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Review

Recent Developments in Advanced Oxidation Processes for Organics-Polluted Soil Reclamation

Crina Calenciuc [1,2], Antía Fdez-Sanromán [2], Gabriela Lama [2], Sivasankar Annamalai [3], Angeles Sanromán [2] and Marta Pazos [1,*]

[1] Department of Environmental Engineering and Management, Faculty of Chemical Engineering and Environmental Protection, Gheorghe Asachi Technical University of Iasi, Bulevardul Profesor Dimitrie Mangeron 73, 700050 Iasi, Romania; biosuv@uvigo.es
[2] CINTECX, University of Vigo, Campus as Lagoas-Marcosende, 36310 Vigo, Spain; antia.fernandez.sanroman@uvigo.es (A.F.-S.); glama@uvigo.es (G.L.); sanroman@uvigo.es (A.S.)
[3] School of Architecture, Civil, Environmental and Energy Engineering, Kyungpook National University, Daegu 41566, Korea; asivasankar87@gmail.com
* Correspondence: mcurras@uvigo.es

Abstract: Soil pollution has become a substantial environmental problem which is amplified by overpopulation in different regions. In this review, the state of the art regarding the use of Advanced Oxidation Processes (AOPs) for soil remediation is presented. This review aims to provide an outline of recent technologies developed for the decontamination of polluted soils by using AOPs. Depending on the decontamination process, these techniques have been presented in three categories: the Fenton process, sulfate radicals process, and coupled processes. The review presents the achievements of, and includes some reflections on, the status of these emerging technologies, the mechanisms, and influential factors. At the present, more investigation and development actions are still desirable to bring them to real full-scale implementation.

Keywords: AOP; soil; pesticides; PAH; petroleum hydrocarbons

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1. Introduction

As a key constituent of the natural environment, the soil is crucial for climate change mitigation, farming production, nature, and biodiversity preservation, and is the foundation of human health and wealth [1]. However, it is polluted in several ways every day, mainly from agricultural activities, but also from the industrial, maritime and urban fields [2]. Accordingly, a series of pollutants end up in the soil due to human activities, but natural events such as hurricanes, floods, and land degradation can also influence the storage, degradation, and toxicity of organic pollutants [3]. Any contaminant present in a soil matrix may lead to the contamination of public health and groundwater and, consequently, prevent the soil's use.

Among pollution types, the one attracting most attention in the scientific field is organic contamination, because of the different nature of these compounds and their significant impact on soils' health. Accordingly, governments and public organizations have recognized the potential harm of organic pollutants and are treating the polluted soil for human health protection, and achieving sustainable development has become a priority. Thus, in May 2020 the European Commission adopted the new EU Biodiversity Strategy for 2030, where among different issues, soil sealing and the rehabilitation of contaminated brownfields will be addressed [4].

The most common organic pollutants found in soils come from a wide range of chemical classes that include pharmaceuticals, personal care products, dioxin-like compounds, nitrosamines, building materials, clothing, food packaging, and pesticides. Based on the recent literature review, the most worrying contaminants are polycyclic aromatic hydrocarbons (PAHs; e.g., acenaphthene, phenanthrene, fluoranthene, and pyrene); monomeric aromatic hydrocarbons (BTEXs; e.g., benzene, toluene, ethylbenzene, and xylene); chlorinated compounds (e.g., polychlorinated biphenyls (PCBs), polychlorinated dibenzo-para-dioxins (PCDDs), and polychlorinated dibenzofurans (PCDFs)); petroleum hydrocarbons (PEH; e.g., alkanes and cycloalkanes); and pesticides (e.g., lindane, atrazine, oxyfluorfen, and chlorpyrifos) with their degradation products [5]. Some of these organic pollutants have a high toxicity, are persistent, bioaccumulative, and can lead to negative impacts on soils' fertility or even human health. They are included in the Stockholm Convention on Persistent Organic Pollutants (POPs), which determines the elimination or reduction of the environmental release of the listed POPs [5]. It is necessary to conduct long-term monitoring of organic contaminants in the soil to estimate the level of pollution and also to have an accurate picture of their potential impacts on soil sustainability; in addition, new, less costly, and more effective methods for pollution prevention are necessary for environmental protection.

Nowadays, numerous technologies are available and can be applied to solve the organic pollution problem. Among them, advanced oxidation processes (AOPs) allow the transformation of organic pollutants with different chemical structures into elements that are more biodegradable and/or less toxic by using oxidizing agents. Thus, AOPs are being widely studied and are promising, efficient, and environment-friendly methods for the removal of diverse types of contaminants. They are based on the use of highly active radical species [6] such as the hydroxyl radical ($^{\bullet}OH$) and another species of similar reactivity such as sulfate radical anion ($SO_4^{\bullet -}$), and these radicals react with the majority of organic substances at rates often approaching the diffusion-controlled limit [7]. In most AOPs, the objective is to produce strong oxidants, mainly hydroxyl radicals ($^{\bullet}OH$), with E^0 ($^{\bullet}OH/H_2O$) = 2.8 V/NHE, being able to non-selectively oxidize any organic pollutant up to its mineralization (i.e., conversion to CO_2 and water), as shown in Reactions (1) and (2) below [8,9]:

$$^{\bullet}\mathrm{OH} + \text{Organic pollutant} \rightarrow \text{Primary intermediates} \tag{1}$$

$$\text{Primary intermediates} + {}^{\bullet}\mathrm{OH} \rightarrow \mathrm{CO_2} + \mathrm{H_2O} + \text{Inorganic ions} \tag{2}$$

Depending on the physicochemical process directing the formation of these species, AOPs can be classified into non-photochemical processes (e.g., ozonation, Fenton based processes, sulfate-based processes, permanganate, and ultrasounds) and photochemical processes (e.g., UV, UV/hydrogen peroxide, UV/ozone, and photo-Fenton processes). However, the photochemical process is not recommendable for in situ soil remediation treatments due to the lack of visible light activity inside the soil matrix [10]. As a result of their properties, AOPs are considered more effective and economical, because the application of mechanical remediation on polluted soils is expensive and invasive and the biological methods can be limited regarding environmental conditions and time-consuming processes [11].

AOPs and their applications were an interest for researchers beginning only around 1995, and it continues nowadays, since the number of investigations regarding its utilization in soil remediation is rising considerably [10]. Consequently, these processes are an attractive alternative for the treatment of contaminated soil with organic pollutants, and in recent years a growing number of papers have been published regarding their single or combined use. Thus, AOPs were combined with biological, chemical, electrokinetic processes, and between themselves to achieve a high degradation efficiency, because combining one or more remediation methods enables a synergy that exploits the strengths and minimizes the disadvantages of individual technologies [12].

The last review regarding soil remediation using AOP was published by Cheng et al. [10]. That review summarized published papers from 1995 to 2015, showing that in that period the majority of studies were focused on the Fenton reaction and ozonation. However, in the most recent years, research has moved to other technologies, such as sulfate-based processes [13]. This evolution was also observed in an analysis of published papers extracted from a Scopus search using the keywords [soil] AND [advanced oxidation processes] AND [organic]. The results of an analysis of the co-occurrence of keywords using bibliographic network maps from VOS Viewer® are shown in Figure 1 [14]. This figure describes the co-occurrence network of the top 26 keywords with the most frequent occurrences. The nodes represent the keywords, and each line denotes the co-occurrence relationship between the connected keywords. As illustrated in Figure 1, the colour of each keyword is based on its average appearing year. Before 2010, the studies were mainly focused on Fenton based processes (Fenton, electro-Fenton, and photo-Fenton). From this year onwards, the number of scientific publications has increased considerably. There is a trend in terms of the solutions being sought, as ozonation techniques, sulfate reactions, and biochar are more frequently used in improving the quality of soils.

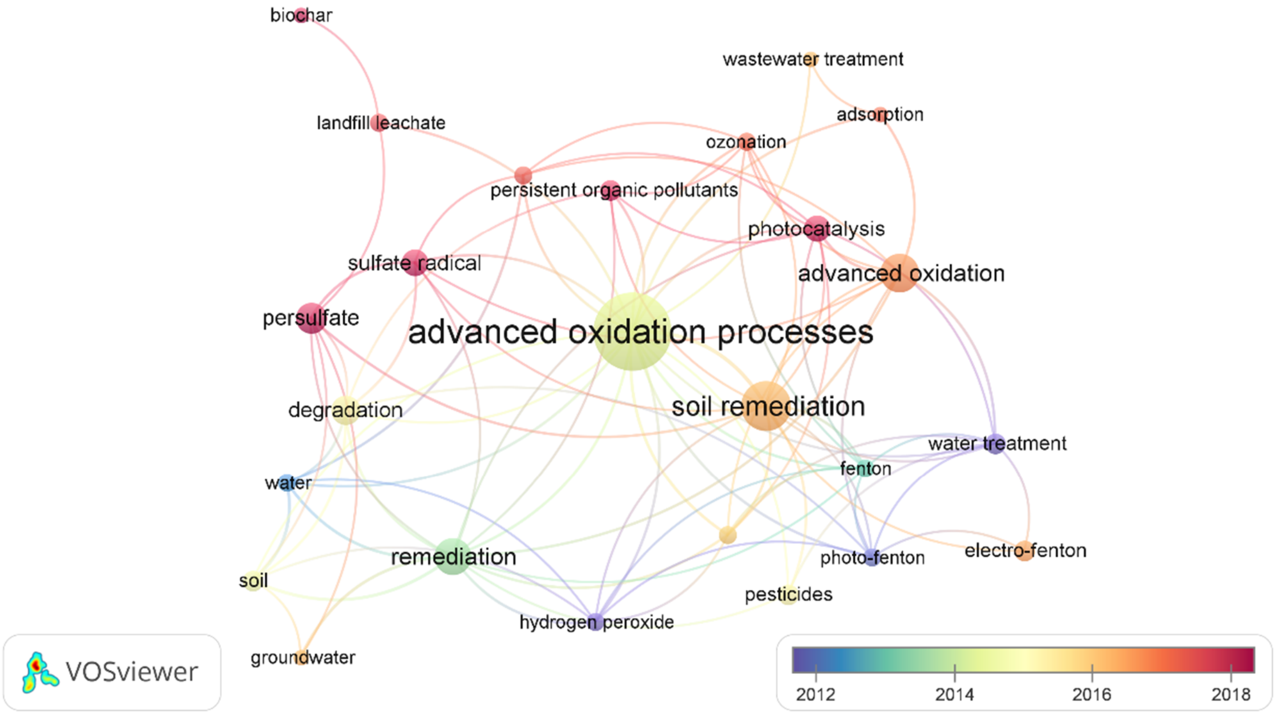

Figure 1. Keyword and overlay visualization co-occurrence analysis of advance oxidation processes to soil research using the VOS Viewer®. The blue nodes represent the keywords appearing earlier, whereas the red nodes reflect their recent occurrence.

The most common organic pollutants found in the research papers in this review are summarized in Figure 2. PEH, PAHs, and pesticides have focused the attention of the current research, and most of the reviewed papers (75%) are related to these pollutants. However, the presence of new emerging pollutants, such as PBDE and PCB, is attracting attention as a result of their prevalence in the environment and negative effects.

This review aims to present a complete description of the recent publications, from January 2016 to June 2021, regarding the use of currently available AOP-based soil remediation techniques used against organic pollutants. Besides these, the main characteristics of the processes, influential factors, and environmental impacts of AOPs will be looked over, delivering an ample reference for choosing the best option that suits the remediation of contaminated soils.

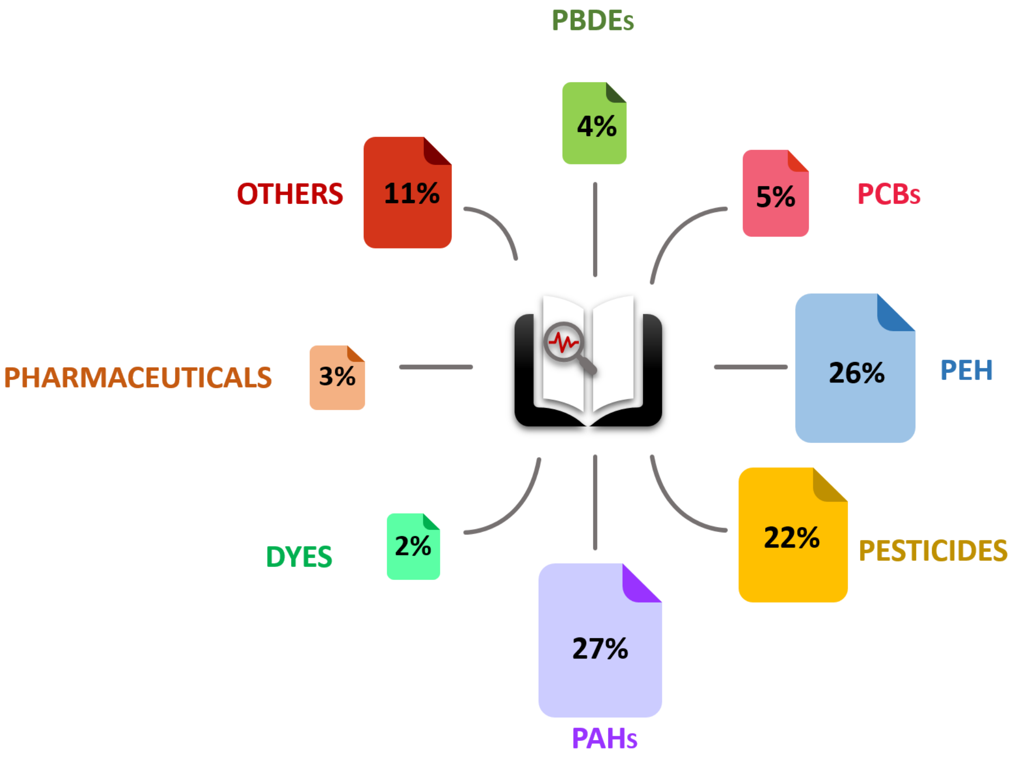

Figure 2. Distribution of contaminants studied in the papers evaluated in the present review from January 2016 to July 2021.

2. Data Sources

The scientific articles on which this literature is based have been searched on Scopus, published up to 15 July 2021. The literature search included the keywords [soil] AND [advanced oxidation processes] AND [organic] and it was limited to articles written in English starting from January 2016.

Next, the main pieces of information about each article were summarized in a table, including the name of the author and the title of the publication, the type of organic pollutants, the oxidation method used with its removal efficiency, the year of publication, and important observations regarding the processes' functionality (e.g., influential factors, advantages, and disadvantages). During this procedure, only the experimental papers suitable to the review topic were selected, giving an ample and accurate representation regarding AOPs in organically contaminated soils, providing process improvements, and new and important factors for the future prospects of AOPs research.

In Figure 3 the experimental publications about soil remediation by AOPs in the last 6 years (up to 15 July 2021) are summarized. It is interesting to point out that, as a result of the interest in the topic and the COVID-19 pandemic, the number of review articles on this issue has increased in the last 2 years.

Figure 3. Published papers on the studied topic from January 2016 to July 2021.

3. Fenton Process

The Fenton process is considered to be one of the first AOP series, and it is attributed to H. J. H. Fenton, who in 1894 reported that H_2O_2 could be activated by ferrous (Fe^{2+}) salts to oxidize tartaric acid. However, it was not applied as a remediation process to degrade organics in aqueous solution until the late 1960s [15], and the use of this treatment for the removal of organics from soils was first evidenced in the 1990s [16]. However, at present it is the most common process for soil remediation [17].

During the Fenton process, H_2O_2 is decomposed in the presence of iron, which produces hydroxyl radicals according to the following Reaction (3) with a kinetic constant value of 70 M^{-1} s^{-1} at pH = 3 [18,19]:

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + {}^{\bullet}OH + OH^- \tag{3}$$

The biggest advantages of the Fenton process are that it can be carried out at room temperature and atmospheric pressure with a high performance and non-toxicity [10]. However, some limitations are found in the conventional Fenton process, an important one of which is a strong dependence on the pH. The optimum pH value is between 2.5 and 4.0, if the pH is under this range, H_2O_2 can solvate protons to form oxonium ions ($H_3O_2^+$), which increases the stability of H_2O_2 and reduces its reactivity with ferrous ion; if the pH is above this range, the dissolved fraction of iron species is reduced as colloidal ferric species appear [10,20].

In soil remediation, a Fenton treatment can be applied in situ or ex situ, and the reactions can be homogeneously catalysed (conventional Fenton) or heterogeneously catalysed (Fenton-like) by the iron mineral content (Figure 4) [21]. However, there is no real difference between the classical Fenton and Fenton-like removal process, because, from a mechanistic point of view, the degradation of the pollutants occurs via the hydroxyl radicals, ${}^{\bullet}OH$, that are generated [22].

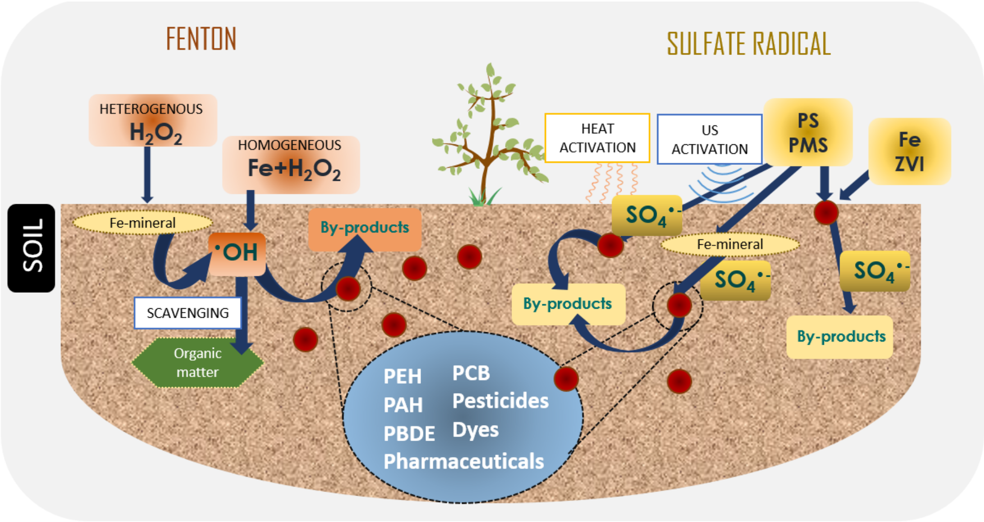

Figure 4. Mechanisms of Fenton and sulfate radical processes in organics-polluted soil.

Different factors can be considered to promote de hydroxyl radical generation inside the solid matrix. Thus the type of soil and pH, iron source, solubilizing agents, and organic matter has been studied in detail. The recently published articles on the Fenton process for soil decontamination are presented in Table 1. It must be pointed out that the reviewed articles develop the treatment processes by mixing the soil with the oxidants (a batch process at lab scale). A few studies have tried to simulate natural conditions in the flow system by using dynamic column experiments under saturated and unsaturated conditions [23,24].

Table 1. Selected published articles on the Fenton-based process for soil decontamination.

Process Description	Process Parameters	Contaminant & Concentration	Higher Removal Efficiency (%)	Reference
Fenton	Fe^{2+}3.0 g/kg; H_2O_2 120 g/kg; (1:67); pH = 3.0–7.1	Aristolochic acid 500 µg/kg	>97	[25]
Fenton	(H_2O_2):$(FeSO_4)$ = 20:1 sequential, four times/day with intervals of 2 h over 5 d.	16 PAHs 933.9–2155.4 mg/kg	40–70	[26]
Fenton	0.1 M EDTA 3% H_2O_2	Diesel 3300 mg/kg	70	[27]
Fenton	30% H_2O_2 = 1.2 mmol/g; $FeSO_4$,$7H_2O$ = 0.2, 0.4 mmol/g	16 PAHs gas plant 263.6 ± 73.3 mg/kg; coking plant 385.2 ± 39.6 mg/kg	60–85.4	[28]
Fenton	Fe^{2+}:H_2O_2 1:5; H_2O_2 0.8 mol/L	Chlorpyrifos (CP) 11,000 mg/kg; 4-bromo-2-chlorophenol (BCP) 10,000 mg/kg.	CP 92; BCP 97	[29]
Fenton	ZVI 0.25 g/g and H_2O_2 2 M	Polychlorobiphenyls (PCB 18-PCB 194) 450.2 ± 31.8 mg/kg	PCB 28 90; PCB 118 49	[24]
Fenton + iron inside oil	H_2O_2 900 mM/oil-absorbing Fe 0.25–0.35 mg oil/mg Fe	short and long-chain alkanes 3937 mg/kg and 1490 mg/kg, respectively	62–74	[30]
Fenton + ethyl lactate (EL)	H_2O_2 0.5 M, Fe^{2+} 0.05 M, EL% = 25%	TPH 5000 mg/kg	96.74	[31]

Table 1. *Cont.*

Process Description	**Process Parameters**	**Contaminant & Concentration**	**Higher Removal Efficiency (%)**	**Reference**
Fenton + haemoglobin (Hb)	H_2O_2:Hb = 3	16 PAHs 259 ± 5.5 mg/kg; benzo(*a*)pyrene (BaP) 10 mg kg	PAHs 89; BaP 85	[32]
Fenton + EDTA	H_2O_2 129.5 mL/kg; $FeSO_4{\cdot}7H_2O$ 14.6 g/kg; EDTA 16.2 g/kg	Pyrene 1200 ± 200 mg/kg	~20	[33]
Fenton + EDTA	H_2O_2/Fe(II)/PAH (10^2–10^5/10^2/1).	PAH 4.23 mg/kg	~30	[34]
Fenton + sodium pyrophosphate (SP)-chelated	Fe^{2+}:SP:H_2O_2 (1:1:20)	PCBs 63.9–739.0 mg/kg	87.5 and 77.1	[35]
Fenton + iron inside soil organic matter	H_2O_2 900 mM/Fe chelated with SOM S/L 5 g/60 mL	TPH 9068 mg/kg	67	[36]
Fenton + iron inside soil organic matter	H_2O_2 900 mM Fe-SOM 837 mg/kg	TPH ~4500 mg/kg	66.8	[37]
Fenton + flow system	H_2O_2 600 mM; Fe^{II} 60 mM	β-HCH 45 mg/kg; lindane 25 mg/kg	β-HCH ~70; lindane ~90	[23]
Fenton like	H_2O_2 = 1.5 mol/L	Gasoline 10 g/kg	~60–90	[38]
Fenton like	H_2O_2 = 2.8%	BTX 0.05–80 mg/kg	Benzene ~99; toluene ~86; xylene ~74	[39]
Fenton-like + flow system	Endogenous Fe 9.8 g/kg; H_2O_2 4 mmol; L/S = 20/1	Pentachlorophenol 6 mg/kg	27	[24]
Fenton-like + ZVI + EDTA	30% H_2O_2/endogenous Fe/EDTA 20/1/1	TPH 30.51 ± 0.46 g/kg	80	[40]
Fenton-like + humic acids	Humic acids (from organic wastes, 10 g/L) and KH_2PO_4 (8.2 g/L); H_2O_2 30%; Fe = 20 g/kg	Diesel-HC > 12 5500 ± 1000 mg/kg	90	[41]
Fenton-like + humic acids	Humic acids 5 L/Kg; H_2O_2 6% *v*/*v*; Fe = 29.7 g/kg	3-chlorophenol	>90	[42]
Fenton-like + pyrophosphate-chelated	Fe:Pyrophosphate:H_2O_2 (1:1:10)	PCB 70.67–80.00 mg/kg	79.4–91.4	[43]
Fenton-like + steel converter slag (SCS)	SCS 80 g/kg; 20 g soil; H_2O_2 solution (15% *w*/*w*) 20 mL;	Antrazine 617.5 mg/kg	93.70	[44]
Fenton-like + magnetite	1200 mL H_2O_2 30%; 6 g magnetite; 6 g soil	16 PAHs 1210 μg/g	80	[45]
Fenton-like + magnetite + preheating	H_2O_2 10% + magnetite (20:1); pre-heating 150 °C	16 PAHs 1089–1121 mg/kg	~50	[46]
Fenton-like + magnetite + ascorbic acid (AA)	Magnetite/AA/H_2O_2 25 g/kg/125 mM/500 mM	Pentachlorophenol 6 mg/kg	95	[47]
Fenton + clay catalyst	Fe clay catalyst = 1.5 g; 30% H_2O_2	Phenanthrene 200 mg/kg	83	[48]
Fenton + $KMnO_4$	(H_2O_2):(Fe^{2+}) = 10:1, followed by $KMnO_4$ 0.4 M, T = 60 °C	27 PAHs 3090 ± 104 mg/kg	71	[49]

The iron mineral present in the soil matrix can allow the generation of reactive oxidant species for the strong degradation of organic pollutants (Figure 4). Thus, iron minerals are efficient in catalysing Fenton-like reactions through a large range of pH values. There are different types of iron minerals, including ferric Fe^{III} (ferrihydrite, goethite) and mixed Fe^{II}–Fe^{III} oxides (magnetite and green rust) and they catalyse oxidation by the heterogeneous reaction. Iron oxides or particular transition metals can catalyse the Fenton-like oxidation in soils at a circumneutral pH, which can be an advantage for the in situ treatment of polluted soils where the pH cannot be set. The utilization of mineral iron as a catalyst has some advantages, such as a wide pH working range or a reduction in the external agents added to the process [38,46]. Hence, Santos et al. [38] showed a 90% degradation of gasoline (10 g/kg) in different soils with the highest concentration of mineral iron (94 g/kg) with a high concentration of hydrogen peroxide (1.5 mol/L) and without the addition of iron. In addition, some results from that same study, suggest that the degradation efficiency of the Fenton processes is also strongly influenced by the physicochemical properties of the soil, including its organic matter and clay contents. In Red Argisol that contains >70% sand but has a reduced iron content (30 g/kg), a high removal efficiency could also be obtained, because sand has a low sorption capacity that enhances the degradation of the pollutants by hydroxyl radicals. In soil with a high clay content (76%) and iron (216 g/kg), the degradation rate was about 60%, because clay stops the action of the hydroxyl radical. In addition, in the study conducted by de Souza et al. [39], iron minerals (hematite) present in the soil specimen (9%) increased the Fenton-like process as applied to the degradation of BTEXs. Furthermore, the process of pre-heating the soil can enhance pollutant degradation by a Fenton-like reaction. Thus, Usman et al. [46] demonstrated a thermal pre-treatment at 150 °C for one week under an inert atmosphere, before being treated by the oxidation process, enhancing the magnetite-catalysed oxidation of PAHs.

The presence of organic matter can have a negative effect on the Fenton process because it can act as a scavenger of hydroxyl radicals (Figure 4) [38]. However, the presence of iron linked to this organic matter could enhance the treatment. Thus, Xu et al. [50] obtained a high long-chain alkane (C21–C30) removal rate in a Fenton system with iron–soil organic matter, which was 1.6 times bigger than that obtained in a Fenton system with typical iron. Similarly, it was found that the oil-absorbing Fe could bind with hydrophobic humic-like acid and fulvic-like acid to enhance the degradation process. Accordingly, Xu et al. [36] reported that the oxidation of macro-crude oil in soil by H_2O_2/oil-absorbing Fe connected with a high solid organic matter content was much greater than that with a low solid organic matter content. It was postulated that the efficient degradation of macro-crude oil using oil-absorbing Fe bound with a high solid organic matter content was catalysed by the H_2O_2. The large proportion of hydrophobic humic-like acid and fulvic-like acid connected to the oil-absorbing Fe increased the hydrophobicity of the oil-absorbing Fe, allowing abundant macro-crude oil to be easily absorbed by the oil-absorbing Fe bound with high solid organic matter.

To avoid a pH limitation and to enhance the process, the use of solubilizing agents (chelating or solvents) were also proposed as a solution [31,40]. Chelating agents are generally organic compounds and can be used at a high pH to establish complexes with Fe(III)/Fe(II), maintain them soluble, thus increasing the production of oxidative species by the reaction of H_2O_2 with Fe(III)/Fe(II) in the Fenton process. In recent years, different types of chelators have been studied, including catechol, cyclodextrin, nitrilotriacetic acid, ethylenediaminetetraacetic acid (EDTA), gallic acid, citric acid, oxalic acid, humic substances, malic acid, sodium citrate, and other amino-poly-acetic acids [15,46,51]. Therefore, Ouriache et al. [40] studied the Fenton-like process with the addition of EDTA and without modification of the pH in the remediation of petroleum-contaminated old polluted soil. According to their results, EDTA led to a high removal efficiency (80%) independently of the iron addition. The EDTA addition enhanced the performance of the Fenton-like process and encouraged the formation of hydroxyl radicals, which efficiently degraded the PEH in the contaminated soil [40]. For another example, Zingaretti et al. [41] carried out the

Fenton-like reaction with the addition of humic acids, extracted from compost obtained from organic wastes, on a diesel-contaminated soil. When the humic acids (10 g/L) were used in combination with KH_2PO_4 (8.2 g/L), the H_2O_2 lifetime growth was around 150 h (without the addition, the H_2O_2 lifetime was only 1 h), and the removal rate was above 90% compared to around 75% in the case of using KH_2PO_4 or humic acids alone.

Similarly, solvents can be used to solubilize pollutants to enhance the removal. Thus, Ahmadkalaei et al. [31] used ethyl lactate to intensify the removal of soil polluted with total petroleum hydrocarbon (TPH). They found that an optimal concentration of 10% of ethyl lactate should be used to achieve a removal of higher than 96%.

Although solubilizing agents are a promising alternative to promote pollutant oxidation, their application is limited by their cost, toxicity, and potential adverse effects on the oxidation efficiency due to their poor self-consumption. Chelating agents like EDTA can be harmful to the environment due to their persistence and their role in enhancing heavy metal mobility/bioavailability. Besides, their self-oxidation would lead to the loss of their chelating ability, which is necessary to improve the pollutant degradation [46].

Similarly, Sun et al. [43] conducted a study regarding the influence of the soil type on the Fenton process, where they used as representative soils red soil (RS), paddy soil (PS), fluvo-aquic soil (FS), and black soil (BS) contaminated with polychlorinated biphenyls (PCBs), and the degradation process of pollutants was in a pyrophosphate-chelated Fenton-like system. They noticed that the soil type affected the degradation efficiency, as an inadequate dosage of pyrophosphate to the amount of exogenous iron, soil components such as iron oxides, and the soil organic matter–Fe complex, could be an important factor in compensating for the insufficient proportion of the Fenton reagent and thereby enhanced the degradation efficiency of PCBs. However, an excess pyrophosphate dosage did not positively effect the useless decomposition of H_2O_2, even though this influenced the increase in soluble Fe^{3+}. Sun et al. [43] achieved high organic pollutant removal rates in all four types of soils (RS > BS > PS > FS; 1 h), which was a synergistic effect of the quantity of soluble Fe^{3+} and the availability of H_2O_2 under the soil components (soil organic matter, clay minerals, and Fe/Al oxides). Furthermore, this showed that the pyrophosphate-chelated Fenton-like reaction is suitable for the treatment of various types of PCB-contaminated soils.

4. Sulfate Radical Process

In recent years, numerous published papers on AOPs' applications have investigated the treatment of water and soil remediation using sulfate-radical-based processes [13,52]. The main reason is that $SO_4^{\bullet-}$ ($t_{1/2}$ = 30 μs), compared to $^{\bullet}OH$ ($t_{1/2} \leq 1$ μs), has a longer half-life and is more electrophilic, which drives a long-term relationship with contaminants [53,54]. Furthermore, several comparative studies have demonstrated that sulfate radicals showed a higher degradation extent than hydrogen peroxide [48,55] and the synergistic effect of the combination of both oxidants [56].

The generation of $SO_4^{\bullet-}$ radicals can be accomplished by using two main precursors, peroxydisulfate (PS, $S_2O_8^{2-}$) or peroxymonosulfate (PMS, HSO_5^-) [53].

PS is stable at ambient temperature and can be activated by heat, transition metals, pH, ultraviolet (UV) light, or other elements to form a highly reactive sulfate radical, $SO_4^{\bullet-}$, as shown in Reaction (4). This radical is a stronger oxidant (E^0 = 2.6 V) than the persulfate anion, giving it a great capacity for organic destruction [57,58].

$$S_2O_8^{2-} + \text{activator} \rightarrow SO_4^{\bullet-}(\text{energy transfer}) + SO_4^{2-}\ (\text{electron transfer}) \tag{4}$$

PMS can be obtained from potassium or sodium salts or as a triple salt called OXONE ($KHSO_5 \cdot 0.5KHSO_4 \cdot 0.5K_2SO_4$) [59]. These reagents can be activated similarly to the process previously described for PS. However, depending on the activation process, PMS can generate sulfate radicals or hydroxyl and sulfate radicals, as shown in Reaction (5) [60]:

$$HSO_5^- + \text{activator} \rightarrow SO_4^{\bullet-} + OH^-\ (\text{electron transfer}) \text{ or } ^{\bullet}OH\ (\text{energy transfer}) \tag{5}$$

Among them, PS is commonly used in soil treatment, because it costs less and has a shorter environmental retention time. Furthermore, the energy consumption for generating $SO_4^{\bullet-}$ by PS is 140 kJ/mol, which is less than that of PMS, which is 213.3 kJ/mol [61].

Activation by heat is an efficient means of generating radicals by PS or PMS. A high temperature (>50 °C) can lead to the fission of the O–O bond to form the sulfate radicals [62]. The activation energy also depends on the pH, accordingly that of the PS reaction is 119–129 kJ/mol, 134–139 kJ/mol, and 100–116 kJ/mol under neutral, basic, and acidic conditions, respectively. Thus, the rate constant of sulfate radical generation is from 1×10^{-7} s^{-1} at 25 °C to 5.7×10^{-5} s^{-1} at 70 °C and pH = 1.3 [57]. However, this method is not recommended for large-scale processes because of its energy consumption.

Chen et al. [63] successfully degraded triclosan (TCS) in various arable topsoils through a thermally activated PS. The activation energy Ea was calculated to be 74.3 kJ/mol when 18.8 mM of PS was used as an oxidant for 88% of TCS removal over 360 min of reaction. The degradation efficiency was affected by the pH; a superior degradation rate was obtained with an acid pH as a result of the TCS' speciation. Chen et al. [63] also reported that both $SO_4^{\bullet-}$ and $OH^{\bullet}$ were present during the following Reaction (6), although $SO_4^{\bullet-}$ was the dominant radical oxidant:

$$SO_4^{\bullet-} + OH^- \rightarrow OH^{\bullet} + SO_4^{2-} \quad (6)$$

Similarly, in a study conducted by Chen et al. [51] it was reported that in p-nitrophenol degradation, the pH had an important effect on the PS degradation process. The pH was adjusted and controlled by the addition of a phosphate buffer solution (25.8 mM-Na_2HPO_4 + NaH_2PO_4), and the results showed a slight reduction in the degradation process, because the buffer salts were competing with the contaminant molecule in consuming the $SO_4^{\bullet-}$. However, the alkaline activation of PS using NaOH was verified for the removal of a complex compound of chlorinated PAHs by García-Cervilla et al. [64] and Lominchar et al. [65].

Different studies showed that microwave heating is higher compared to conventional heating, which leads to a faster contaminant degradation. Microwave heating distributes the required energy faster, leading to the movement and collision of PS molecules, rupturing molecular bonds and determining the formation of $SO_4^{\bullet-}$ [57,66]. The microwave/PS system degraded up to 90% of ethyl-parathion, demonstrating that it is time-saving, cost-effective, and can be conducted under moderate conditions. Hence, some researchers have claimed that the microwave heating activation process was a promising technology for the treatment of pesticide-contaminated soils [66].

Likewise, in the Fenton treatment, the pre-heating process can increase the degradation process by PS. Thus, the investigation accomplished by Ranc et al. [67] showed the efficiency of using pre-heating. Two types of ageing PAH-polluted soils were heated to 60 °C for PS oxidation and soil pre-heating to 90 °C followed by permanganate oxidation. The effectiveness of PS was higher than permanganate due to its thermal activation.

Iron salts or iron minerals have long been known as efficient activators of PS and PMS [68,69]. Ferrous ion (Fe^{2+}) reacts with PS to generate the $SO_4^{\bullet-}$, as shown in Reaction (7), with an activation energy of 50.23 kJ/mol:

$$S_2O_8^{2-} + Fe^{2+} \rightarrow Fe^{3+} + SO_4^{\bullet-} + SO_4^{-} \quad (7)$$

A PS to iron ratio of 1:1 is usually necessary to achieve the maximum contaminant removal; however, different authors have reported other ratios which depend on iron form states, the presence of chelating agents, addition methods, and target analytes [57,68].

Accordingly, Liu et al. [70], investigated the removal of ibuprofen through using a PS activated with pyrophosphate chelated Fe(II) system to demonstrate the positive effect of chelating agents in the process. Thus, at a dosage of Fe(II)-pyrophosphate:PS = 1:10, the ibuprofen degradation efficiency was 72.1% at an initial pollutant concentration of 48.4 μM/kg, showing that pyrophosphate coupled with Fe(II) notably improved the soil pollutant degradation.

Kakosová et al. [71] evaluated PS activation by Fe^{2+}, Fe^{3+}, Fe-EDTA, and Fe-citric acid chelate on a real contaminated soil collected from a petroleum lagoon deposit. Among the different studied systems, the highest contaminant removal efficiency for the degradation of petroleum contaminants was achieved by using Fe^{3+} activated PS system (around 78%), demonstrating that the iron speciation also affects the removal process and the low influence of the complexing agents used. However, Liao et al. [28] demonstrated a positive influence in the removal of PAH when citric acid was in the media. In addition, Silva-Rackov et al. [72] evaluated a diatomite-supported iron catalyst (a mixture of ferrous (Fe^{2+}) and ferric (Fe^{3+}) ions in the form of precipitated iron hydroxides) in the remediation of phenanthrene-contaminated soil. After 168 h of treatment, at a molar ratio of PS to iron content of 1:1, the degradation rate was 98%, and a small amount of PS was used, which reduced the cost of the process without compromising the removal efficiency.

Zero-valent iron (ZVI) can also be used for PS activation. It has been proposed that ZVI, as an iron source, gradually releases Fe^{2+}, as shown in Reaction (8), avoiding the scavenging effect at a high iron concentration (Reaction (9)) and enhancing the sulfate radical production seen in Reaction (7). Thus, Kang et al. [73] described that para-chloronitrobenzene reduction was enhanced significantly from 10.8 to 90.1% with an increase in the ZVI dosage from 0.1 mmol/g to 1.0 mmol/g in the ZVI-PS process.

$$\text{ZVI Fe}^{0} + S_2O_8{}^{2} \rightarrow Fe^{2+} + 2\ SO_4{}^{2-} \tag{8}$$

$$Fe^{2+} + SO_4{}^{\bullet-} \rightarrow Fe^{3+} + SO_4{}^{2-} \tag{9}$$

However, other mechanisms have been reported for the removal of pollutants in the presence of PS and ZVI. Thus, Chen et al. [74] described a two-stage remediation process: first, the reduction of para-nitrochlorobenzene by ZVI; and second, the oxidation with PS. They reported that the reduction of para-nitrochlorobenzene by using ZVI was enhanced from 66.3% to 94.1% when PS was used as an oxidant. In addition, ZVI-activated persulfate is widely utilized. Song et al. [75] tested various types of ZVI (micro/nanostructured, stearic-coated micro/nanostructured, and commercial micron-sized) for the in situ pilot-scale remediation of a PAH-polluted soil. The removal percentage achieved was higher than 60% with the studied ZVI. However, the presence of persulfate oxidizing agents provoked a long-term inhibitory effect on the soil microbial community.

Pardo et al. [76] evaluated the effect of the type of iron source (Fe^{3+} or nanoparticles of zerovalent iron (nZVI)) in the remediation of contaminated soils under flow-through conditions by the PS system. Higher efficiencies were achieved utilizing nZVI than Fe^{3+} under similar amounts of iron. Because of the acidic media, Fe^{3+} was more strongly retained in the soil column than nZVI particles, thus having a big concentration at the column entry. However, as a result of the nZVI injection, a soft profile of Fe on the column was noticed.

Similarly, Bajagain and Jeong [77] also studied the remediation of diesel oil in soil, and determined that the optimal concentrations of PMS and nZVI were 3% and 0.2%, respectively, showing the best degradation efficiency (61.2% in 2 h), and that around 96% of removal could be achieved by the consecutive application of the reagents. They also demonstrated that hydroxyl and sulfate radicals could be generated via PMS activation by nZVI.

An interesting method to synthesize nZVI for PS activation was described in the study conducted by Desalegn et al. [78]. In their study, nZVI synthesized using green mango peel extracts (GMP) was evaluated as an activator of PS in the oxidation of PEH in oil-sludge-contaminated soil. They showed a GMP-nZVI activated PS oxidation system that achieved a >90% degradation over one-week treatment. Green synthesized nZVI has greater advantages, such as a low production cost and low environmental effects. Some studies have found that the utilization of ultrasound is efficient in the activation of PS. For example, this method is found in the experiment conducted by Lei et al. [79] on the treatment of diesel-hydrocarbon contaminated soil. The author obtained a degradation of 93% of the diesel hydrocarbons in the soil, and although neither the US nor the temperature alone was responsible for the activation of the PS, the heat produced by the US was the decisive parameter in successfully activating the PS. Later, in another paper, Lei et al. [80] used the same process on polyfluoroalkyl substances (PFAS) in water and soil with encouraging results, efficiently degrading 14 PFAS in the contaminated soil, with a 62–71% degradation in soil and 100% in water. In addition, a high efficiency was obtained by Li et al. [81], who applied ultrasound and Fe in PS activation to the treatment of soil contaminated with petroleum; the degradation rate of the US/Fe/PS system was 82.23%.

As well as in the activation methods presented above, PS can be activated to produce sulfate radicals by the utilization of non-conventional methods. Biochar is environment-friendly, enhances soil fertility, and sequesters carbon, and can also be used as an efficient sorbent for pollutants. Several researchers reported its efficiency mostly in the degradation of organic contaminants in water, but Liu et al. [82] investigated the performance of PS activation by biochar to degrade bisphenol A in soil and obtained great results, showing that the addition of biochar can overcome soil acidification by counteracting the pH drop in soil. Additionally, the combination of PMS with a mechanical treatment was evaluated by Fan et al. [83] for the degradation of phenanthrene in different contaminated soils. They evaluated PMS as a co-grinding reagent in the ball-mill treatment. Their results showed that the organic matter content was negatively correlated with the degradation efficiency. Under the optimal condition (10:1 Soil:PMS), a phenanthrene removal of higher than 98% was reached. Additionally, the combination of Portland cement with persulfate oxidation was evaluated by Ma et al. [84] and Srivastava et al. [85]; their findings established that the presence of cement enhances the PAH removal by improving the sulfate radical generation and reducing the pollutants' leachability. Following in Table 2 a summary of the decontamination process using PS is presented.

Table 2. Summary of soil decontamination results reported in the literature based on the PS activation methods.

Process Description	Process Parameters	Contaminant & Concentration	Removal Efficiency (%)	Reference
PS	PS 43.0 g/kg	Phenanthrene 110 ± 20 mg/kg	36	[33]
Preheating + PS	T = 60 °C; PS 1.5 stoichiometric oxidant demand/g	PAH 14.9 g/kg	>50	[67]
Thermally activated PS	PS 18.8 mM; T 50 °C	TCS 50 mg/kg	>88	[63]
Thermally activated PS	PS 60 mmol/kg; T 80 °C	p-Nitrophenol 93 (±2) mg/kg	100	[51]
Thermally activated PS	PS 20 mM; T 60 °C	Ibuprofen 1–10 mg/kg	92–95	[86]
Thermally activated PS	PS 0.5 M; T = 50 °C;	Decabromodiphenyl ether (BDE209) 20 mg/kg	52.8	[87]
Thermally activated PS	PS 2.1 M; T = 60 °C	6 PAHs 497.47 ± 18.71 mg/kg	38.28–79.97	[88]
Thermally activated PS + Fe	PS:Fe^{2+} 1:2; T = 60 °C	Polychlorinated dibenzo-p-dioxins (PCDDs) 1350.543 pg/g; dibenzofurans (PCDFs) 2152.601 pg/g	98.3	[89]
Thermally activated PS + Fe + EDTA	PS 267 g/kg; $FeSO_4 \cdot 7H_2O$ 14.6 g/kg; EDTA 16.2 g/kg; T = 65 °C	Phenanthrene 1200 ± 200 mg/kg; pyrene 1200 ± 200 mg/kg	91 and 96	[33]
Microwave-activated PS	PS 1.0 M; T = 60 °C	Ethyl-parathion 60 mg/kg	90	[66]
Microwave-activated PS	PS 50 g/L; T = 80 °C	Phenanthrene ~1000 mg/kg	99	[90]
PS activation by Fe	PS 60 g/kg; Fe^{3+} 17 g/kg	PEH C10-C14 60.2 g/kg	78	[71]
PS activation by Fe	PS 0.2 mol/L; Fe(II)/SP 0.5/1 M	Decabromodiphenyl ether 10 mg/kg	66	[91]

Table 2. *Cont.*

Process Description	**Process Parameters**	**Contaminant & Concentration**	**Removal Efficiency (%)**	**Reference**
PS activation by Fe	Fe^{2+}:PS 1:1 M; Fe^{2+} 8.3 mM; PS 8.3 mM	Atrazine 100 mg/kg	80	[92]
PS activation by Fe	PS 20 g/L; Fe(II) 0.78 g/L	Anthracene 1.87 mmol/kg	>99.9	[93]
PS activation by Fe	PS 18.37 g/L; $FeSO_4$ 4.25 g/L	Naphthalene 80 mg/kg	62	[94]
PS activation by pyrophosphatechelated Fe(II)	Fe(II) = 10 mM/kg; PS = 100 mM/kg	Ibuprofen 48.4 μM/kg	72.10	[82]
PS activation by diatomite-supported iron	PS:diatomite-supported Fe 1:1	Phenanthrene 200 mg/kg	98	[72]
PS activation by ZVI	ZVI 1.0 mmol/g; PS 4.0 mmol/g	*para*-chloronitrobenzene (p-CNB) 425.0 mg/kg	90.1	[73]
ZVI + PS two stage process	ZVI 0.8 mmol/g; PS 5.0 mmol/g	*para*-nitrochlorobenzene (pNCB) 2.87 mmol/kg	94.1	[74]
PS activation by mango peel-nZVI	nZVI 5 g/L; PS 0.5 M	PEH 6.41 (0.17) g/kg	>90	[78]
PS activation by ZVI flow system	PS 0.2 mmol/cm^3; Fe^{3+} 0.0558 mg/cm^3; nZV 0.0558 mg/cm^3	Anthracene 5.75 mg/kg; anthraquinone 97.7 mg/kg; phenantrene 137.7 mg/kg; pyrene 125.5 mg/kg; BaP 112.9 mg/kg	BaP 100	[76]
PS activation by nZVI	nZVI 0.03 g/g; T= 30 °C; n(SMX)/n(PDS) = 1:75	Sulfamethoxazole 20 mg/kg	96.1	[95]
PS activation by nZVI	PS 50,000 mg/L; nZVI 1 mM	Anthracene 97 mg/kg; pyrene 3 mg/kg; benzo(a)pyrene 102 mg/kg; phenanthrene 89 mg/kg	All 100 except phenantrene (80)	[96]
PS activation by ZVI in an in situ pilot-scale study	nZVI 3.5 g/L; PS 30 g/L;	PAHs ∼17 mg/kg	62.78–82.21	[75]
PS activated by Fe_3O_4 nanoparticles	PS 180 mM; Fe_3O_4 0.25 g/g	Polychlorobiphenyls (PCB 18-PCB 194) 450.2 ± 31.8 mg/kg	PCB 28:99; PCB 118:90	[55]
PS activation by Fe and citric acid	PS 2 mmol/L; $FeSO_4$ 0.2 mmol/g; citric acid 0.04 mmol/g;	16 PAHs 263.6 ± 73.3 and 385.2 ± 39.6 mg/kg	81.5–86.54	[28]
PS activated by magnetite	1% magnetite; 5.5% PS	PEH 4200 ± 124 mg/kg	95	[97]
PS activated by siderite	PS 400 mM; siderite 0.4 g; T = 60 °C; PS:Fe (II) 400:1	PEH 5000 mg/kg	41	[69]
PS activation by US	US 104 W; PS = 50 g/L; DHC 6500 mg/kg,	Diesel hydrocarbons 3250–16,250 mg/kg	23.29–92.56	[79]
PS activation by US	US 20 kHz; $(PS)_0$ = 5, 50 g/L	PFAS 0.045 mg/kg	62–71	[80]
PS activation by US/Fe	PS 1 M; US 200 W; Fe^0 = 0.28 g	PEH 19,850 mg/kg	82.23	[81]
PS activation by biochar	PS 8 mM; biochar 4 wt%	Bisphenol A 31.93 mg/kg	~99	[98]

Table 2. *Cont.*

Process Description	Process Parameters	Contaminant & Concentration	Removal Efficiency (%)	Reference
PMS activation by nZVI	0.3% PMS; 0.2% nZVI	TPH 6625 ± 115 mg/kg.	>96	[77]
PMS activation by ball-mill	PMS:soil 10:1; mass ratio of ball to material 30:1	Phenantrene 200 mg/kg	98	[83]
PS activation by alkaline	PS 100 g/L; NaOH:PS 4:1	PEH 5000 mg/kg	98	[65]
PS activation by alkaline	PS 400 mmol/L; NaOH 200 mol/L	Chlorinated organic compounds (COCs) 3060–9822 mg/kg	96–70	[64]
Cement-activated PS	PS 19.20 mmol/kg; Portland cement 10%	PAHs 214.4 mg/kg	57.3	[84]
Cement-activated PS	Portland cement:PS 2:1; PS 1.5%	BTEX 2,685–6836 mg/kg; 17 PAHs 54,001–30,372 mg/kg; naphthalene 23,140 ± 1673–13,445 ± 976 mg/kg	BTEX 91.1; PAHs 33.3; naphthalene 81.1	[85]

5. Coupled Processes

The comprehensive soil remediation by a single treatment technology can be complicated when pollutants of different natures are present in this heterogeneous matrix. In addition, some limitations of AOPs can be overcome by their combination. Thus, in recent years there has been a tendency towards the use of coupled processes (Table 3). Accordingly, different processes were coupled with AOPs such as biological methods, soil washing, electrochemical techniques, and also a combination of different AOPs. In the following the most notable processes are described.

Table 3. Recently published articles on soil decontamination by coupled processes.

Process Description	Contaminant & Concentration	Removal Efficiency (%)	Reference
EK–Fenton	Organochlorine pesticides: hexachloro-cyclohexane soprocide (HCH) and dichloro-diphenyl-trichloroethane (DDT) 7.79–14,025.2 mg/kg	71.5–82.6	[99]
EK–Fenton	Petroleum 10,000 mg/kg	89	[100]
EK–Fenton	Rhodamine B 0.16 g dye/kg; PEH 80.4 mg/kg	Dye:54.4; Petroleum:58.2	[101]
EK–Fenton pilot level	Gasoline 1126 mg/kg	80	[102]
EK–PS	Decabromodiphenyl ether (BDE-209) 50 mg/kg	BDE:209 85.6	[103]
EK–PS	Pyrene 200 mg/kg	100	[104]
EK–PS	PAHs 4.88 mg/kg	93.1	[105]
EK–Fenton–surfactants	THP Spanish soil 80,356.42 mg/kg; THP Romanian soil 45,557.10 mg/kg	Spanish soil 25.7–81.8; Romanian soil 15.1–71.6	[106]
EK–PS–surfactants	Organochlorine pesticides (OCPs) 462.08 to 20,335.09 ng/g	88.05	[107]
EK–Fenton–phytoremediation	PEH 8956 mg/kg	>85	[108]
Fenton–$KMnO_4$	16 PAHs 1210 and 1136 µg/g	80 and 84	[45]
Fenton–KMnO4	27 PAH 3090 ± 104 mg/kg	71	[49]

Table 3. *Cont.*

Process Description	Contaminant & Concentration	Removal Efficiency (%)	Reference
Fenton–HPCD surfactant	16 PAH listed by USEPA 1090 mg/kg	99	[109]
Fenton–bioremediation	16 PAHs 263.6 ± 73.3 and 385.2 ± 39.6 mg/kg	5–6 rings 78–90; 2–4 rings 52–85	[28]
Fenton–bioremediation	1,4-dioxane	100	[110]
Fenton–bioremediation	Lubricants 10,000 mg/kg	99.2	[70]
Fenton–bioaugmentation serial foam	PEH 7470 mg/kg	92	[111]
Fenton–anoxic–biodegradation	16 PAHs 350.07 mg/kg	33.2–95.9	[112]
Fenton–pre-oxidation–bioremediation	PEH 12,178 ± 390 mg/kg	42 ± 1.43	[113]
Fenton–pre-oxidation–bioremediation	Macro crude oil (C_{19}–C_{29} and C_{17}–C_{29}) 8853 mg/kg-S1 11.719 mg/kg-S2	57.1 and 64.4	[114]
Fenton–pre-oxidation–bioremediation	Crude oil 23,440 ± 390 mg/kg	53	[115]
Fenton-like treatment–bioremediation	Diesel 700–2600 mg/kg	75	[116]
Fenton-like reaction combined with *Phanerochaete chrysosporium*	Polybrominated diphenyl ethers 18.70 ± 0.08 ng/g	55.5 ± 6.0–72.6 ± 3.6	[117]
Tween 80 surfactant–electro–Fenton	Diesel 50 g/kg	87.2	[118]
Sono–photo–Fenton	TPH 15 g/kg	99	[119]
PMS in presence of Fe (V)	PCB > 1000 mg/kg	40	[120]
PS–H_2O_2	o-NCB 400 mg/kg	~80	[56]
PS-SDS surfactant	PAHs 100 mg/kg	34–75	[121]
PS–bioremediation	Phenanthrene 1200 ± 200 mg/kg; pyrene 1200 ± 200 mg/kg	90	[122]
PS–bioremediation	PAHs 214 ± 21 mg/kg	66	[123]
PS–bioremediation	PEH 7996.86 ± 1173.40 mg/kg	62.61 ± 1.23	[124]
PS–bioremediation	PBDEs 53.8 ± 0.5 mg/kg	94.60	[125]
PS–anoxic–bioremediation	16 PAHs-562.81 ± 10.29 and sterilized soil 481.35 ± 6.63	36.37–94.91	[88]
PS–pre-oxidation–bioremediation	BaP 0.7 mg/kg	98.7	[126]
PS–bioaugmentation serial foam spraying technique	PEH 100 mL/kg	80	[118]

5.1. Electrokinetic Treatment–AOP

Electrokinetic (EK) treatment is a well-studied technology applied in the remediation of contaminated soils for the removal of contaminants (Figure 5). The process is based on the use of a low-voltage gradient (1 DCV/cm), utilizing two electrodes (anode and cathode) directly inserted into the soil [127]. The effectiveness of this method is highly dependent on the capacity of the contaminants to move under the electric field's action, and the migration and removal of the contaminants under the electric field are made through electromigration, electroosmosis, and electrophoresis processes [99,128,129]. When the electric field is applied to the soil, various events take place, such as electrochemical reactions, the production of flow processes and a pH gradient, mobility, sorption, desorption, precipitation of pollutants, and complexation. The low solubility of most organic pollutants reduces the efficiency of the electrokinetic treatment's ability to enhance the removal efficiency and assure an optimal performance; it is necessary to combine this technology with other techniques [106]. The Fenton process, one of the frequently used AOPs is often combined with electrokinetic methods, giving high removal rates of organic pollution. The electrokinetic Fenton (EK–Fenton) process can contribute to the transport of the H_2O_2 through the soil and, in the presence of transition metal minerals, mainly iron minerals, in the soil, decompose the H_2O_2 delivering ·OH, which is capable of oxidizing contaminants [101]. By using this technology, there are studies such as that developed by Ochoa et al. [102], who achieved encouraging results at the pilot level.

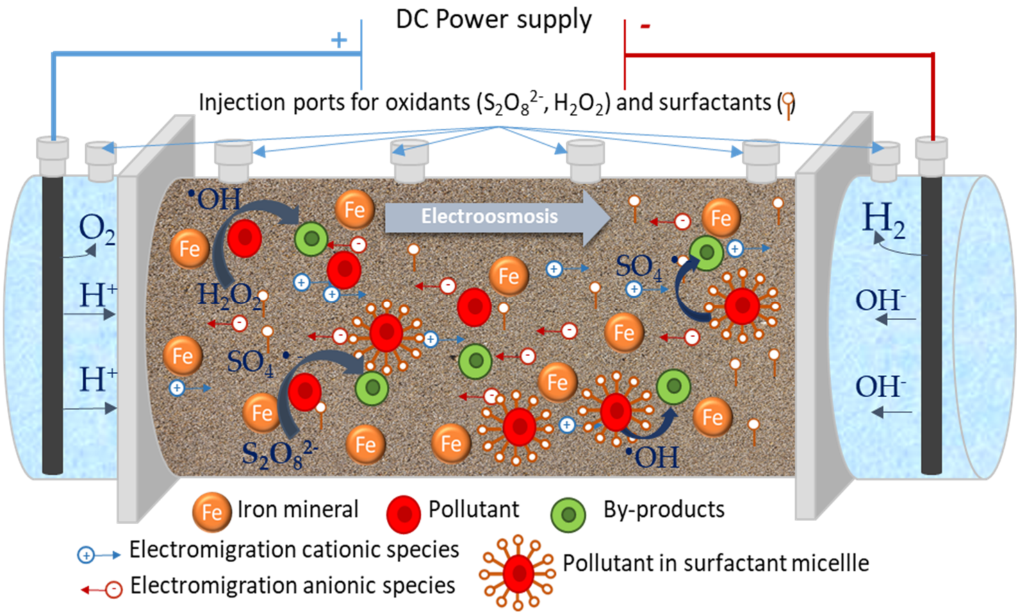

Figure 5. Schematic of the EK–Fenton process.

Ni et al. [99] demonstrated the compatibility of electrokinetic remediation with the Fenton process for a high degradation to soil historically polluted by organochlorine pesticides. The individual EK method showed a reduced removal efficiency, while the enhanced EK–Fenton process with the addition of the oxidant and electrolyte successfully removed the pesticides from the actual historically polluted soil. The high efficiency of this process was also shown without the addition of any supporting electrolyte. Furthermore, a high efficiency was found in EK–persulfate experiments. Thus, Chen et al. [103] applied to the remediation of soil contaminated with decabromodiphenyl ether the electrokinetics–persulfate process (EK–PS), where the EK process highly increased the transport of the injected PS and supported the in situ chemical oxidation of the pollutant. EK–Fenton has been successfully combined with the Tween 80 and Triton X-100 surfactants in the treatment of soils historically polluted with PEH, giving a high removal efficiency [106]. Similarly, a surfactant combined with persulfate and EK remediation was tested by Suanon et al. [107,130] on the removal of organochlorine pesticides from soil. The utilization of Triton X-100 as co-solvent led to a significantly improved contaminant degradation, demonstrating that surfactant-enhanced advanced oxidation is a qualitative treatment for organochlorine contaminated soil. Moreover, this synergic combination was tested by Abtahi et al. [104] on the remediation of PAH-spiked soil, where they obtained a complete degradation of pyrene using Tween 80 of 20 mL/kg concentration and a voltage gradient of 1 V/cm. Other removal technologies can also be coupled with AOP–EK that enhanced the efficacy of the electrokinetic and Fenton process by phytoremediation [108]. Results showed that the use of a polarity reversal benefits the germination and growth of plants and produces a high PEH degradation after 20 days.

5.2. AOP–AOPs

The combination of several AOPs at the same time has been also achieved encouraging results to enhance the limitations of using a single process. Therefore, a combination of ultraviolet and ultrasonic irradiation and nano zero-valent iron (UV/US/nZVI) improved by Fenton's reagent was studied by Gharaee et al. [119]. They demonstrated that the combination of these treatment processes led to a 15% synergistic effect. In the paper, the rate constant of the combined process was 52×10^{-3} min^{-1}, which was greater than the sum of the rate constants of the individual methods (21×10^{-3}/min and 23×10^{-3}/min).

A dual oxidant system has been used by Liu et al. [56], using hydrogen peroxide and PS, where the reactivity of H_2O_2 in the decrease of compounds is combined with the improved stability of persulfate; moreover, the iron species present in the soil contributing to the degradation process proved to be promising in the removal of ortho-nitrochlorobenzene (o-NCB) in soil.

Permanganate is the oxidant that is used most often, because it easily decomposes into permanganate ions and is cheap and secure compared to H_2O_2. However, an excess generation of MnO_2 could reduce the soil's permeability. It may affect the soil quality, and it is also not effective in the removal of chlorinated alkanes and most of the aromatic compounds. To overcome these negative factors, coupling permanganate with AOPs for a higher removal rate was proposed in some studies [45]. It was reported by Bendouz et al. [49] that the degradation of 27 PAHs using the Fenton reaction followed by $KMnO_4$ oxidation (or $KMnO_4$ followed by Fenton) was superior (at around 71%) to that in the application of the Fenton or $KMnO_4$ treatment alone (43% for each process).

In recent times, Ferrate (Fe(VI)) has emerged as an efficient oxidant to remediate contaminated soils. Accordingly, Monfort et al. [120] integrated the use of Fe(VI) and conventional oxidants and found that the degradation efficiency was increased by the combination of Fe(VI)/PMS, obtaining a 40% removal of PCBs from historically contaminated soil.

5.3. Surfactants–AOPs

During the soil washing remediation, the addition of surfactants in order to enhance the removal process used to be mandatory [131]. These compounds are complex amphiphilic molecules formed from a hydrophobic (nonpolar) hydrocarbon "tail" and a hydrophilic (polar) "head" group [131], and they can be divided into four classes: anionic, cationic, amphoteric, and nonionic (e.g., Tween 80 and Triton X-100). The surfactant treatment of the generated effluent is essential to achieve a total pollutant removal; the presence of these compounds in the effluent can be an obstacle to its appropriate management, because the removal process is even harder, since the contaminant is trapped in the micelle core of the surfactants. Thus, the selection of a suitable process requires careful consideration. In these cases, AOPs have been found to be a promising option leading to a high effectiveness in eliminating the refractory target contaminants [121]. Thus, Liu et al. [118] demonstrated that the combination of soil washing using Tween 80 and electrooxidation or electro-Fenton could achieve the efficient remediation of soil heavily contaminated with diesel. In addition, as demonstrated in previous sections, the surfactants (during Fenton oxidation) can result from the partial oxidation of hydrocarbons and/or the native soil organic matter and could enhance the degradation of the contaminants [46].

In other research, surfactants were used to enhance the AOP, and it was observed that the compatibility between surfactants and oxidants is affected by some factors. The matrix composition has a great influence on the process efficiency, especially the natures of the surfactant agents, soil organic matter, inorganic ions, and/or the organic pollutants' composition. In Mousset et al.'s [132] study (Fenton process and surfactants) a slower PAHs removal was observed in the case of Tween 80 solutions than with 2-hydroxypropyl-β-cyclodextrin (HPCD). In addition, in a previous work by the same authors [109], they observed that a high yield was achieved after 150 min with HPCD (10 g/L) without the supplement of organic matter. This was because of the high reactivity of $^{\bullet}$OH with the organic matter, which led to a competitive reaction between the organic molecules and $^{\bullet}$OH in the case of Tween 80 solutions. Thus, as well as leading to a slower PAHs removal, the degradation rates decreased as the number of the PAHs' rings increased. Nevertheless, overall, Tween 80 showed a better performance than HPCD regarding some parameters, such as extraction efficiency, costs, and global soil microbial activity [109,132,133].

5.4. AOPs–Biological Methods

Coupling AOPs with a biological treatment can allow the removal of organic pollutants from the soil in a cost-effective way. Biological remediation is an environmentally friendly method that applies microorganisms to degrade organic pollutants. AOPs utilize a few chemicals but produce secondary contaminants, and biological methods rely on enzymatic reactions (laccases, peroxidases, tyrosinases, etc.) that are safe for the environment. Harmful by-products produced through AOP remediation can be eliminated from the soil with the help of microorganisms [124]. The results of many studies showed that AOPs are an efficient treatment when combined with bioremediation for high degradation rates of organic contaminants. For instance, Bajagain et al. [111] achieved a 92% PEH removal utilizing hydrogen peroxide (without amendment by supplementary iron) followed by biodegradation. Meanwhile, biodegradation alone degraded the contaminants by only 25%. Similarly, Xu et al. [114] applied Fenton and bioremediation in the degradation of macro crude oil, obtaining significant results. The bioremediation efficiency of macro crude oil improved and the bioremediation period was cut by two to three times. Moreover, the genera *Sedimentibacter*, *Caenispirillum*, and *Brevundimonas* became the dominant bacteria after a matching Fenton pre-oxidation. The macro crude oil was the primary carbon source for the increased rate of the indigenous hydrocarbon degraded by the utilization of matching Fenton pre-oxidation, so it further enhanced the biodegradation of macro crude oil. In another study, a great removal efficiency (99.2%) in the removal of lubricant was obtained by a combination of biopile for 56 d followed by a modified Fenton process [70].

In another study conducted by Liao et al. [28], the effects of different oxidants on the removal of PAHs from real polluted soils were tested. Whereas Fenton oxidation provides nutrients to enhance the bioremediation, and had a mild effect on the indigenous microbial diversity, potassium permanganate significantly affected microbial diversity and delayed the population's recuperation. In addition, in the study performed by Xu et al. [113], Fenton pre-oxidation at a high H_2O_2 concentration inhibited the indigenous bacteria that degrade C_{15}–C_{30} alkanes, but in the final result at a low H_2O_2 concentration, indigenous bacterial populations were not harmed and the process enhanced the nutrient mobilization and further bioremediation. Some studies have found this general decreasing/increasing biodiversity trend with different oxidation reactions. Miao et al. [110] applied oxidation with H_2O_2 followed by bioaugmentation to 1,4-dioxine degradation and they observed that the microbial population, biodiversity, and richness were inhibited after their exposure to peroxide but recovered during the biodegradation process. They obtained a complete removal of 1,4-dioxane from contaminated soil, even though the bioremediation process was generally inhibited by VOCs, proving the effectiveness of treatment trains.

In the case of persulfate coupled with bioremediation, results showed that the utilization of low doses of sodium PS in the presence of low soil-moisture levels was the most efficient variant for phenanthrene degradation that can be compatible with bioremediation [33]. PS facilitated the occurrence of more pollutant-degrading microorganisms. The effects of pre-oxidation followed by the biodegradation of BaP were assessed, and the degradation efficiency of BaP by PS was 81.8% after 7 days of pre-oxidation. The pre-oxidation improved the activity of soil microorganisms and enhanced the biodegradation of BaP in soils within 7–60 days [114]. Likewise, Bajagain et al. [134] applied the persulfate–bioaugmentation serial foam spraying technique on diesel-oil-contaminated soil, and nearly 80% of the pollutant was degraded by this combined method, while 52%

was removed by bioaugmentation alone. They reported that persulfate-foam spraying is superior to peroxide for diesel degradation even at a reduced concentration (0.005 N). This fact is due to persulfate-foam is more permeable, persistent, and does not influence the soil pH in the subsurface.

Furthermore, the addition of biochar for PS activation was used in combination with bioremediation on the treatment of soil contaminated with PEH. Zhang et al. [124] applied the degradation treatment over 60 days in two stages, using remediation by PS with biochar supported nano iron (BC-nZVI) and biodegradation. The pollutants were degraded efficiently during the chemical oxidation period and afterwards were converted to be more disposable in the next step. The biochar addition enhanced the optimal conditions for bioremediation, obtaining favourable results during the whole remediation period.

6. Conclusions and Prospects for the Future

From the review performed in the present review article, it can be established that AOPs have been demonstrated to be effective in removing a wide range of contaminants from soil, such as pesticides, PEH, PHAs, and recalcitrant organics. Among the different AOPs, the scientific community is focusing its attention on the process based on the Fenton reaction and the sulfate radical generation. This last one is the most studied in recent years as a result of the fact that no pH limitations and minor activation requirements are necessary in comparison to the Fenton process. However, for both treatments, the main factor that influences the removal process is the delivery of reagents to carry out an efficient removal process. In addition, at present, scarce studies in flow system have been reported and no in situ treatments have been accomplished. To increase the process efficiency and reduce the associated costs, the coupled methods have been identified as viable techniques for the treatment of different polluted soils. The reviewed research determined that the use of coupled processes tends to decrease the operational costs, especially the coupling with biological remediation. However, the next stages to achieve the field scale application should be focused on the identification of intermediate by-products, the impact on soils' physical-chemical and biological properties, and the determination of scale-up parameters.

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