105]. To improve the recovery of activated carbon, Meng et al. [106] made magnetic activated carbon (MAC) from Fe_3O_4 and PAC to adsorb PFCs. This retained the adsorption capacity and increased the magnetic separation performance. In addition to electrostatic attraction, there is also surface complexation and hydrogen bonding in the adsorption of PFCs by natural minerals, so it also shows a good adsorption effect. Wang and Shih [107] found that it takes 48 h for PFOS and PFOA to reach equilibrium adsorption on alumina. Moreover, the larger adsorption capacity of PFOS can be attributed to the higher affinity of sulfonate functional groups for the alumina surface. In the study of the adsorption of PFOS and PFOA by boehmite (AlOOH), Wang et al. [108] found that the adsorption amounts of PFOS and PFOA were 2–3 times higher than that of alumina due to the abundant hydroxyl groups on the surface of boehmite. An ion exchange resin can remove pollutants by exchanging ions with the same electrical properties in an aqueous solution. In this process, the hydrophobicity of the resin plays an important role. Zaggia et al. [109] investigated the adsorption properties of A532E (highly hydrophobic), A520E (generally hydrophobic), and A600E (non-hydrophobic) anion exchange resins for PFOA and PFOS in drinking water. The adsorption equilibrium capacities of the three resins for PFOA were 142.1, 134.7, and 125.2 mg/g, respectively, and the adsorption equilibrium capacities for PFOS were 260.5, 210.4, and 186.2 mg/g, respectively. In addition, they also observed that PFOA and PFOS can form macromolecular aggregates in the inner pores of resin particles. This indicates that the removal mechanism may involve the complex mechanisms of single-molecule ion exchange and molecular aggregate retention. Carbon nanotubes (CNTs) are effective adsorbents to remove PFOS from water. Chen et al. [110] studied the adsorption on willow charcoal (W400), maize straw-origin ash (MA), and single-walled carbon nanotubes (SWCNT) for PFOS. The results of adsorption kinetics showed that CNTs reached equilibrium within 2 h, which was much faster than carbon (384 h) and ash (48 h) (see Table 2). The results of adsorption isotherms show that the adsorption capacity of CNTs is higher than 700 mg/g, and the adsorption capacity of carbon is lower than 170 mg/g. Deng et al. [111] found that the adsorption of PFCs with the same functional group on CNTs increases with the increase in chain length, but the adsorption amount (surface area normalization) of CNTs containing hydroxyl and carboxyl groups is much lower than that of the original CNTs, indicating that hydrophobic interaction is dominant. However, Bei et al. [112] found that the adsorption amounts of PFOS on CNTs are MWCNTs-Pri < MWCNTs-COOH < MWCNTs-OH (see Table 2). This is due to the obvious difference in\surface area of three kinds of multi-walled carbon nanotubes (MWCNT). It can be seen that the physical structure of carbon nanotubes plays an important role.

Table 2. Adsorption of PFCs by different adsorbents.

PFCs	Concentration	Adsorbents	Dosages	Balance Time	Adsorption Isotherms		Ref.
					Langmuir Constants $q_e = \frac{K_L q_m C_e}{1+K_L C_e}$	**Freundlich Constants** $q_e = K_F C_e^n$	
PFOS	50 mg/L	GAC	0.1 g/L	168 h	K_L = 39 L/mmol, q_m = 0.37 mmol/g	K_F = 0.43 ($mmol^{1-n}L^n$/g), n = 0.18	[105]
		PAC	0.1 g/L	4 h	K_L = 55 L/mmol, q_m = 1.04 mmol/g	K_F = 1.27 ($mmol^{1-n}L^n$/g), n = 0.18	
		Anion exchange resin AI400	0.1 g/L	168 h	K_L = 69 L/mmol, q_m = 0.42 mmol/g	K_F = 0.52 ($mmol^{1-n}L^n$/g), n = 0.17	

Table 2. *Cont.*

PFCs	Concentration	Adsorbents	Dosages	Balance Time	Adsorption Isotherms		Ref.
					Langmuir Constants $q_e=\frac{K_L q_m C_e}{1+K_L C_e}$	**Freundlich Constants** $q_e=K_F C_e^n$	
PFOA	50 mg/L	GAC	0.1 g/L	168 h	$K_L = 18$ L/mmol, $q_m = 0.39$ mmol/g	$K_F = 0.47$ ($\mathrm{mmol}^{1-n}\mathrm{L}^n$/g), $n = 0.28$	
		PAC	0.1 g/L	4 h	$K_L = 69$ L/mmol, $q_m = 0.67$ mmol/g	$K_F = 0.83$ ($\mathrm{mmol}^{1-n}\mathrm{L}^n$/g), $n = 0.20$	
		Anion exchange resin AI400	0.1 g/L	168 h	$K_L = 69$ L/mmol, $q_m = 2.92$ mmol/g	$K_F = 3.35$ ($\mathrm{mmol}^{1-n}\mathrm{L}^n$/g), $n = 0.13$	
PFOS	100 µg/L	alumina	10 g/L	48 h	$K_L = 0.0587$ L/µg, $q_m = 0.252$ µg/m^2	$K_F = 0.0400$ (µg/m^2)(µg/L)$^{-n}$, $n = 0.398$	[107]
PFOA	100 µg/L	alumina	10 g/L	48 h	$K_L = 0.00908$ L/µg, $q_m = 0.157$ µg/m^2	$K_F = 0.00239$ (µg/m^2)(µg/L)$^{-n}$, $n = 0.772$	
PFOS	200 µg/L	boehmite	5 g/L	48 h	$K_L = 0.00622$ L/µg, $q_m = 0.877$ µg/m^2	$K_F = 0.0139$ (µg/m^2)(µg/L)$^{-n}$, $n = 0.678$	[108]
PFOA	200 µg/L	boehmite	5 g/L	48 h	$K_L = 0.00512$ L/µg, $q_m = 0.633$ µg/m^2	$K_F = 0.0155$ (µg/m^2)(µg/L)$^{-n}$, $n = 0.559$	
PFOS	100 mg/L	W400	0.2–1.2 g/L	384 h	$K_L = 0.010$ L/mg, $q_m = 91.6$ mg/g	$K_F = 5.23$ (mg/g)(mg/L)$^{-n}$, $n = 0.492$	[110]
		MA	0.2–1.2 g/L	48 h	$K_L = 0.012$ L/mg, $q_m = 811$ mg/g	$K_F = 26.8$ (mg/g)(mg/L)$^{-n}$, $n = 0.571$	
		SWCNT	0.2–1.2 g/L	2 h	$K_L = 0.044$ L/mg, $q_m = 712$ mg/g	$K_F = 122$ (mg/g)(mg/L)$^{-n}$, $n = 0.324$	
PFOA	0.5 mM	SWCNT	0.2 g/L	15 h	-	$K_F = 0.259$ ($\mathrm{mmol}^{1-n}\mathrm{L}^n$/g), $n = 0.391$	[111]
PFOS	0.5 mM	SWCNT	0.2 g/L	15 h	-	$K_F = 1.605$ ($\mathrm{mmol}^{1-n}\mathrm{L}^n$/g), $n = 0.402$	
PFHxS	0.5 mM	SWCNT	0.2 g/L	15 h	-	$K_F = 157.1$ ($\mathrm{mmol}^{1-n}\mathrm{L}^n$/g), $n = 1.504$	
PFHxA	0.5 mM	SWCNT	0.2 g/L	15 h	-	$K_F = 0.054$ ($\mathrm{mmol}^{1-n}\mathrm{L}^n$/g), $n = 0.298$	
PFBS	0.5 mM	SWCNT	0.2 g/L	15 h	-	$K_F = 5.887$ ($\mathrm{mmol}^{1-n}\mathrm{L}^n$/g), $n = 1.539$	
PFBA	0.5 mM	SWCNT	0.2 g/L	15 h	-	$K_F = 0.065$ ($\mathrm{mmol}^{1-n}\mathrm{L}^n$/g), $n = 0.597$	
PFOA	0.5 µM	MWCNTs-Pri	50 mg/L	-	$K_L = 12.98$ L/mmol, $q_m = 1.18$ mmol/g	$K_F = 1.56$ ($\mathrm{mmol}^{1-n}\mathrm{L}^n$/g), $n = 2.29$	[112]
		MWCNTs-COOH	50 mg/L	-	$K_L = 13.39$ L/mmol, $q_m = 1.54$ mmol/g	$K_F = 2.05$ ($\mathrm{mmol}^{1-n}\mathrm{L}^n$/g), $n = 2.30$	
		MWCNTs-OH	50 mg/L	-	$K_L = 11.58$ L/mmol, $q_m = 1.85$ mmol/g	$K_F = 2.47$ ($\mathrm{mmol}^{1-n}\mathrm{L}^n$/g), $n = 2.19$	
PFOS	100 mg/L	BAC	0.1 g/L	24 h	$K_L = 52.2$ L/mmol, $q_m = 2.20$ mmol/g	$K_F = 3.20$ ($\mathrm{mmol}^{1-n}\mathrm{L}^n$/g), $n = 3.41$	[113]
PFOA	81 mg/L	BAC	0.1 g/L	24 h	$K_L = 157.1$ L/mmol, $q_m = 1.03$ mmol/g	$K_F = 1.34$ ($\mathrm{mmol}^{1-n}\mathrm{L}^n$/g), $n = 5.28$	
PFOS	50 mg/L	GAC	1 g/L	-	$K_L = 0.124$ L/mg, $q_m = 236.4$ mg/g	$K_F = 60.9$ (mg/g)(mg/L)$^{-n}$, $n = 0.289$	[114]
PFOA	50 mg/L	GAC	1 g/L	-	$K_L = 0.038$ L/mg, $q_m = 112.1$ mg/g	$K_F = 11.8$ (mg/g)(mg/L)$^{-n}$, $n = 0.443$	
PFBS	50 mg/L	GAC	1 g/L	-	$K_L = 0.034$ L/mg, $q_m = 98.70$ mg/g	$K_F = 9.3$ (mg/g)(mg/L)$^{-n}$, $n = 0.463$	

Table 2. *Cont.*

PFCs	Concentration	Adsorbents	Dosages	Balance Time	Adsorption Isotherms		Ref.
					Langmuir Constants $q_e=\frac{K_L q_m C_e}{1+K_L C_e}$	Freundlich Constants $q_e=K_F C_e^n$	
PFOS	50 mg/L	AER	2 g/L	2 h	-	K_F = 7300 (mg/g)(mg/L)$^{-n}$, n = 0.90	[115]
		GAC	2 g/L	24 h	-	K_F = 120 (mg/g)(mg/L)$^{-n}$, n = 0.29	
PFOA	50 mg/L	AER	2 g/L	2 h	-	K_F = 200 (mg/g)(mg/L)$^{-n}$, n = 0.97	
		GAC	2 g/L	24 h	-	K_F = 63 (mg/g)(mg/L)$^{-n}$, n = 0.35	

In addition to the properties of the adsorbent material itself, there are various factors affecting the physical adsorption of PFCs, including pH value, anions, and cations in water, and the types of PFCs. The pH can affect the adsorption effect by influencing the electrostatic interaction between PFCs and the adsorbent. For example, Wang and Shih [107] observed that an increase in pH value led to a decrease in the adsorption of PFOS and PFOA on alumina. This may have been due to the decrease in electrostatic interaction. However, Yang et al. [12] found that the adsorption of PFOA by river sediments was not significantly affected by the pH value and ionic strength of the solution. This indicates that electrostatic interaction was not the main reason for the adsorption. They also found that the increase in the ionic strength of four cations (Na^+, K^+, Mg^{2+}, and Ca^{2+}) led to a decrease in the adsorption of PFOS and PFOA. This may have been due to the compression of the electric double layer, and both Ca^{2+} and Mg^{2+} can be bridged with PFOA anions, whereas PFOS can only be bridged by Ca^{2+}. On the contrary, in the presence of anions, MWCNTs tend to aggregate and the surface for PFOS adsorption is reduced, resulting in a 15% reduction in the PFOS removal rate. In the presence of the cations Cu^{2+} and Pb^{2+}, the removal rate of PFOS increases rapidly with the increase in metal concentration. The enhanced adsorption of PFOS under high concentrations of Cu^{2+} and Pb^{2+} is attributed to the further adsorption of PFOS by metal cations [112]. Du et al. [113,116] found that divalent cations in wastewater can promote the adsorption of PFCs on bamboo activated carbon (BAC), whereas dissolved organic matter (DOM) is not conducive to the adsorption process. In addition, Ochoa-Herrera and Sierra-Alvarez [114] demonstrated that the adsorption of PFOS on GAC is stronger than that of PFOA and perfluorobutane sulfonic acid (PFBS) (see Table 2), which indicates that the chain length and the properties of functional groups of fluorocarbons affect the adsorption process.

Adsorption technology can effectively remove PFCs in a wide concentration range, and it has the advantages of simple operation, low cost, and low energy consumption. Future research on adsorption should focus on how to reuse the adsorption materials and how to release the adsorbed PFCs safely so as to avoid secondary pollution.

4.1.2. Membrane Separation

Membrane separation is a method of selectively separating a mixture based on the ability of different particle sizes in the solution to flow through the membrane. The pore size of each membrane is different, and the substances that can be separated are also different. The common membranes used for the separation of perfluorinated compounds include reverse osmosis (RO) membranes and nanofiltration (NF) membranes [117]. Tang et al. [118] studied the feasibility of using RO membrane to treat PFOS wastewater and found that the PFOS rejection rate reached more than 99% in a wide range of feed concentrations (0.5–1500 ppm). There is no clear relationship between membrane flux and original membrane potential, but it decreases as PFOS concentration increases. The flux of loose RO membrane decreased, but the flux of dense RO membrane remained stable. Using a suitable membrane, the PFOS concentration can be reduced while the flux is stable, and a multistage membrane array can be designed to further improve the removal efficiency. Their next study showed that the rejection rate of the NF membrane (90–99%) was lower than that of the RO membrane [119]. This is consistent with the fact that the NF membrane has larger pores and a thinner interception layer. Pramanik et al. [120] investigated the efficiency of GAC, ultrafiltration (UF), and NF treatment in removing PFOS and PFOA from lake water. The effectiveness was ranked as NF > GAC > UF, and the lower removal rate of UF was due to the larger pore size of the membrane. Lower pH increases the electrostatic repulsion between the solvent and the membrane. This facilitates the removal of PFOS/PFOA, and the organic matter in the solution competes for the pores of the membrane, leading to lower retention of PFOA/PFOS—the effect of protein is greater. Liu et al. [121] integrated a microwave catalytic reaction into ceramic membrane filtration to remove PFOA in wastewater. A Fenton-like reaction on the reactive membrane, assisted by microwaves, broke down approximately 65.9% of PFOA. As microwaves penetrate through to the surface and form nano-bubbles there, issues with membrane fouling and scaling could be fixed.

Membrane separation is still a physical method and will not affect the structures of PFCs. Using this method, not only is the wastewater purified, but the raw materials are recovered as well. However, in practice, the membrane fouling will cause the treatment efficiency to decrease, so it can be combined with other treatment methods. Moreover, the composition of the actual wastewater is complex, in which organic matter dissolved in the wastewater can interfere with the membrane flux due to the particle size, thereby reducing the perfluorinated compound removal rate. In reality, pretreatment should be used to reduce the impacts of other organic compounds.

4.2. Chemical Methods

The physical removal method only transfers PFCs from one medium to another, realizing the effect of material separation. However, the chemical properties of pollutants cannot be changed because the C–F bond cannot be broken fundamentally. It is now generally accepted that the chemical degradation of PFCs is an effective means to reduce their toxicity and persistence. Several chemical treatment methods for PFCs will be described below, mainly photochemical oxidation, electrochemical oxidation, sonochemical oxidation, Fenton oxidation, and other chemical methods. The relevant parameters and degradation effects of several common chemical methods for degrading PFCs are shown in Table 3.

Table 3. Treatment effects of several chemical methods for degrading PFCs.

Methods	PFCs	Concentration	pH	Temperature	Time	Rate Constant	Removal Degree	Defluorination Degree	Mineralization Degree	Ref.
Photochemistry	PFOA	1.35 mM	-	-	72 h	-	89.5%	95.9%	-	[122]
	PFOS	40 µM	-	36–46 °C	10 d	-	68%	-	-	[123]
	PFOA	50 mg/L	3	25 °C	15 min	0.514 h^{-1}	99.9%	22.4%	-	[124]
	PFOA	20 mg/L	4.6	-	28 d	-	97.8 ± 1.7%	12.7 ± 0.5%	-	[125]
Electrochemical	PFOA	100 mg/L	5	25 °C	2.5 h	1.158 h^{-1}	95.11 ± 3.9%	75.7 ± 2.8%	-	[126]
	PFOA	50 mg/L	3	32 °C	1.5 h	2.568 h^{-1}	100%	60%	-	[127]
	PFOA	50 mg/L		32 °C	2 h	1.930 h^{-1}	99%	63.8%	-	[128]
Sonochemical	PFOS	100 µM	-	30 °C	5.5 h	-	-	100%	55.8%	[129]
	PFOS	10 mg/L	4.8	20 °C	1 h	0.960 h^{-1}	60%	-	-	[130]
	PFOA	10 mg/L	4.7	20 °C	1 h	1.920 h^{-1}	85%	-	-	
Fenton	PFOA	100 µg/L	3.5	20 ± 2 °C	2.5 h	-	89%	-	-	[131]
	PFOA	20 µM	3	-	5 h	-	100%	53.2%	-	[132]
	PFOA	50 mg/L	5.0	-	5 min	-	95%	-	50%	[133]
	PFOA	20 mg/L	3	-	2 h	1.600 h^{-1}	99%	-	91%	[134]
Ozonation	PFOS	50 µg/L	11	25 °C	4 h	0.602 h^{-1}	85%	-	-	[135]
	PFOA	50 µg/L	11	25 °C	4 h	0.753 h^{-1}	90%	-	-	

4.2.1. Photochemical Oxidation

Photochemical oxidation is the process of degrading pollutant molecules into harmless substances by irradiating them with light. Hori et al. [122] decomposed PFOA in water for the first time by a photochemical method, and PFOA was degraded by 89.5% after direct photolysis for 72 h. Subsequently, Yamamoto et al. [123] confirmed for the first time that PFOS could be degraded by 68% after UV irradiation for 10 days. As direct photolysis of PFCs is time-consuming and the effect is unstable, photocatalytic degradation technology has been developed. Ochiai et al. [136] compared the effects of direct photolysis and TiO_2 photocatalysis on the degradation of PFOA and observed that TiO_2 photocatalysis was six times faster than direct photolysis of PFOA. Chen et al. [124] showed that the rate constant for the decomposition of PFOA by the UV/Pb-TiO_2 system was 0.5136 h^{-1}, which was 32.5 times higher than that of the UV/TiO_2 system. Moreover, the degradation rates of the two systems differ significantly: 99.9% for the former and 18.3% for the latter. Figure 1 shows the process in which PFOA is decomposed by the UV/Pb-TiO_2 system. The assumed reaction equations are as follows.

$$TiO_2 + hv \rightarrow TiO_2(h^{+vb} + e^{-cb}) \quad (1)$$

$$TiO_2(h^{+vb}) + H_2O \rightarrow TiO_2 + \bullet OH + H^+ \quad (2)$$

$$TiO_2(e^{-cb}) + O_2 \rightarrow TiO_2 + \bullet {O_2}^- \quad (3)$$

$$\bullet {O_2}^- + H_2O \rightarrow \bullet HOO + OH^- \quad (4)$$

$$2HOO\bullet \rightarrow H_2O_2 + O_2 \quad (5)$$

$$H_2O_2 + TiO_2(e^{-cb}) \rightarrow \bullet OH + OH^- + TiO_2 \quad (6)$$

$$C_7F_{15}COOH + \bullet OH \rightarrow C_7F_{15}COO\bullet + H_2O \quad (7)$$

$$C_7F_{15}COO\bullet \rightarrow C_7F_{15}\bullet + CO_2 \quad (8)$$

$$\bullet C_7F_{15} + \bullet OH \rightarrow C_7F_{15}OH \quad (9)$$

$$C_7F_{15}OH \rightarrow C_6F_{13}COF + F^- + H^+ \quad (10)$$

$$C_6F_{13}COF + H_2O \rightarrow C_6F_{13}COOH + F^- + H^+ \quad (11)$$

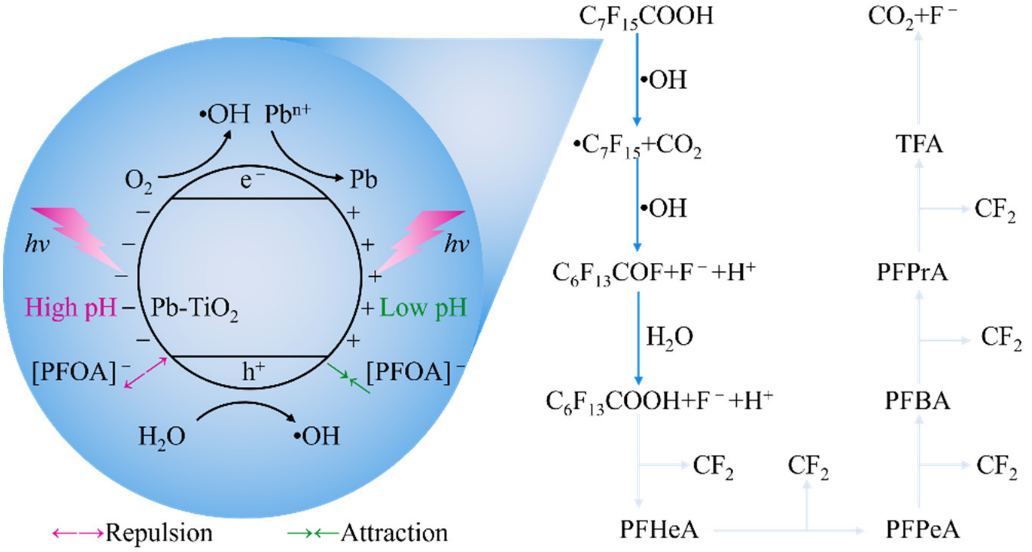

Figure 1. Photocatalytic pathways of PFOA decomposition. Reprinted with permission from reference Mengjia Chen, Shanglien Lo, Yuchi Lee, Jeff Kuo, Chunghsin Wu, 2016, [124].

As shown in Equations (1)–(6), under the action of photocatalysis, oxygen can inhibit the recombination of photogenerated electron–hole pairs and produce additional •OH in the UV/Pb-TiO_2 system, thus accelerating the decomposition and defluorination of PFOA. Additionally, adding Pb to TiO_2 particles could make it more photoactive because Pb could act as an electron trap and prevent electron–hole pairs from recombining.

In addition, Taniyasu et al. [137] showed that even PFASs, which are considered non-degradable in the environment, may undergo photodegradation under intense solar radiation. Long-chain PFCAs, PFSAs, fluorotelomer alcohols (FTOHs), fluorotelomer unsaturated carboxylic acids (FTUCAs), and fluorotelomer carboxylic acids (FTCAs) can degrade to short-chain PFASs such as perfluorobutyric acid (PFBA) and perfluorobutane sulfonic acid (PFBS), but short-chain compounds remain stable even after 106 days of exposure to the environment. Liu et al. [125] found that PFOA can be degraded by Fe(III) under solar radiation. This result provides a natural attenuation process that may be ignored. Photoinduced electron transfer from PFOA to Fe(III) or a new pathway involving •OH attack, both of which result in the formation of organic free radicals that could be used to catalyze the decomposition reaction. Due to the rich content of Fe(III) in natural water and easy access to sunlight, this study provides an innovative and interesting way to develop a new generation of feasible, sustainable, and cost-effective in situ remediation of PFCs-contaminated water.

4.2.2. Electrochemical Oxidation

In the process of electrochemical oxidation of PFCs, the anodic material is the main factor affecting the oxidation effect. Anodic electrode materials need high oxygen evolution potentials and stability. This is because a large amount of oxygen will be released, resulting in low current efficiency and the inability to carry out effective reactions. The degradation rate and defluorination rate of PFOA by a partial anode electrode under optimal conditions are shown in Figure 2 [138]. In addition to the electrode materials, other factors also affect the oxidation of PFCs, such as electrode distance, current density, and the initial pH of the solution. At the same current density, the effect of electrode gap distance on PFOA degradation is similar to that of electrode voltage. Ma et al. [126] changed the plate spacing to 5, 10, 15, and 20 mm; and the corresponding PFOA degradation rates were 95.11 ± 3.9%, 89.05 ± 1.1%, 85.23 ± 1.4%. and 67.92 ± 3.8%, respectively. This shows that at a certain distance, the shorter the distance between electrodes, the higher the degradation efficiency. The longer the electrode distance is, the longer the electrolysis time is, due to the longer diffusion distance. Zhuo et al. [127] investigated the electrochemical oxidation of 1H,1H,2H,2H-perfluorooctane sulfonic acid (6:2 FTS). They found no degradation of 6:2 FTS on $Ti/SnO_2–Sb_2O_5–Bi_2O_3$ anodes at a low current density of 1.42 mA/cm^2, and the degradation rate increased when the current density was in the range of 4.25–6.80 mA/cm^2. The current density affects the degradation and defluorination rates of PFOA by influencing the electron transfer capacity and hydroxyl radical generation of the electrolytic system. In general, the removal rate of PFOA increases with the increase in current density, and the acidic condition is more conducive to the electrochemical degradation of PFOA. The reason is that oxygen evolution cannot easily occur under acidic conditions. Therefore, the oxygen evolution potential is higher, so the yield of •OH is higher. Under alkaline conditions, OH^- will lose electrons on the electrode surface under the action of electrostatic force and compete with $CF(3CF_2)_6COO^-$ anions for active sites, thererby inhibiting its decarboxylation [127]. Zhuo et al. [128] also revealed the reaction mechanism of electrochemical oxidative degradation of PFOA. The carboxyl group in PFOA transfers an electron to the anode at a potential of 3.37 V (vs. saturated calomel electrode, SCE) to decompose PFOA. The PFOA radical is then decarboxylated to form a perfluorinated heptyl radical, which will undergo a defluorination reaction with $•OH/O_2$. The oxidation mechanism of PFOA at the Ti/SnO_2-Sb-Bi electrode is shown in Figure 3.

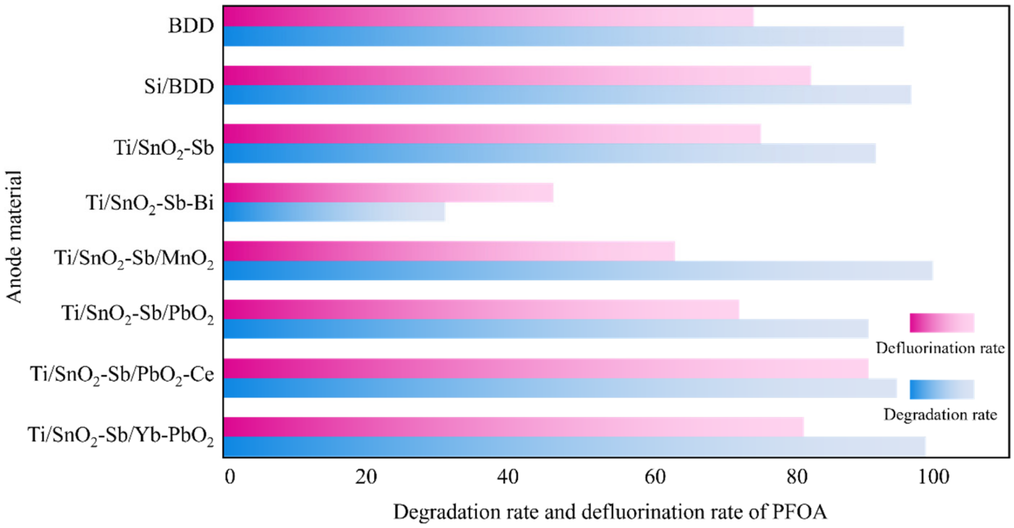

Figure 2. Oxidation effect of PFOA with different anodes.

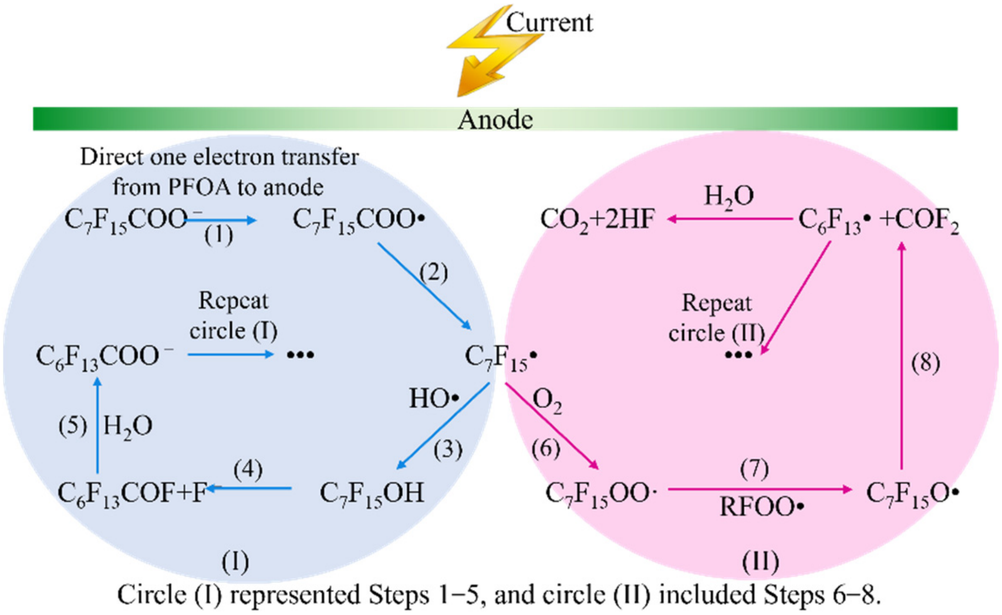

Figure 3. Oxidation mechanism of PFOA on the Ti/SnO_2−Sb−Bi electrode. Reprinted with permission from reference Qiongfang Zhuo, Shubo Deng, Bo Yang, Jun Huang, Gang Yu, 2011, [128].

At present, the mechanism of electrochemical removal of PFCs in water is not fully understood. The removal efficiency is easily influenced by the pH, target concentration, and different ions in the wastewater. In order to remove PFCs from water, we must understand the electrochemical removal pathway, the reaction interface mechanism, the catalyst's structure, and its oxidation performance at the molecular level. Electrochemical oxidation is an advanced oxidation technology used to effectively remove PFCs from water. However, anode materials still have problems, such as high cost, low activity, and short service life. Therefore, it is especially critical to create anode materials with high efficiency, affordability, and high stability. Future research on electrochemical oxidation technology must focus on how to handle low-concentration pollutants while also reducing treatment time, extending electrode life, and treating them with a catalyst addition or a changed electrode.

4.2.3. Sonochemical Oxidation

When acoustic waves propagate through a medium such as water, they usually generate acoustic flow and acoustic cavitation. Cavitation has been shown to be the main precursor for the oxidation or pyrolysis of organics, a method known as sonochemistry [129]. The performance of sonochemical degradation of PFCs may be influenced by factors such as sound frequency, coexisting organics, and inorganic ions. Rodriguez-Freire et al. [129] showed that the use of mega-frequency sound waves can effectively carry out sonochemical treatment of high concentrations of PFOS. The sound frequency affects the available interface sites for molecular adsorption, the oscillation frequency of bubbles, and the intensity of bubble rupture. Compared with frequencies of 25 and 500 kHz, the formation rate of •OH at 1 MHz frequency is 15.2 and 4.1 times higher, respectively, and the degradation rate of PFOS is higher, under mega-frequency sound waves. Cheng et al. [139] found that organic components can reduce the sonochemical degradation rate of PFOS and PFOA by competing for adsorption sites on the bubble-water interface or reducing the average interface temperature. Among them, volatile organic compounds (VOCs) play a major role, whereas the effect of dissolved natural organic compounds (DOM) is not significant. In addition, they also found that there is a Hofmeister effect on the degradation rate of PFOS and PFOA by anions in the range of 1–10 mM [140]. The order of influence is $ClO_4^- > NO_3^- \sim Cl^- \geq MQ > HCO_3^- \sim SO_4^{2-}$, and the influence of common cations of 5 mM can be ignored. When Moriwaki et al. [130] used sonochemistry to degrade PFOS and PFOA, they found that PFOA was degraded approximately twice as fast in argon as in the air. The presence of argon produced higher temperatures and enhanced reaction yields during the reaction, suggesting that the reaction was mainly promoted by thermal decomposition. In addition, no degradation of PFOS and PFOA was observed when the Fenton reagent was added to the PFOS and PFOA solutions. The results show that PFOS and PFOA, in order to be broken down by ultrasound, need to be attacked by OH and be in a high-temperature and high-pressure environment.

4.2.4. Fenton Oxidation

As the C–F bond of PFCs has strong stability, it is generally considered that •OH alone cannot effectively decompose PFCs. However, •OH still plays an instrumental role in the oxidation of PFCs by Fenton. Mitchell et al. [131] degraded 89% of PFOA in 150 min using 1 M H_2O_2 and a 0.5 mM Fe^{3+} composition system. The removal rate was reduced to 24% after adding isopropanol to remove the •OH. This shows that •OH is a necessary condition for PFOA degradation in the Fenton system. Tang et al. [132] investigated the degradation of PFOA by the UV–Fenton method and found that it was divided into two stages: degradation and defluoridation, as shown in Figure 4. In the first stage (<1 h), UV irradiation and Fe^{2+} together triggered the decomposition of H_2O_2 to produce •OH. •OH rapidly degraded PFOA with a removal rate of about 90% and a defluoridation rate of about 35.8%. In the second stage (>1 h), H_2O_2 was almost completely decomposed. At this time, Fe^{3+} generated by the first stage and UV acted simultaneously to continue to degrade the residual PFOA. The removal rate was nearly 100%, and the defluorination rate increased to 53.2%. Santos et al. [141] proposed a method for combining PFOA solutions with humic acid ([HA] = 600 mg/L) and Fenton reagent ($[Fe^{3+}]$ = 3 mmol/L, $[H_2O_2]$ = 165 mmol/L) to PFOA solutions. It was found that HA was oxidized in such a way that PFOA was quantitatively and irreversibly trapped and would be separated from the aqueous phase as a precipitate. However, the structure of PFOA was not destroyed, and its biological toxicity remained. Li et al. [133] developed a Fenton-like technique (MW-Fenton-like) using Pb-$BiFeO_3$/rGO catalysis to degrade PFOA. After microwave irradiation, the Pb-$BiFeO_3$/rGO was improved to break down H_2O_2 and generate •OH, and the PFOA degradation rate reached 95% in 5 min.

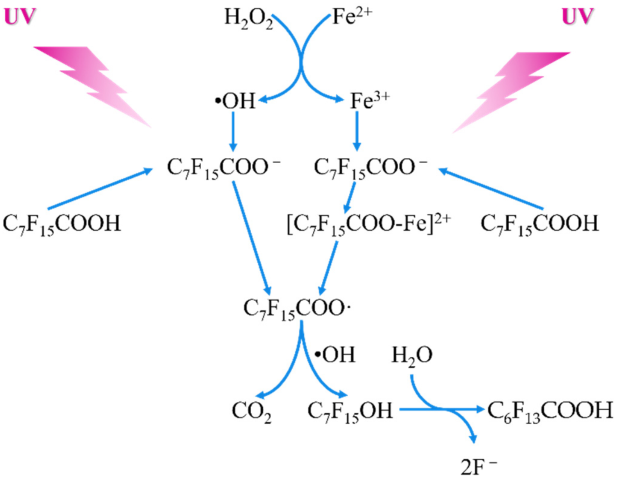

Figure 4. A two-stage mechanism for the degradation of PFOA in the UV–Fenton process. Reprinted with permission from reference Heqing Tang, Qingqing Xiang, Min Lei, Jingchun Yan, Lihua Zhu, Jing Zou, 2012, [132].

4.2.5. Other Chemical Methods

Several types of chemical methods described previously can not only treat PFCs alone, but can also be combined to form new technologies with more advantages. For example, Wang et al. [134] proposed a solar photo-electro-Fenton-like (SPEF) system constructed of MOFs/carbon nanofiber (MOFs/CNF) composite membranes which can effectively degrade PFOA. The degradation mechanism is shown in Figure 5. First, PFOA loses an electron on the anode to form $C_7F_{15}COO\bullet$. Then, decarboxylation occurs to form $C_7F_{15}\bullet$, which is converted to $C_7F_{15}OH$ by combination with •OH. This unstable intermediate then undergoes intramolecular rearrangement and hydrolysis, losing the shorter carbon chain and producing $C_6F_{13}COO\bullet$. Repeating the above process will completely mineralize PFOA. In the process of degradation, sunlight can catalyze the rapid formation of •OH from H_2O_2. The process of electrochemical degradation enhances the ability of electron transfer and also improves the mineralization efficiency of the SPEF system.

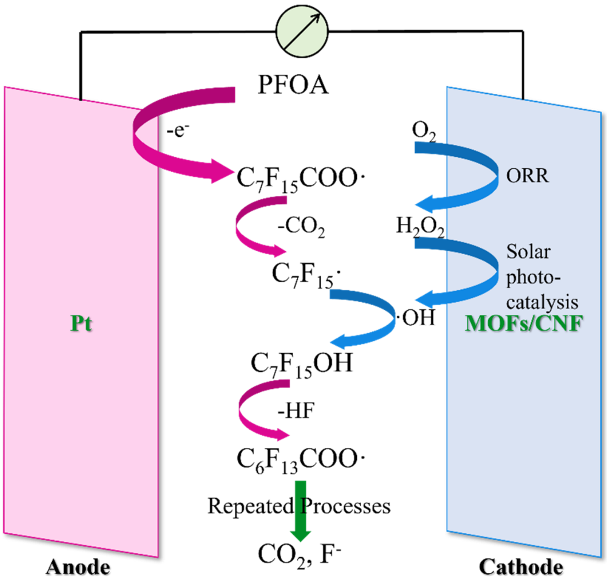

Figure 5. Mechanism of PFOA mineralization by the MOFs/CNF constructed SPEF system. Reprinted with permission from reference Yang Wang, Mingzhen Zhao, Chen Hou, Wenqiang Chen, Shuaishuai Li, RongKai Ren, Zhijian Li, 2021, [134].

In addition, chemical methods to degrade PFCs include coagulation, ozonation, activated persulfate, photocatalytic reduction, and so on. The effect of coagulation on the removal of PFOA in water increased as initial turbidity and coagulant dosage increased but decreased as the pH value, temperature, and NOM increased [142,143]. Alkaline ozonation shows potential in terms of energy demand, half-life period, and removal degree. Moreover, it is easy to implement on a large scale and can be implemented in existing ozonation reactors for water and wastewater treatment. Lin et al. [135] found that alkaline ozonation effectively degrades PFOA and PFOS by 85–100%. Thermally activated persulfate can produce $SO_4^{\bullet-}$ degraded PFOA, and its degradation rate and defluorination rate are reported to be 93.5% and 43.6%, respectively [144]. Wang and Zhang [145] studied the heterogeneous photocatalytic decomposition of PFOA by TiO_2 under 254 nm UV light. They found that the addition of oxalic acid could significantly accelerate the decomposition of PFOA. Oxalic acid not only provides acidity and acts as a hole scavenger in the process of PFOA decomposition, but also plays other significant roles. The reaction mechanism is shown in Equations (12)–(17). It can be seen that PFOA is effectively converted into various short-chain PFCAs and F^- by reductive dissociation, rather than oxidative decomposition, and finally mineralized them completely.

$$C_2O_4{}^{2-} + h^+ \rightarrow CO_2 + CO_2{}^{\bullet -} \tag{12}$$

$$C_7F_{15}COOH + e^- \rightarrow C_7F_{15}\bullet + HCOO^- \tag{13}$$

$$C_7F_{15}COOH + CO_2{}^{\bullet -} \rightarrow C_7F_{15}\bullet + HCOO^- + CO_2 \tag{14}$$

$$\bullet C_7F_{15} + \bullet OH \rightarrow C_7F_{15}OH \tag{15}$$

$$C_7F_{15}OH \rightarrow C_6F_{13}COF + H^+ + F^- \tag{16}$$

$$C_6F_{13}COF + H_2O \rightarrow C_6F_{13}COOH + H^+ + F^- \tag{17}$$

4.3. Biological Methods

Microbial treatment is the use of bacteria, fungi, and other microorganisms in the dissolution, enzymatic hydrolysis, phagocytosis, and other physiological processes to deal with degradation targets. Huang and Jaffé [146] used the autotrophic *Acidimicrobium* sp. Strain A6, which is responsible for the Feammox pathway (Equation (18)), to treat PFOA/PFOS and achieve up to 60% removal after 100 days. Dissolved organic carbon (DOC) was tracked during incubation, and a slight decrease in DOC was observed over time. This suggests that as PFOA/PFOS defluorination occurs, some products are metabolized by other heterotrophic bacteria in the medium. In conclusion, the A6 strain was able to use ammonium or hydrogen as electron donors to defluorinate PFOA/PFOS while reducing iron, as shown in Figure 6.

$$6(Fe_2O_3 \cdot 0.5H_2O) + 20H^+ + 2NH_4{}^+ \rightarrow 12Fe^{2+} + 17H_2O + 2NO_2{}^- \tag{18}$$

Harris et al. [147] isolated *Delftia acidovorans* from PFAS-contaminated soil. This bacterium was able to grow in a medium with only PFOA as a carbon source, and an increase in fluoride concentration was observed. After identification and characterization, it was found that *Delftia acidovorans* dehalogenase has potential for enzymatic defluoridation. Dehalogenases may also be designed so they can be produced in *E. coli* for rapid purification, which might provide new possibilities for addressing the pollution of PFCs on a global level. However, the questions of whether the enzyme can function extracellularly, whether enzyme activity is affected during purification, and whether the enzyme acts alone or what other factors are required to observe enzyme activity need further investigation. Albert et al. [148] developed an iron nanofunctionalized diatom cell bioreactor Fe-Dt that degraded about 93.7% of PFOA and 89% of PFOS within 24 h. Diatoms produce endogenous reactive oxygen species (ROS), and the loaded iron nanoparticles undergo a non-homogeneous Fenton reaction. The synergistic effect of both promotes the complete breakdown of PFOA and PFOS. When Fe-Dt is kept stable and used repeatedly over a long period of time, there is no cytotoxicity observed. This diatom-assisted bioremediation method suggests that in the future it will be possible to treat PFCs contamination in a sustainable and environmentally friendly way.

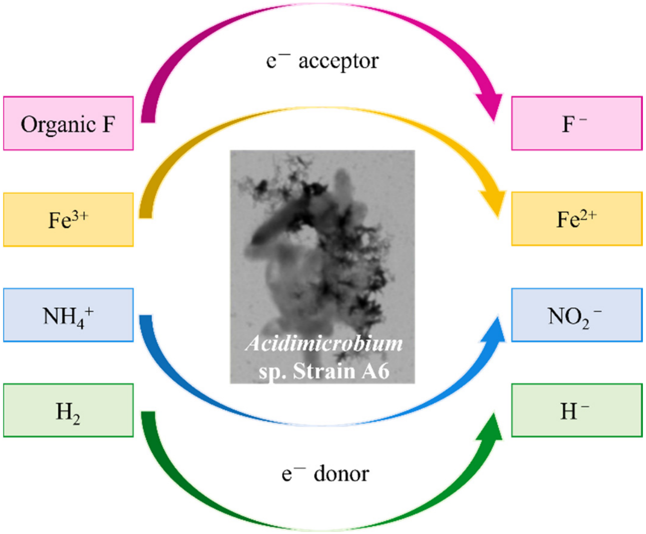

Figure 6. Defluorination of PFOA and PFOS by *Acidimicrobium* sp. strain A6.

Although PFCs are biologically inert, the above reports show their potential to be biodegraded. At present, studies on the biodegradation of PFCs are still very limited. Degradation using microbial strains is limited to reports on strains and degradation rates, without elucidating the degradation mechanism. In addition, the degradation rate of PFOA or PFOS by simple biological treatment is low, and it is difficult to highlight the advantages of biodegradation compared with other methods. Therefore, the main issues to be addressed in future research will be how to properly understand the processes behind the biodegradability of PFCs and how to increase their biodegradation rates.

5. Conclusions and Prospect

PFCs are a class of organic compounds with specific structures that have been widely detected worldwide. They have environmentally persistence, bioaccumulation, and other potential hazards. We provided an overview of the hazards, detection technologies and treatment methods of PFCs. We also analyzed the treatment effects, degradation mechanisms, problems, and future directions of different technological approaches. This can provide support and suggestions for PFC pollution-control technology.

With the development of instrument analysis technology, the detection technology of PFCs has diversified. Different detection techniques are applicable to different types of the sample matrix, and the technical requirements for sample pretreatment are also different. In the future, we should continue to optimize the pre-processing methods so as to reduce the requirements for the resolution of testing instruments. The derivatization method should also be improved so that gas chromatography or high-performance liquid chromatography may be used more often to analyze PFCs in all types of matrices.

Research on the treatment technologies of PFCs is gradually deepening, but they still need to be further improved and perfected. Among them, physical adsorption can only transfer PFCs, not change their chemical properties. Therefore, the future of this technology will focus on the re-treatment of pollutants and the regeneration of adsorbents. The biodegradation of PFCs is not yet mature. We should study their degradation mechanisms, determine the degradation genes, and study gene modification in the future. Chemical removal of PFCs is effective, but the defluorination rate and mineralization rate are low. Future research will focus on improving the defluorination rate through improved technology.

In addition, the removal of PFCs is still in the experimental stage. There is generally only a single solute in the solution, but the composition of pollutants in actual water containing PFCs is complex. Therefore, the removal efficiency of actual wastewater containing PFCs should be further investigated in the future. At the same time, the optimal combined process with various methods should also be discussed. This would remove PFCs from water more effectively, with lower energy consumption and lower cost.

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