

Advanced Oxidation Processes Used in The Treatment of Perfluoroalkylated Substances in Water

Procesos de oxidación avanzada utilizados en el tratamiento de sustancias perfluoroalquiladas en agua

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Abstract

Perfluoroalkylated and polyfluoroalkylated substances (PFAS) are a large family of synthetic chemicals widely used to manufacture various commercial products. However, they are hazardous to health and the environment. These substances are very persistent and their degradation requires advanced degradation techniques. One of the most widely used technologies for this purpose is advanced oxidation processes (AOP). This study aims to evaluate the use of AOP in PFAS degradation based on their efficiency, parameters influencing each process, advantages, disadvantages, and associated research challenges. According to the results, plasma, electrochemical oxidation, sonochemical oxidation, and heterogeneous photocatalysis stand out among the AOP applied for PFAS degradation. In addition, perfluorooctanesulfonic acid (PFOS) and perfluorooctanoic acid (PFOA) were found to be the main toxic compounds studied. In this regard, future research should focus on a wider variety of PFAS. Finally, the drawbacks associated with the application of these processes to eliminate the substances of interest were found to be an urgent need to be overcome.

Keywords: perfluorinated substances; advanced oxidation processes; efficiency; water treatment; perfluorooctanoic acid; perfluorooctanesulfonic acid

Resumen

Las sustancias perfluoroalquiladas y polifluoroalquiladas (PFAS, por sus siglas en inglés) son una gran familia de sustancias químicas sintéticas ampliamente utilizadas para fabricar diversos productos comerciales. Sin embargo, son peligrosas para la salud y el medio ambiente. Estas sustancias son muy persistentes y su degradación requiere la aplicación de técnicas de degradación avanzadas. Una de las tecnologías más utilizadas con este fin son los procesos de oxidación avanzada (POA). Este trabajo pretende evaluar el uso de POA en la degradación de PFAS basado en su eficiencia, parámetros que influyen en cada proceso, ventajas, desventajas y retos de investigación asociados. De acuerdo con los resultados, el plasma, la oxidación electroquímica y sonoquímica, así como la fotocatalisis heterogénea, destacan entre los POA implementados para la degradación de PFAS. Además, el ácido

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perfluorooctanesulfónico (PFOS) y el ácido perfluorooctanoico (PFOA) resultaron ser los principales compuestos tóxicos estudiados. En este sentido, se debería estudiar en futuras investigaciones una variedad más amplia de PFAS. Por último, se hace necesario investigar cómo superar los inconvenientes asociados al uso de estos procesos en la eliminación de las sustancias de interés.

Palabras clave: sustancias perfluoradas; procesos de oxidación avanzada; eficiencia; tratamiento de aguas; ácido perfluorooctanoico, ácido perfluorooctanesulfónico.

1. Introduction

Perfluoroalkylated and polyfluoroalkylated substances (PFAS) are chemicals that can be used in numerous commercial applications due to the high chemical and thermal stability of their covalent carbon-fluorine bonds [1], [2]. They can be found in a wide range of everyday products, including cleaning products and non-stick coatings for pans [1], [2]. Additionally, these substances are frequently detected in the environment at trace and ultra-trace concentrations, especially in water, adversely affecting aquatic species due to their carcinogenic, mutagenic and teratogenic potential [1], [2]. The persistence of PFAS in the environment raises significant concerns. Due to their resistance to degradation, they can accumulate in soil, water and living organisms over time [3]. In this regard, they are hardly eliminated from the environment by natural processes.

Due to the persistence of these pollutants, conventional water treatment methods, including traditional oxidation, filtration or coagulation and flocculation processes, are not efficient in their removal [4]. In this regard, the implementation of alternative treatments, such as advanced oxidation processes (AOP) is required. These processes have a high efficiency in the degradation and mineralization of persistent organic pollutants (POP) such as PFAS [5]. AOP include a variety of techniques that promote the formation of free radicals, such as hydroxyl radicals ($\bullet\text{OH}$), among other radical species. It can lead to the complete degradation of recalcitrant pollutants [6], since organic compounds with complex molecular structures can be destroyed and innocuous substances can be formed, including water molecule (H_2O), carbon dioxide (CO_2) and inorganic ions. Plasma, heterogeneous photocatalysis, electrochemical oxidation, and sonochemical degradation are among the main AOP that can be used individually or in combination with other processes for the treatment of PFAS [7], [8].

There are many studies describing in detail the application of AOP for the treatment of PFAS in water, including their evolution, factors influencing the oxidation system, advantages, or future perspectives [3], [9], [10], [11], [12], [13], [14], [15], [16]. However, according to the authors' knowledge, a compilation on the

application of AOP for PFAS degradation in the water matrix has not been carried out.

Aiming to fill this gap, this study is proposed to examine the application of AOP in the treatment of PFAS in water. The first part of the work refers to the methodological aspects, where the procedure for the selection, analysis and synthesis of the reported studies are described. Subsequently, the results of the search are analyzed and discussed focusing on the process efficiency and operating parameters of each one of the advanced oxidation technologies used in the treatment of these recalcitrant organic compounds in water. Additionally, future challenges are described, including the high costs of implementation associated with the use of these technologies, as well as the generation of degradation by-products more toxic than the parent compounds and the limitations of large-scale applications, among other challenges ascribed.

2. Materials and methods

2.1. Search methodology

Interested in the available information about the application of AOP in PFAS degradation, a high amount of the latest studies that analyzed this topic was considered. For this purpose, Google Scholar was used, as well as the search keywords, time interval (2020-2023), language (English) and document type (mainly, compiling articles). A general algorithm was used that assembled both Boolean operators and keywords. The search algorithm utilized was: (Perfluoroalkyl OR Polyfluoroalkyl OR PFAS OR "Perfluorinated Compounds") AND ("Advanced Oxidation Processes" OR AOP) AND (Water or Wastewater) AND Treatment AND Pilot.

2.2. Inclusion and exclusion criteria

Of the total number of documents retrieved, those whose abstracts met the following selection criteria, were included mainly focusing on compiling documents:

- ✓ Inclusion of AOP
- ✓ Inclusion of PFAS treatment
- ✓ Inclusion of water matrix

If this information was not explicit in the abstract or more general concepts were mentioned (e.g. remediation technologies, emerging contaminants, persistent organic pollutants), a reading of the full text was performed, identifying again the aspects mentioned above.

2.3. Analysis and synthesis

For each selected document, the following information was withdrawn: title, abstract, authors, year and the AOP studied. This information allowed to identify the utmost AOP for the issue of study. Then, a further analysis to extract more specific information about the AOP used was performed. The following information was compiled:

- ✓ Description of the AOP used, influential parameters and removal efficiencies
- ✓ Advantages and disadvantages of the AOP assessed
- ✓ Research challenges related to the issue of interest

The aspects listed above defined the structural order and organization of this study.

Finally, an analysis of the retrieved aspects was conducted, and conclusions and recommendations from the findings were withdrawn.

3. Results and discussion

3.1. Preliminary results

310 documents were retrieved by using the search algorithm described above. From these documents, only 32 were selected after the application of the inclusion and exclusion criteria mentioned previously. It was found that the application of AOP for PFAS treatment in water has been widely studied in the last 3 years, which highlights the importance of the study carried out.

3.2. Advanced oxidation processes used in the treatment of perfluoroalkylated substances

The most discussed AOP that were found to be addressed were electrochemical oxidation, sonochemical degradation and plasma. Other AOP were also discussed, such as processes including persulfate oxidation ($S_2O_8^{2-}$), and ultraviolet radiation coupled to ozone (UV/ O_3) and to hydrogen peroxide (UV/ H_2O_2), represented as UV/oxidizing agent. In addition, heterogeneous photocatalysis and Fenton and photo-Fenton processes were also addressed. In Figure 1, the percentage distribution referring the use of these AOP is illustrated.

Some of the issues discussed in the documents included the efficiency of AOP in PFAS degradation, operational

factors influencing PFAS degradation efficiency and the advantages and disadvantages of AOP application in PFAS treatment, and these are discussed below. Additionally, some research challenges related to the topic proposed by the authors, are presented.

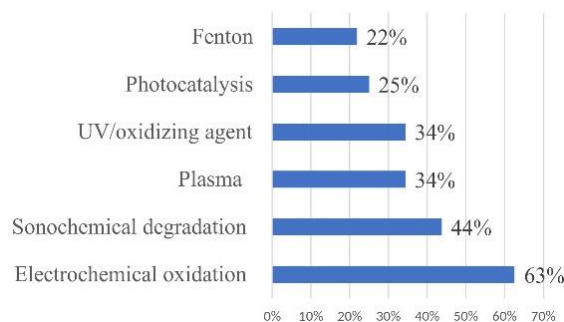


Figure 1. Distribution of advanced oxidation processes used for perfluoroalkylated substances treatment in water.

3.3. Process efficiency and operational parameters

AOP can be classified into photochemical and non-photochemical processes. Figure 2 lists each of the AOP discussed in the documents, with their corresponding classification and the number of papers addressing each technology (in brackets).

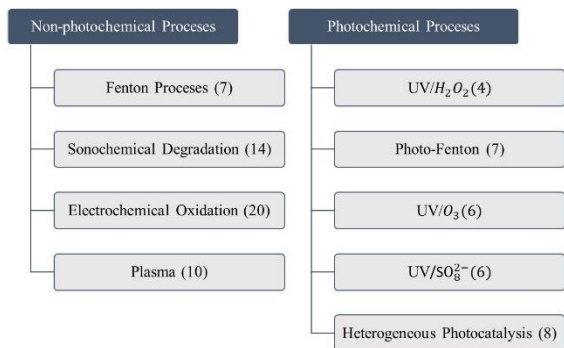


Figure 2. Classification of AOP discussed and number of documents that addressed each process.

3.3.1. UV/oxidizing agent

Photochemical processes involve the combination of a light source with an oxidant. This results in photolysis to generate reactive radical species that attack the pollutants and cause their degradation. When the radical generated in the process is an oxygen species, the process is called a reactive oxygen species (ROS) process. Oxidants used in ROS processes include hydrogen peroxide (H_2O_2), persulfate ($S_2O_8^{2-}$), ozone (O_3) and peroxymonosulfate (HSO_5^-), among others [17].

In some studies, PFAS degradation through photochemical processes has been ineffective. However, in others the application of these processes has been promising. X. Wang [6] reported that the combined O_3/H_2O_2 and O_3/UV systems were ineffective in perfluorooctanesulfonic acid (PFOS) treatment in processes applied at laboratory scale under different operating conditions.

On the other hand, the $UV/S_2O_8^{2-}$ system has been extensively studied in recent years due to the significantly high efficiency of $S_2O_8^{2-}$ in the removal of PFAS. In one of the studies analyzed by Wanninayake [11], $S_2O_8^{2-}$ activated by radiation emitted from a Xenon lamp resulted in a complete degradation of perfluorooctanoic acid (PFOA) and a minor formation of short-chain perfluoroalkyl carboxylates (PFCA). The operating conditions were 40 μM of PFOA, 12 g/L of $S_2O_8^{2-}$, and 4 h of irradiation at wavelengths ranging from 220 to 460 nm (at laboratory scale). Several studies concluded that $S_2O_8^{2-}$ radical-based processes are promising options for PFAS removal from wastewater effluents and drinking water sources under different conditions [10], [11], [18], [19].

In the researchers analyzed by Wang [10], the degradation rate through photochemical processes ranged from 5% to ~100%. The defluorination rate ranged from 10% to 82%, depending on the target pollutants and reaction conditions. Due to the chemical stability of PFAS, reaction times were mostly longer than 2 h, reaching 12 h of treatment. Different studies showed that the defluorination rate was much lower than the degradation rate, indicating that PFOA and PFOS were not completely transformed into fluoride (F^-) ions. According to Hajalifard et al. [20], sulfate radicals decomposed PFCA, while PFAS resisted their attack.

The influence of certain factors affecting the efficiency of PFAS degradation and defluorination through photochemical processes has been demonstrated. These include oxidant concentration, type and wavelength of UV lamps, C_0 of target pollutants, reaction time and solution pH [10], [11], [18], [19], [21]. With regard to the concentration of the oxidizing agent, several studies reported that the degradation rate of PFAS increased as the concentration of the oxidizing agent increased up to the optimum point. An excess of the oxidizing agent can hinder the decomposition of PFAS [10], [18]. In terms of wavelength, it was found that the performance of PFAS degradation with irradiation at 185 nm was more frequently studied and more efficient compared to that of UV irradiation at 254 nm [6].

It is noteworthy that the initial concentration of PFAS has a direct impact on degradation and defluorination rates. In the study carried out by Wang et al. [10], a lower concentration of PFOA was evidenced to be more easily degraded. Regarding the pH value of the solution, it was reported as a key factor affecting the distribution of oxidizing chemical species and free radical transformation. The analyzed studies reported different optimal pH values, all of which were in the acidic range, since more free radicals are produced under acidic conditions [18]. In the studies considered by Hajalifard [20], the effectiveness of the $UV/S_2O_8^{2-}$ process was reduced in the presence of solutes such as bicarbonate (HCO_3^-), chloride (Cl^-), and organic matter, included in real water matrices.

3.3.2. Photocatalysis

A photocatalytic system involves the use of a radiation source, catalyst and, sometimes, oxidizing agents [22]. When the catalyst is in the same phase as the reaction system, the photocatalysis is homogeneous; otherwise, it is heterogeneous. The homogeneous photocatalytic degradation of PFAS consists of the photo-Fenton process, where the reaction generates $\bullet OH$ through the Fenton reaction between ferrous ion (Fe^{2+}) and H_2O_2 [23]. It should be mentioned that the photo-Fenton process is discussed in the section corresponding to the Fenton process. For the heterogeneous photocatalytic processes, a semiconductor material is activated by electromagnetic radiation, leading to the triggering of a series of oxidation-reduction reactions [24].

According to Olatunde and coworkers [18], photocatalysis is the most studied process for PFAS degradation. Heterogeneous photocatalysis provides one of the best PFAS remediation techniques [18], [25]. In photocatalysis, the photooxidation and photoreduction pathways act together to efficiently degrade PFAS into PFCA through the progressive removal of CF_2 groups [26].

Ahmed et al. [27] and Leonello et al. [19], [26] indicated that heterogeneous photocatalysis can efficiently degrade PFAS. Specifically, titanium dioxide (TiO_2)-based photocatalysts have been widely explored for PFAS degradation. Up to 100% degradation of these compounds has been achieved using these photocatalysts. TiO_2 is widely used for environmental purposes in photocatalytic treatment due to its availability, chemical stability and low cost [19], [27], [28]. Some authors point out that the combination of UV/TiO_2 with metals and modification of the molecular structure of some compounds are effective approaches to

improve the photodegradation efficiency of PFAS [18], [26], [27], [29], [30].

Leonello et al. [26] highlighted that the UV/TiO₂ system with photocatalyst modified by the introduction of heavy metals (e.g., Cu, Fe, Pb) or transition metals (e.g., Pt, Pd, Ag), has a high efficiency in the degradation of PFAS. The highest efficiencies are obtained with Pt, with 100% PFOA degradation. This result was reached in 300 min of treatment (C₀: 60 mg/L, 0.5 g/L TiO₂/Pt, 47.5 m²/g surface area, 365 nm) at laboratory [27], [29]. Additionally, the reaction rate of the UV/TiO₂/Pt system has been reported to be 12.5 times higher than that of the UV/TiO₂ [26]. The improvement of catalytic ability when the catalyst is modified with metals is attributed to the formation of PFOA-metal-TiO₂ complexes, thus increasing the degradation capacity of the pollutants of interest [26], [29].

Nanostructured gallium oxide (Ga₂O₃) and indium oxide (In₂O₃) with modifications in their morphology have more potent photocatalytic activity than TiO₂ [19]. They can also degrade 100% of PFOA in 40 min at laboratory scale (C₀: 30 mg/L, 0.5 g/L TiO₂, 36.1 m²/g surface area, 254 nm) and 17 min (C₀: 0.5 mg/L PFOA, 0.5 g/L TiO₂, 156.9 m²/g surface area, 254 nm), respectively [27]. Ag nanoparticles immobilized on molecularly imprinted polymer-modified TiO₂ nanotubes also proved to be a suitable option in the degradation of PFOA and short-chain perfluorinated compounds [19], [29].

The parameters that affect heterogeneous photocatalysis include the pH of the solution, the wavelength, bandgap and surface area of the catalyst, the initial concentration of PFAS, the presence of inorganic ions in the system and the dissolved oxygen in the water to be treated [18], [26], [27], [29].

According to Leonello et al. [26], most research on the photocatalytic degradation of PFAS was conducted at a pH value below 4 since the highest degradation efficiencies are obtained at this pH. Similarly, in the study conducted by Ahmed et al. [27], the degradation efficiency of PFOA was reported to be higher and faster when the solution pH was low.

Regarding the wavelength, studies compiled by Olatunde et al. [18] and Ahmed et al. [27] showed that, although PFAS degradation was achieved through visible light (400-800 nm), the photodegradation efficiency was higher when UV radiation (185-254 nm) was used. Likewise, in the research conducted by Leonello et al. [26], an efficient degradation of PFOA was evidenced when a wavelength of 254 nm was used. However, PFOA degradation decreased drastically with longer

wavelengths (315-400 nm). According to Ahmed and coworkers [27], a wavelength of less than 190 nm is required for efficient degradation of fluorinated substances. Nevertheless, for Jhon et al. [31], PFAS do not absorb light at a wavelength longer than 220 nm.

Regarding the catalyst dosage, Ahmed and coworkers [27] reported that with a higher amount of catalyst (particularly TiO₂) the degradation efficiency of PFOA increased. This is due, in part, to the presence of greater surface area available for adsorption and degradation of PFOA, although it should be noted that an excessive amount of the photocatalyst in suspension can also inhibit the photodegradation process, since the particles inhibit the penetration and distribution of photons within the solution [26].

On the other hand, it is important to note that the presence of certain substances can influence the efficiency of the photocatalysis process by either inhibiting or accelerating the degradation of pollutants. In this regard, some inorganic anions such as chlorides and sulfates negatively affect the degradation process of PFAS through photocatalysis [18], [29], [30]. Similarly, the presence of organic matter detracts from the efficiency of the photocatalytic degradation process of PFAS [18], [31].

3.3.3. Sonochemical oxidation

Sonochemical degradation consists of chemical bond breaking and free radical formation using high or low frequency ultrasound (US). The application of US is considered an effective technology for treating water contaminated with PFAS [12], [25], [32], especially with PFOS and PFOA [28]. However, sonochemical degradation is energy-intensive and therefore not cost-effective on its own [16]. Additionally, its large-scale implementation has become challenging due to the elevated costs [11]. Nevertheless, coupling of US with other energy sources (e.g., UV radiation or chemical oxidation) presents interesting and attractive approaches according to this study. Recently much effort has been devoted to improving the efficiency of sonochemical reactions by integrating US with other processes. Cao et al. [16] reported that the use of UV-assisted US for the treatment of PFAS can speed up the defluorination process and decrease operational energy consumption. Compared to the use of US alone, the UV-assisted US system increased the defluorination and PFOS degradation rates by 12.01% and 8.76%, respectively, after 6 h of treatment. It has been shown that photocatalysis with TiO₂ combined with US increases the degradation of several PFAS, and that visible light can be used in the process. In turn, Panchangam and coworkers

[33] proved that US-assisted photocatalysis at laboratory scale can remove up to 64% of PFOA (C_0 : 120 Mm, pH: 4.55-6.22) after 8 h of treatment.

Wanninayake et al. [11] analyzed the remediation of wastewater polluted with PFAS using US in the presence and absence of other AOP. It was concluded that hybrid processes involving US, such as US/O₃, sono-photocatalysis, and sono-Fenton and sono-photo-Fenton processes, have a superior performance in PFAS degradation than that of any of the techniques when operating individually. In addition, the research conducted by Foote et al. [34] and Verma et al. [35] concluded that US can successfully improve the performance of other PFAS oxidation techniques.

Verma et al. [29] reported on the application of full-scale sonolysis in the degradation of PFOS and PFOA in groundwater extracted from a landfill. In that study, US showed to be effective in the mineralization of PFOA and PFOS in aqueous matrices (C_0 : 30 µg/L (PFOA), 60 µg/L (PFOS), pH: 6.9-7.9, TOC: 20 mg/L, T: 10-15 °C and frequency: 354 kHz) [36]. The elimination of PFOA and PFOS reached 44% and 39%, respectively.

A comparative analysis of treatment technologies to remove PFAS from water reported that sonochemical degradation works better for PFOA than for PFOS when the pollutant is at low concentrations. However, at high concentrations, PFOS has a higher degradation efficiency than PFOA. The difference between these results is likely due to the significantly greater number of cavitation bubbles involved in PFOS degradation. Additionally, a tendency was observed on the part of sulfonate groups to thermally degrade at higher temperatures than carboxylic groups [34].

In some studies, the degradation rate of PFAS increased significantly with increasing perfluoroalkyl chain length groups [34]. This phenomenon may be attributed to the increased hydrophobicity of the compound and thus the concentration at the cavity-water interface. It is then less likely that short-chain PFAS are efficiently degraded by sonochemical oxidation due to their less hydrophobic character [34], [37].

The performance of processes involving US was found to be highly dependent on the operating parameters, including the power density, frequency, temperature, room conditions, additives, inorganic compounds presence (e.g., bicarbonate and sulfate), pollutant concentration and the US reactor design [11], [15], [16], [19], [34], [38], [39]. Additionally, Yadav and coworkers [19] suggested that the presence of volatile organic

compounds decreased the degradation efficiency of PFOS and PFOA due the temperature reduction.

The degradation rate of PFAS generally increases as the power density is increased [16], [25], [39], [40]. In the case of ultrasonic frequency, Cao et al. [16] reported that 358 kHz was the most effective frequency for the degradation of perfluorohexanoic acid (PFHxA), perfluorohexanesulfonic acid (PFHxS), PFOA and PFOS. In turn, the sonochemical degradation rates of perfluorobutanoic acid (PFBA) and perfluorobutane sulfonic acid (PFBS) reached the maximum at 610 kHz. When the frequency exceeded its optimum value, the degradation rates of PFHxA, PFHxS, PFOA and PFOS decreased with increasing frequency.

Regarding temperature, PFOA removal was reported to decrease with increasing temperature [16], [31], [34]. Concerning room conditions, it has been observed in different studies that cavitation efficiency is higher in the presence of argon (Ar) than air, due to the higher polytropic index of Ar [16].

Additives that can generate a large number of free radicals during sonochemical reactions, such as S₂O₈²⁻, periodate (IO₆⁵⁻) and permanganate (MnO₄⁻), can reduce the energy consumption and accelerate the degradation rate of PFAS [16], [31], [34], [40]. Regarding the initial PFAs concentration, Cao and coworkers [16] reported that the degradation and defluorination rates of PFOS and PFOA increased as the initial concentration is increased.

3.3.4. Fenton and photo-Fenton processes

The Fenton process involves the reaction between H₂O₂ and Fe to produce •OH. This process can be carried out in the presence of UV radiation, giving rise to what is called the photo-Fenton process. It should be noted that the photo-Fenton process is characterized by increasing the oxidation power of the classical Fenton system and is usually used to improve the performance of the former, as the number of •OH also increases [41].

No consensus exists on the efficiency of Fenton in the degradation of PFAS in water. Some studies have reported that this process is not effective for PFAS degradation. Verma and coworkers [29] informed that Fenton's reagent was not suitable for the degradation of 20 mg/L PFOS at laboratory scale.

In turn, Meegoda et al. [38] reported a limited effectiveness of Fenton's reagent in PFAS mineralization due to the strength of C-F bonds and the high electronegativity of fluorine. Pilli et al. [39] did not find

significant PFOS removal by Fenton-based processes at laboratory scale (pH: 3.5, 500 mg FeSO₄ and 5 ml H₂O₂).

However, Ahmed et al. [27] reported good efficiencies in PFAS degradation, such as 89% removal of PFOA in 150 min using 1 M H₂O₂ and 0.5 mM Fe. Ahmed and coworkers [27] improved the performance of the Fenton process when combining it with UV radiation. In the study conducted by Tang et al. [42] PFOA degradation efficiency of 87.8% and defluorination of 12.7% were evidenced in a solution containing 20 mg/L PFOA and 26.8 mg/L Fe³⁺. Several articles mentioned that the presence of S₂O₈²⁻ accelerates the photolytic degradation rate of PFOS and PFOA in the photo-Fenton process [27], [29]. According to Hajalifard et al. [20] although the Fenton process itself cannot degrade PFAS, it has been successfully used in removing PFOA.

Studies analyzed by Verma et al. [29] also highlighted the advantages associated with Fenton reagent irradiation, reporting 60% PFOA and 80% PFOS removal (C₀: 20 mg/L) when these compounds were exposed to UV irradiation (254 nm, 10 W) for 24 h. [18] also reported the degradation of PFOA through the photo-Fenton process, reaching more than 90% and 53.2% elimination and defluorination, respectively, after 5 h, using 20.0 μM as the initial concentration of the pollutant.

The parameters that influence the Fenton/photo-Fenton processes include the Fe²⁺ and H₂O₂ concentration, wavelength and the solution pH [27], [28], [29], [39]. Some authors reported that the degradation rate of PFAS increases as the concentrations of H₂O₂ and Fe²⁺ increase, although an excess of these reactants can reduce the degradation of PFAS since side reactions are favored [25], [27], [29]. Regarding the solution pH, several authors agreed that Fenton's reagent works more efficiently at a pH value ranging from 3 to 4, since Fe²⁺ have a higher solubility in acidic solutions [27], [29], [39].

3.3.5. Electrochemical oxidation

The treatment consists of the direct transfer of electrons from an anode to molecules inside electrochemical cells equipped with anodes, cathodes and electrolytes [20], [43], [44]. In this process, electrical energy is used as an energy source to generate electrons [45]. Currently, electrochemical treatment is being used in PFAS treatment, evidencing an effective transition from laboratory-scale studies to those conducted at pilot and real scales [38]. However, this process has been found to be ineffective in the degradation of short-chain PFAS and its application involves the formation of undesirable

toxic products (e.g. hydrogen fluoride, chlorine gas, bromate, perchlorate and organic halides) [25], [27], [38].

Different materials have been used to obtain electrodes utilized in the electrochemical degradation of PFAS. The most recent studies reported in the literature have focused on the application of boron-doped diamond (BDD) [11], [13], [20], [21], [34], [37], [39], [46]. Nonetheless, other materials including Ti/SnO₂, Ce/PbO₂, TSO and Ti/RuO₂ have also been used to a lesser extent [47]. For example, Román et al. [46] mentioned that systems with TSO reported faster and more complete degradation in experiments containing longer chain PFAS and showed that the degradation of this compound can be achieved using cheaper alternatives to BDD electrodes. Nevertheless, several studies agreed that BDD outperforms other electrode material options due to its strong oxidizability, chemical, mechanical and thermal stability, commercial availability, high reactivity, low adsorption capacity and flexibility [11], [31], [34], [38], [39].

BDD electrodes can effectively degrade PFOA, PFOS, PFBA, PFHxA, perfluorodecanoic acid (PFDA), PFBS and PFHxS under optimized operating conditions [20], [31], [39]. A case study conducted in the field was reported by Wanninayake et al. [11] and Sharma et al. [13], in which BDD was used in the electro-oxidation of PFAS present in discharges from an industrial wastewater treatment plant. In this study, 99.7% removal of various PFAS was achieved after 10 h of operation (1402 μg/L PFAS, pH 8.4, 256 kWh/m³).

In one of the selected studies by Lu et al. [48], electrochemical oxidation combined with nanofiltration was used in a treatment train process aiming to remove and degrade PFHxA from industrial water. The target compound was evaluated at concentrations between 60 and 200 mg/L, obtaining degradation levels of the compound of interest ranging from 91% to 98% after 90 min. In another study reported by Lenka et al. [49], nanofiltration with electrochemical oxidation was used, reaching a removal of approximately 99% of PFAS in less than 3 h. These results were obtained at pollutant concentrations lower than 1000 mg/L (pH 7.7 and conductivity of 1.05 mS/cm) [8].

Operating parameters influencing electrochemical oxidation include electrode material, applied current density, reactor configuration, electrolyte type, PFAS concentration and chain length, solution pH and temperature [20], [25], [28], [37], [39], [44], [47], [48]. Regarding the electrode material, titanium (Ti), tin (Sn) and BDD are regarded as the most used electrodes due to

their strong oxidation ability [34], [38]. A higher current density could increase the direct oxidation of PFAS, specifically PFOA and PFOS, through direct electron transfer between the molecule and the anode [25]. Nonetheless, a complete oxidation of PFOA was reported to occur at current densities < 25 mA/cm [13].

Finally, it is noteworthy that the electrolyte influences the conductivity, so electrolytes with lower conductivity may decrease the electroactive surface area for oxidation and vice versa [50]. With respect to pH, the electrochemical degradation process is reported to operate best at acidic pH ranges [47], [49], [50].

3.3.6. Plasma

Plasma is the fourth state of aggregation of matter and consists of ions, atoms in higher energy state, atomic fragments and free electrons, among others. The use of plasma in various applications has increased significantly in recent years. One of these applications is water treatment [27].

Two types of plasmas are distinguished, thermal and non-thermal, the latter being those usually used for the removal of contaminants from water. According to Palma and coworkers [51], there is a large amount of recently published literature on PFAS treatment using different types of non-thermal plasma reactors. Both artificial matrices, such as deionized and real water matrices (e.g., contaminated groundwater or landfill leachate) containing PFAS have been treated with non-thermal plasma [51], [52]. According to Marquínez-Marquínez et al. [44], this is a novel process for the remediation of PFAS-contaminated water that could greatly improve the degradation efficiency of PFAS.

Non-thermal plasma is produced by different methods including (DC), alternating current (AC), microwave energy supply, radio frequency sources and pulsed discharge method [53]. Plasma water treatment is a technology that, using electricity, converts water into a mixture of highly reactive species. When used for water treatment, the plasma is generally formed by an electrical discharge between two electrodes, one high voltage and one grounded electrode located within or in contact with the pollutant water. Several types of electrical discharge plasma reactors with different electrode arrangements have been used to treat a wide range of organic and inorganic pollutants, including PFAS [27], [54].

Plasma is considered a new technology for the treatment of PFAS-polluted water and is still in the experimental stage [11], [30]. However, promising PFAS removal efficiencies have been reported at laboratory scale

through this technology. For example, in a study analyzed by Phong Vo et al. [55], plasma eliminated 90% of PFOA at a concentration of 8.3 mg/L in 60 min of treatment (pH: 4.6 and electrical conductivity: 300 μ S/cm using 0.1 M of NaCl). For plasma generation, a high-voltage power supply was used. The plasma was generated from Ar and the gas flow was 4 L/min. Pulses of 40 Hz frequency were generated by charging a 2 nF capacitor and discharging it through a rotating spark gap. The plasma reactor consisted of a glass vessel 17.3 cm in diameter and 19 cm in height (the total reactor volume was 3.8 L), with a sharp nickel-chromium rod (diameter 2.2 mm) as the high-voltage electrode, and an aluminum ring (outer and inner diameter 9.8 and 6 cm, respectively) as the ground electrode. The high voltage electrode was placed in the headspace region, and the ground electrode was placed as a ring around the circumference of the diffuser immersed in the liquid. The distance between the high voltage electrode and the ground electrode was 2.7 cm (1.5 cm in liquid and 1.2 cm in gas) [54].

This process has been applied at pilot scale in order to evaluate its feasibility. It was reported in a study compiled by Phong Vo et al. [55] that a large variety of PFAS can be degraded with plasma at a concentration of 1.4 mg/L between 1 and 50 min (electrical conductivities of the solution ranged from 22 to 26,300 μ S/cm and pH from 5.3 to 8.0).

Parameters influencing plasma degradation include feed gas, plasma reactor configuration, electrical conductivity, energy level of the electrical discharge, solution pH, the molecular structure of the pollutant, nitrate concentration, and the presence of dissolved organic matter, among others [11], [38], [51].

Wanninayake et al. [11] found that PFOA in drinking water, using Ar, significantly improved the PFOA decomposition efficiency compared to air. In turn, in Saleem and coworkers [14] analyzed several plasma reactors for PFOA treatment ("7-wire" DC plasma reactor, "hollow electrode" AC plasma reactor; self-pulsing discharge DC plasma reactor). In this study, large differences in PFOA degradation yields were observed clearly. The 7-wire DC plasma reactor was the least efficient, whereas the best efficiencies were obtained using the autopsulating discharge DC plasma reactor [14].

Regarding the energy level, the degradation efficiency of some PFAS was shown to increase with the increase in the discharge voltage regardless of the treatment time. This can be attributed to a stronger discharge voltage that induces a higher electric field, promoting the formation of active species and, thereby, the degradation of the

target pollutants [29]. Regarding the solution pH, in a study on the degradation of perfluoroalkyl phosphinic acids (PFPIA) through plasma oxidation, alkaline conditions were reported to promote the degradation of these substances [51], [52], [56]. Other parameters that influence plasma degradation are the initial concentration of the pollutant, type of reactor and gas used, and the characteristics of the solution among others [28], [31], [52].

3.4. Advantages and disadvantages of the use of AOP for PFAS degradation

Table 1 shows some advantages and disadvantages regarding the use of different AOP in the degradation of PFAS contained in water both synthetic and real matrices.

3.5. Research challenges

3.5.1. Application of AOP in the degradation of other PFAS

There is extensive and notable research on the treatment of PFAS with AOP. Some studies conclude that these works have mainly focused on the treatment of PFOS and PFOA. In this regard, future research covering a broader range of PFAS is proposed [6], [38], [55], [57], [58]. According to the Organization for Economic Cooperation and Development, 4,700 PFAS are known to exist to date [59]. This vast number of PFAS includes compounds with different physical and chemical properties, including gaseous compounds (e.g., perfluorobutane), liquids (e.g., fluorotelomeric alcohols) and high molecular weight solid polymeric materials (e.g., polytetrafluoroethylene), among others [60]. The uncertainties and lack of knowledge about the different PFAS in terms of their properties, toxicity, environmental fate and risks to human health are a major and growing drawback, as the number of PFAS continues to increase in quantity and complexity. In-depth knowledge of different aspects related to PFAS would lead to the development of safe, more efficient and cost-effective remediation and degradation strategies [11], [20], [38].

In addition, the operating conditions of the different treatment systems aimed at the removal of short-chain PFAS, such as perfluoropentanoic acid (PFPeA), PFHxA and perfluoroheptanoic acid (PFHpA), etc., need to be improved [49], [55]. Current legislation and guidelines with respect to PFAS should not only consider PFOA and PFOS, since short-chain PFAS are also known to have negative effects on the environment and public health [38], [55]. In this regard, and with respect to human

health, exposure to short-chain PFAS has been reported to be associated with a decreased in young men semen quality, low birth weight and infertility [61]. Moreover, these substances, like long-chain PFAS, are extremely persistent and will remain in the environment for decades or centuries. They also have a low adsorption potential and their transport capacity is relatively high compared to their long-chain counterparts. In fact, there is already evidence that short-chain PFAS is present in remote areas [62].

3.5.2. Unintended consequences of PFAS treatment

There is widespread concern about the undesirable consequences of PFAS treatment in water. One of these undesired outcomes is the formation of toxic substances. Therefore, an assessment of the formation of hazardous transformation by-products should be performed and, to this end, thorough study of the degradation mechanisms of PFAS through toxicological investigations on intermediate products is recommended. Additionally, estimation of the ecological risk posed by degradation products must also be considered in further research in order to avoid secondary pollution [6], [16], [27], [39], [48]. Likewise, Hajalifard et al. [20] suggest that further studies are required to identify the decomposition by-products of PFAS and their toxicity. Verma et al. [29] suggest investigating the secondary contamination generated using nanotechnology in the treatment of these pollutants. There is a marked tendency to use nanoparticles, mainly in photocatalytic processes, which can cause secondary contamination, generating risks to public health.

Another undesirable aspect of water treatment through the implementation of AOP is the corrosion that can be generated in the infrastructure of drinking water treatment systems. In this regard, Wang and coworkers [10] inform on the need to study the drawbacks associated with corrosion to avoid damaging the distribution systems, achieving efficient and cost-effective removal of a wide range of PFAS and thereby protecting public health.

According to the mentioned authors, any treatment that changes the pH, bicarbonate/carbonate ions, sulfates, chlorides, and natural organic matter in water can contribute to the corrosion of the distribution system, and lead to changes in water stability and solubility, reducing water quality and putting human health at risk.

3.5.3. Implementation of large-scale AOP

Another important aspect discussed in the studies analyzed is the nascent application of AOP in real water

to treat PFAS. Most research has been conducted at laboratory scale [11], [16], [18], [27], [34], [39], [50]. Although there are some full-scale trials, they have not been remarkable [27].

In the research conducted by Cao et al. [16], many studies aimed at US removal of PFAS used small (up to a few liters) reactors, with limited scalability, finding that bringing this process to full scale has several associated limitations, particularly high costs. Additionally, the studies mentioned highlight the complexity of automating the process and the corrosion phenomena of large-scale US reactors after continuous use, resulting in reduced energy transfer, and causing problems such as poor heat dissipation. Therefore, further research aimed at solving the aging problem of large-scale US reactors is required. On the other hand, according to Meegoda et al. [28], research on PFAS degradation by photocatalysis is still on a laboratory scale. Hajalifard and coworkers [20] also found that many AOP explored for PFAS removal have been conducted on a laboratory scale rather than in wastewater treatment plants. Thus, the authors proposed that commercial-scale wastewater treatment systems should be the focus on future research.

For some authors, the effects of operating parameters in the laboratory have been extensively studied [16], [18], [16], [18]. However, others, such as Ambaye et al. [30], are of the opinion that more laboratory-scale studies with matrix solutions and real wastewater are needed to understand the optimal operating conditions and to elucidate the degradation mechanisms of such a complex matrix. More research is required, both laboratory and full-scale, on the influence of operating parameters in real waters, such as temperature, pH of the water to be treated, its organic matter content, and inorganic ions, dissolved oxygen concentrations, sediment geochemistry, etc. [20], [25], [44]. Comprehensive studies are needed to address the effects of field conditions, which are more complex than laboratory conditions [38]. Additionally, further works are suggested to be done at the laboratory level with PFAS concentrations similar to those found in the environment. Some studies conducted report concentration levels approximately between 10 and 50 $\mu\text{g/L}$ [10] and PFAS in the environment are usually found at trace levels [63].

Before evaluating the performance of AOP in PFAS degradation with real water matrices, determining the degradation parameters and decomposition products of each treatment technology is of the utmost importance [10], [27], [34]. Additionally, developing a design basis to confidently employ PFAS treatment technologies and optimize processes, with knowledge of the specific conditions of the actual water to be treated, is a crucial

aspect [34]. Moreover, it is necessary to address the drawbacks related to high costs, complex operation and secondary pollution associated with the process [10], [27], [48].

Regarding the heterogeneous photocatalysis process, Leonello et al. [26] stated that, despite the different attempts to increase the photocatalytic activity of TiO_2 in PFAS degradation, this photocatalyst generally exhibits relatively low activity, which would reduce the implementation of the process at large scale. An alternative way to improve degradation is to seek other semiconducting materials that show a better activity than TiO_2 . For Wang et al. [10] the modification of photocatalytic materials warranted further research into its use in industry. On the other hand, future research should focus on the development of electrodes with new materials, as they play a very important role in electrochemical oxidation [50].

3.5.4. AOP Operating costs

One of the major limitations of the field application of AOP in the treatment of PFAS is the high cost associated with this. In this regard, some authors consider that research should be conducted on the development of new AOP that do not require excessive use of chemicals, high energy consumption and are easy to operate and implement [16], [18], [20], [27], [40], [48], [55]. To reduce the cost of electrochemical oxidation, Foote et al. [34] proposes the use of solar panels, which could likely serve as a source of electricity for this treatment technology and thus significantly reduce the costs related to energy consumption. Similarly, Foote et al. [34] and Pilli et al. [39] consider that further research should be conducted on increasing the lifetime of the BDD-based electrode, whose use is efficient in electrochemical oxidation treatments, although it should be noted that its construction is still a costly and complex task.

As an alternative to enhance the economic and environmental advantages of PFAS treatment by means of AOP, treatment trains are proposed [13], [20], [29], [34], [35], [44], [48], [64]. These consist of the combination of individual treatment processes either using several AOP at the same time or their combination with other treatment methods [13], [20], [29], [34], [35], [44], [48]. Several treatment trains have been proposed for the degradation of PFAS in water. For Lu and coworkers [48], a treatment train system combining nanofiltration, electrochemical anodic oxidation and electro-Fenton degradation (NF-EO/EF) could be an optimal technical solution.

Table 1. Advantages and disadvantages of the use of AOP for PFAS degradation in water

Process	Advantages	Disadvantages
Photocatalysis	<ul style="list-style-type: none"> • Catalyst regeneration, so that it can be reused [18]. • Operation under visible radiation [29]. • Availability of several catalytic materials [18], [26]. • TiO₂-based photocatalysts have up to 100% removal of PFAS [18]. • Operation at room temperature [28]. • Low energy consumption [28]. 	<ul style="list-style-type: none"> • Extreme operating conditions and large amounts of chemicals are sometimes needed [18]. • Secondary pollutants can be produced during the degradation process [28], [29]. • Low efficiency [28].
UV/S ₂ O ₈ ²⁻	<ul style="list-style-type: none"> • Efficient process for the degradation of PFAS under a wide variety of operating conditions [18], [57]. 	<ul style="list-style-type: none"> • A high chemical consumption is required [18], [57]. • Relatively long treatment times are required [18], [57].
Plasma	<ul style="list-style-type: none"> • Efficiency in the degradation of PFAS and their by-products [11], [38], [55]. • Fewer chemicals are required compared to other AOP [29]. 	<ul style="list-style-type: none"> • High energy consumption, although there is still no data on the economic feasibility of this technology [11], [38], [51], [55]. • Limited efficiency for pollutant trace levels [11]. • Formation of undesirable toxic products [51].
Sonochemical oxidation	<ul style="list-style-type: none"> • Usually does not generate secondary pollutants [16]. • High PFAS degradation efficiencies [16], [39]. • Ease of operation [16], [38], [39]. • Operation at room temperature [28]. • No need of adding chemicals [28]. 	<ul style="list-style-type: none"> • High power consumption [16]. • Large-scale US reactors can corrode transducer materials after continuous use, resulting in a limited energy transfer and problems such as a poor heat dissipation [11], [16]. • Lack of large-scale experiments [16], [28], [30].
Electrochemical oxidation	<ul style="list-style-type: none"> • Feasible and efficient for a complete mineralization of PFOA and PFOS [27], [34]. • Operation at room temperature and pressure [27], [34], [37]. • Ease of automation. • It can be conducted through direct and indirect anodic oxidation [20]. • It is the most effective AOP for PFOS [28], [37]. 	<ul style="list-style-type: none"> • Reduced efficiency in the degradation of short-chain PFAS [11], [27]. • Formation of undesirable toxic products is evident [11], [20], [21], [27], [37]. • High cost and difficult construction of some electrodes, like BDD [11], [13], [20], [28], [38]. • High energy costs [40]. • Easy electrode corrosion and short lifetime [11], [57]. • Limited feasibility of large-scale process [28], [55].
Treatment train (combination of treatment technologies)	<ul style="list-style-type: none"> • Improved overall efficiency of in situ degradation treatment of PFAS [16], [34], [38], [48]. • Decreased likelihood of secondary pollution due to PFAS degradation by-products [16], [38], [48]. • Reduced chemical and energy consumption, so that extreme conditions to activate or operate the treatment process are not required (high acidity, alkalinity, temperature, etc.), unlike technologies used individually [38], [48]. 	<ul style="list-style-type: none"> • The costs associated with the combination of technologies have not been reported yet [11], [48].

For Foote et al. [34], the most viable treatment trains were the use of anion exchange resins with electrolysis and nanofiltration with ozonation. Both investigations agreed that the treatment trains would substantially improve the efficiency and cost-effectiveness of PFAS removal from water. Combining electrochemical oxidation with other remediation options (e.g., biological treatment, adsorption and membrane filtration, among others) can reduce the treatment costs and improve PFAS degradation efficiency [50].

However, further studies investigating the optimal combination of technologies are required [47]. On the other hand, Marquinez-Marquinez et al. [44] proposes that to reduce costs, renewable energies should be used in the AOP. Therefore, the application of renewable energies in AOP performance should be improved, which could result in an ecological and economic stability of the process.

In this regard, the importance of further improving treatment technologies and reducing the associated total cost of chemicals and energy consumption for real water applications are highlighted [6], [16], [27], [34], [48].

4. Conclusions and recommendations

The degradation of PFAS represents a major challenge for several sectors (academic, public health, industrial and regulatory, among others). It is evident that significant efforts have been made to develop different technologies capable of degrading these substances. Several studies agreed on the advantages of the application of AOP for the degradation of PFAS. However, most of these oxidation systems have only been tested at laboratory scale, and full-scale industrial and practical applications have rarely been carried out, high cost being one of the main reasons for the lack of scalability of the processes.

Larger and more rigorous studies are crucial to produce scientific evidence on the efficiency of AOP in PFAS degradation. Furthermore, studies are needed to help mitigate the drawbacks related to the application of AOP in PFAS degradation. In this regard, some authors recommend the use of solar panels to reduce the costs associated with energy consumption. However, it is suggested that other alternative energies are explored, to optimize the treatment of PFAS through AOP and to favor the scalability of these processes. Other authors suggest that by using raw materials with photocatalytic properties can be economically manufactured, as well as fixing these materials directly to the surface through which water is passed to reduce the cost associated with the consumption of chemical products. Nonetheless,

these new materials could increase the toxicity of the process.

Several AOP used for treating PFAS in water are still in the development stage and others are still evolving. Therefore, in the near future, it is expected that the limitations related to their application will have been overcome to favor the viability of these processes at a real scale, generating a positive impact on human health and the environment.

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Autor Contributions

L. Marín-Marín: Investigation, Methodology, Writing – original draft. A. Rubio-Clemente: Conceptualization, Methodology, Writing –review& editing. G. Peñuela: Supervision, Writing -review & editing.

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Conflicts of Interest

The authors declare no conflict of interest.

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