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Review

Electrochemical Advanced Oxidation Processes Using Diamond Technology: A Critical Review

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Abstract: Re-evaluation of conventional wastewater treatment processes is of paramount importance to improve the overall quality of our aquatic environment. Electrochemical Advanced Oxidation Processes (EAOPs) are the most promising alternative methods with application in wastewater treatment facilities since in situ electrogenerated oxidant agents degrade and mineralize a wide range of water pollutants. Boron-doped diamond (BDD) technology has proven its excellency in the anodic oxidation (AO) of different pollutants. In this work, we describe the use of a systematic literature review (SLR) methodology and a bibliometric analysis tool for the assessment of a representative sample of work (hundreds of publications) concerning the synergism between AO using BDD technology and other oxidation methods. One section of the discussion relates to different techniques used to enhance the AO performance of BDD technology, namely persulfate radicals or ozone and photoelectrocatalysis, whereas the second one considers Fenton-based reactions. A standard synergism effect occurs between AO using BDD technology and the add-ons or the Fenton-based methods, resulting in the enhancement of the degradation and mineralization efficiencies. The future of EAOPs using BDD technology must include renewable energy sources to self-sustain the overall process, and further research on the subject is mandatory to enable the effective acceptance and application of such processes in wastewater remediation facilities.

Keywords: EAOP; diamond; Fenton; electrooxidation; photocatalysis; ozonation; synergism; water; treatment; remediation

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1. Introduction

The water crisis has triggered the urgent need to develop economical and efficient methods to remove contaminants without the common drawbacks associated with the widely applied conventional bioremediation techniques [1], such as residual sludge production, phase transfer of pollutants, generation of toxic gases, and demand for large territorial areas [2]. Although these methods remove many of the dissolved organic carbons, they are insufficient due to the presence of persistent organic pollutants (POPs) in wastewaters, threatening the environment and human health [3,4]. Significant attempts are being developed to reduce the impact caused by water pollution and to ensure safe wastewater disposal onto the environment. Numerous alternative methods have been proposed as more efficient and sustainable water treatment methods. They include enhanced photocatalytic degradation [5], chemical coagulation [6], electrocoagulation [7], membrane bioreactor [8], membrane filtration [9], electron beam [10], wet air oxidation [11], electroreduction [12], adsorption [13], electroadsorption [14], electrochemical hydrodechlorination [15], electrokinetic separation [16], ozonation [17], and the so-called Electrochemical Advanced Oxidation Processes, EAOPs (Figure 1) [18].

The advances in electrochemical technology led to the development of EAOPs [19,20], and are considered the most promising and innovative alternative water treatment technologies [2,21,22]. The basis of EAOPs is the electrochemical generation of highly reactive oxidizing species, such as hydroxyl radicals, capable of mineralizing the target pollutants [23,24]. These techniques aim to mineralize contaminants to CO_2 and water, or, at least, convert them into harmless, easily degradable products whilst avoiding the formation of new toxic species [20]. Since electrochemical methods are based on the transfer of electrons, EAOPs require no or low amounts of additional chemical reagents or catalysts during the process [19]. Thus, EAOPs are particularly interesting because they are environmentally compatible, versatile, and a highly effective means to eliminate a large variety of pollutants from wastewater [19,25].

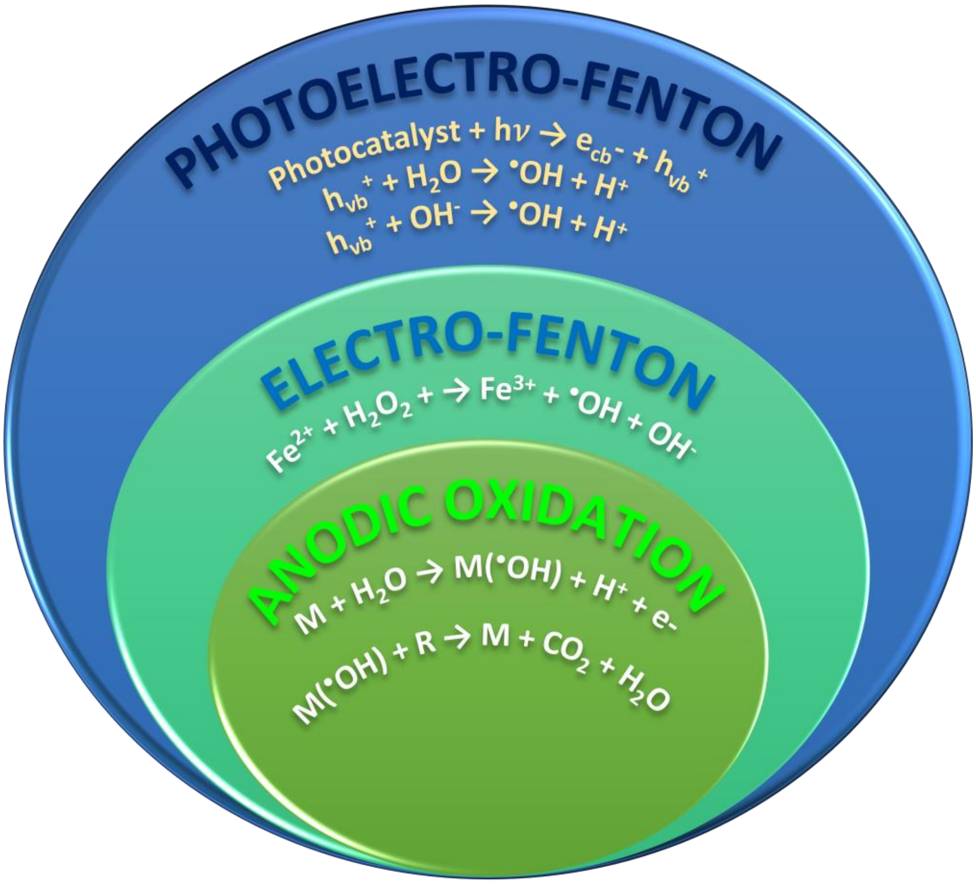

Figure 1. Diagram illustrating the main chemical reactions in EAOPs.

The materials used as anodes in EAOPs are crucial for achieving efficient water treatment, since they are responsible for the generation of the oxidant species [26]. These electrodes must have a high oxygen evolution overpotential (OEP), such as lead dioxide (PbO_2), tin dioxide (SnO_2), sub-stoichiometric TiO_2, boron-doped diamond (BDD), and some composites such as Sb-SnO_2, Co-PbO_2, TiO_2-nanotubes with PbO_2, Ti/Sb-SnO_2, and La-Y-PbO_2 [24,27]. It has also been established that the higher the OEP, the higher the oxidation output of the anode [28]. PbO_2, SnO_2, and sub-stoichiometric TiO_2 present high OEP and are generally cheap and simple to manufacture, though with poor electrochemical stability [29]. Graphite and glassy carbon electrodes also have relatively high OEPs but are highly vulnerable to surface oxidation and fouling [30]. Among the many materials tested as potential anodes, BDD has proven its excellent ability in the electrochemical oxidation of pollutants showing the highest overpotential for water decomposition, the widest known electrochemical potential window, and excellent efficiency in producing hydroxyl radicals [31,32]. Furthermore, BDD has a high electrochemical stability and resistance to corrosion, even in chemically aggressive media, presenting inert surfaces with very low adsorption, and works under the whole pH range [33–35].

Diamond's natural low conductivity and large bandgap have limited its use in electrochemical applications [36]. However, with the development of Chemical Vapour Deposition (CVD) methods, the use of diamond in such applications became possible. CVD growth of diamond requires the generation of carbon radicals and a high concentration of dissociated hydrogen [37]. This is achieved by activating a gas mixture of a carbon-containing source gas, such as methane, mixed with molecular hydrogen. The dissociation of molecular hydrogen to react with the carbon-containing gas source and produce reactive carbon-containing radicals, the growth precursors, is typically done using various methods such as hot-filament CVD, RF-plasma CVD, microwave plasma CVD, DC plasma, DC arc-jet, or even by using an oxy-acetylene flame [38]. The CVD growth parameters such as deposition time, substrate temperature, pressure, bias voltage, substrate nature, and gas composition control the final properties of the diamond film [39]. Introducing boron atoms into the CVD reactor's environment has unlocked the possibility of combining high electrical conductivity with the unique diamond properties [40]. Due to its relatively small charge carrier activation energy (0.37 eV) and small atomic radius, boron can effectively occupy the same position as displaced carbon atoms [41]. Thus, a high enough boron concentration can be incorporated in the diamond lattice to achieve metal-like conductivity [37]. For this reason, although other impurity atoms have been applied in diamond doping, such as nitrogen [42] and phosphorus [43], BDD will be the focus of this critical review since it is more well-suited for electrochemical studies such as electrochemical oxidation, electroanalysis, electrosynthesis, and energy conversion [41,44].

The individual application of BDD technology as an EAOP for wastewater treatment is widely published, proving to be one with the highest degradation efficiency, including the elimination of several sources of water contamination, such as refractory organics [45], dyes [46], pharmaceuticals [47], pesticides [48], by-products of industrial processes [49], domestic sewage [50], and landfill leachate [51], amongst many others. Consequently, the reported literature concerning BDD technology is quite extensive, but, to the best of our knowledge, a critical review gathering information on overcoming the few drawbacks of BDD technology by combining it with other EAOPs would be very useful. Conventional wastewater facilities employ a combination of different treatment techniques and perhaps the "apparent" high cost of BDD technology is the main reason why it has not seriously been considered or adopted. It is also intended, with this review, to clearly demonstrate to the industrial business that the effective and sustainable application of BDD technology will shortly add up value to all of its benefits.

Thus, a systematic literature review (SLR) and bibliometric analysis are conducted in this critical review to select publications and extract information on the trends and future directions of EAOPs using BDD electrodes for wastewater treatment. The anodic oxidation (AO) EAOP, also referred to as electrochemical oxidation (EO), is the most straightforward and applied EAOP using BDD technology. This is basically carried out by applying an electric current directly to a pair of electrodes (anode and cathode) immersed in the aqueous solution to be treated, and then promoting the direct oxidation of pollutants through the generation of reactive oxygen species (mainly hydroxyl radicals) generated during water splitting [22]. The main drawback of this technology is the fact that these short-lifetime oxidant species are limited to the surface reaction layer of the BDD anode [22]. It is our main goal to identify and discuss EAOPs that employ diamond technology but go beyond the apparently simple AO process. We will discuss the advantages, disadvantages, and limitations of the methods identified through the SLR, as well as the role and performance of diamond electrodes. We will also identify the synergism between EAOPs and BDD technology as a possible solution to the main disadvantage of AO based on BDD electrodes.

2. Electrochemical Advanced Oxidation Processes Using Diamond Technology: A Bibliometric Analysis

The number and diversity of scientific publications in the last few decades have exponentially increased, which leads to the constant need for consistent and updated review articles [52]. Systematic Literature Review (SLR) is a method of scientific research used when the objects of analysis are significant literature sources, enabling the authors to identify, select, and critically evaluate the research results to answer a clearly formulated question [53]. SLR is a highly standardized and structured approach that follows a well-defined protocol in which the criteria are clearly stated before the analysis is performed, leading to a comprehensive, unbiased, transparent, and accountable study [54].

In this critical review, we applied the SLR method to identify what is known, what is unknown, the limitations, the trends, and the future directions of existing research on Electrochemical Advanced Oxidation Processes (EAOPs) using BDD technology. A previous rough analysis to determine the most applied EAOP methods using diamond electrodes was performed, followed by advanced Boolean search queries in the Scopus database for each identified method (anodic oxidation- AO or EO, anodic oxidation with electrogenerated H_2O_2, electro-Fenton- EF, photoelectro-Fenton- PEF, and solar photoelectro-Fenton- SPEF), adopting the main keywords commonly used for each of the processes. Table 1 provides details on the advanced search queries by EAOP type.

Table 1. Details of the advanced Boolean search queries performed in the Scopus database regarding the most applied EAOP type using diamond technology (source: the authors).

EAOP	Boolean Advanced Search Query	Scopus Results (September 2022)
Anodic oxidation/electrochemical oxidation (AO/EO)	(TITLE-ABS-KEY (bdd OR “doped diamond”) AND TITLE-ABS-KEY (“anodic oxidation” OR “electrochemical oxidation”)) AND (LIMIT-TO (LANGUAGE, “English”))	1410
Anodic oxidation with electrogenerated H_2O_2 (AO-H_2O_2)	(TITLE-ABS-KEY (bdd OR “doped diamond”) AND TITLE-ABS-KEY (“anodic oxidation” OR “electrochemical oxidation”) AND TITLE-ABS-KEY (“electrogeneration of H_2O_2” OR “electrogenerated H_2O_2” OR “AO-H_2O_2” OR “EO-H_2O_2” OR “AO/H_2O_2” OR “EO/H_2O_2”)) AND (LIMIT-TO (LANGUAGE, “English”))	67
All Fenton-based processes	(TITLE-ABS-KEY (bdd OR “doped diamond”) AND TITLE-ABS-KEY (*fenton)) AND (LIMIT-TO (LANGUAGE, “English”))	364
Electro-Fenton (EF)	(TITLE-ABS-KEY (bdd OR “doped diamond”) AND TITLE-ABS-KEY (fenton) AND NOT TITLE-ABS-KEY (“photo-Fenton” OR “photoelectro-Fenton” OR “PEF” OR “Solar photoelectro-Fenton” OR “SPEF” OR “photoassisted” OR “photo-assisted” OR “photo enhanced” OR “photo-enhanced” OR “sunlight” OR “solar”)) AND (LIMIT-TO (LANGUAGE, “English”))	209
Photoelectro-Fenton (PEF)	(TITLE-ABS-KEY (bdd OR “doped diamond”) AND TITLE-ABS-KEY (fenton) AND TITLE-ABS-KEY (“photo-Fenton” OR “photoelectro-Fenton” OR “PEF” OR “photoassisted” OR “photo-assisted” OR “photo enhanced” OR “photo-enhanced”)) AND (LIMIT-TO (LANGUAGE, “English”))	153
Solar photoelectro-Fenton (SPEF)	(TITLE-ABS-KEY (bdd OR “doped diamond”) AND TITLE-ABS-KEY (fenton) AND TITLE-ABS-KEY (“Solar photoelectro-Fenton” OR “SPEF” OR “sunlight” OR “solar”)) AND (LIMIT-TO (LANGUAGE, “English”))	55

The bibliometric searches were carried out through search strings connecting the topics by the title, abstract, and keyword fields of articles published until September 2022. The full period of research results and all types of documents were considered, but searches were limited to articles published in the English language. The combination of all search queries performed in the first sampling results in a total of 1590 publications (Figure 2).

The focus of this work was restricted to articles that do not consider the anodic oxidation (AO) process using BDD technology as the common keyword. Therefore, our study was based on the number of publications excluded by the AO cluster (light-blue line in Figure 2), i.e., contemplating 181 out of the total 1590 found publications. Therefore, the selection of articles for the SLR following a first line of thought would be based on these

181 publications. As one of the principles behind SLR methods is to make the selection of documents easily reproducible by anyone, and the method used in this first search requires running many search queries and combining them to arrive at the final selection, we decided to test a second approach to simplify the selection method with only one advanced search string, detailed in Table 2.

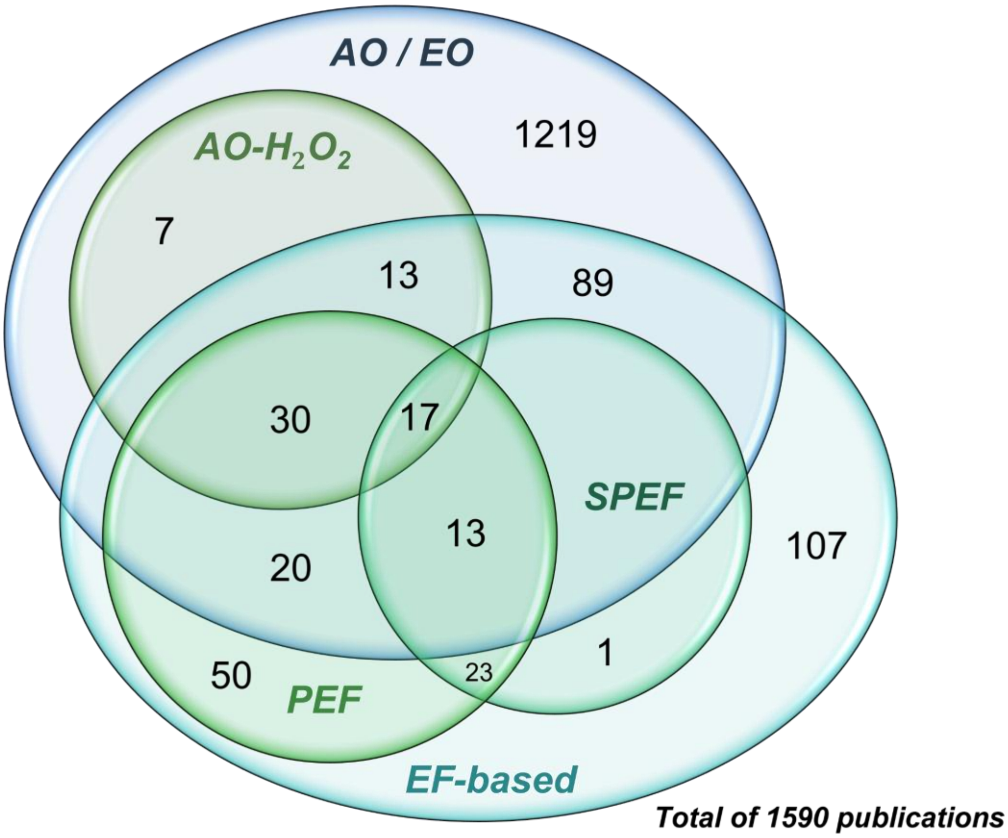

Figure 2. Venn diagram representing EAOP combinations from the first SLR test sample (source: the authors).

Table 2. Details of the Boolean advanced search query: theme, strings, search query, and results, based on the Scopus database (source: the authors).

Theme	Boolean Advanced Search	Scopus Results
Electrochemical Advanced Oxidation Processes using diamond technology	(TITLE-ABS-KEY (diamond AND eaop*) OR TITLE-ABS-KEY (diamond AND aop*)) AND (LIMIT-TO (LANGUAGE, "English"))	164 (September 2022)

After performing a previous criteria sampling, the keywords "diamond", "EAOP*", and "AOP*" (abbreviation for Advanced Oxidation Process) were selected for composing this simpler search query. As previously, the search string also connected the topics by the title, abstract, and keyword fields of articles published until September 2022, including the full period of research and all types of documents published in the English language. A total of 164 publications met the set criteria.

By combining the two selection criteria (Table 3), we identified that 154 publications are common to both selections, which represent 94% of the simpler search string results.

Table 3. Details of the Boolean advanced search query combining both tested sample selection methods (source: the authors).

Boolean Advanced Search	Scopus Results
(((TITLE-ABS-KEY (bdd OR “doped diamond”) AND TITLE-ABS-KEY (“anodic oxidation” OR “electrochemical oxidation”))) AND NOT (((TITLE-ABS-KEY (bdd OR “doped diamond”) AND TITLE-ABS-KEY (“anodic oxidation” OR “electrochemical oxidation”) AND NOT TITLE-ABS-KEY (“electrogeneration of H_2O_2” OR “electrogenerated H_2O_2” OR “AO-H_2O_2” OR “EO-H_2O_2” OR “AO/H_2O_2” OR “EO/H_2O_2” OR “Fenton” OR “electro-Fenton” OR “Fered-Fenton” OR “EF” OR “photoelectro-Fenton” OR “PEF” OR “Solar photoelectro-Fenton” OR “SPEF” OR “photoassisted” OR “photo-assisted” OR “photo enhanced” OR “photo-enhanced”))) AND NOT (((TITLE-ABS-KEY (bdd OR “doped diamond”) AND TITLE-ABS-KEY (“anodic oxidation” OR “electrochemical oxidation”) AND TITLE-ABS-KEY (“electrogeneration of H_2O_2” OR “electrogenerated H_2O_2” OR “AO-H_2O_2” OR “EO-H_2O_2” OR “AOH_2O_2” OR “EO/H_2O_2”))) OR ((TITLE-ABS-KEY (bdd OR “doped diamond”) AND TITLE-ABS-KEY (fenton) AND NOT TITLE-ABS-KEY (“photo-Fenton” OR “photoelectro-Fenton” OR “PEF” OR “Solar photoelectro-Fenton” OR “SPEF” OR “photoassisted” OR “photo-assisted” OR “photo enhanced” OR “photo-enhanced”))) OR ((TITLE-ABS-KEY (bdd OR “doped diamond”) AND TITLE-ABS-KEY (fenton) AND TITLE-ABS-KEY (“photo-Fenton” OR “photoelectro-Fenton” OR “PEF” OR “photoassisted” OR “photo-assisted” OR “photo enhanced” OR “photo-enhanced”))) OR ((TITLE-ABS-KEY (bdd OR “doped diamond”) AND TITLE-ABS-KEY (fenton) AND TITLE-ABS-KEY (“Solar photoelectro-Fenton” OR “SPEF” OR “sunlight” OR “solar”)))))) AND NOT ((TITLE-ABS-KEY (diamond AND eaop*)) OR (TITLE-ABS-KEY (diamond AND aop*))) AND (LIMIT-TO (LANGUAGE, “English”))	154 (September 2022)

Therefore, the second approach was adopted to select the articles for this review since it is unbiased and can be easily replicated, whilst the first method requires a combination of several queries limited to the methods previously identified (biased). Furthermore, including EAOP and AOP in the search string makes the selection more representative, including other EAOPs that are not so frequently applied with diamond technology and were not evaluated in the first selection, such as ozonation, photocatalysis, and peroxone process, among others. These alternative EAOPs explain the extra 10 results identified in the second selection in relation to the total 154 documents common to both approaches. From this point onwards, the entire discussion of the bibliometric analysis is based on the 164 articles identified in the second sample (Table 2). These also form the basis of discussion for the specific topics for each type of EAOP discussed in subsequent sections. When necessary, publications relevant to each type of EAOP that have not been selected by the SLR are added to deepen the discussion on each section.

Over the last twenty years, the topic of EAOPs using diamond technology has gained increased interest, especially after the year 2009, reaching a peak in the number of publications ten years later. Figure 3 presents the temporal analysis of the obtained SLR results.

Although the number of published articles has decreased over the last two years, it remains scientifically relevant, with 10 publications in the year 2022. The subject areas of Environmental Science, Chemistry, and Chemical Engineering appear as the areas of interest most related to the theme. It is also important to highlight the areas of Engineering and Materials Engineering which are directly related to the manufacture and optimization of diamond electrodes used in EAOPs. Figure 4 shows the map distribution of the related subject areas.

In these 20 years of research on the subject, most communications were presented in the form of original papers, representing 88% of the SLR results. Six reviews published on the topic were identified, concentrated in the years 2014, 2019, and 2020. In addition, four book chapters were published, and the subject was presented at, at least, nine conferences. Figure 5 shows the distribution of the document type found in the SLR results.

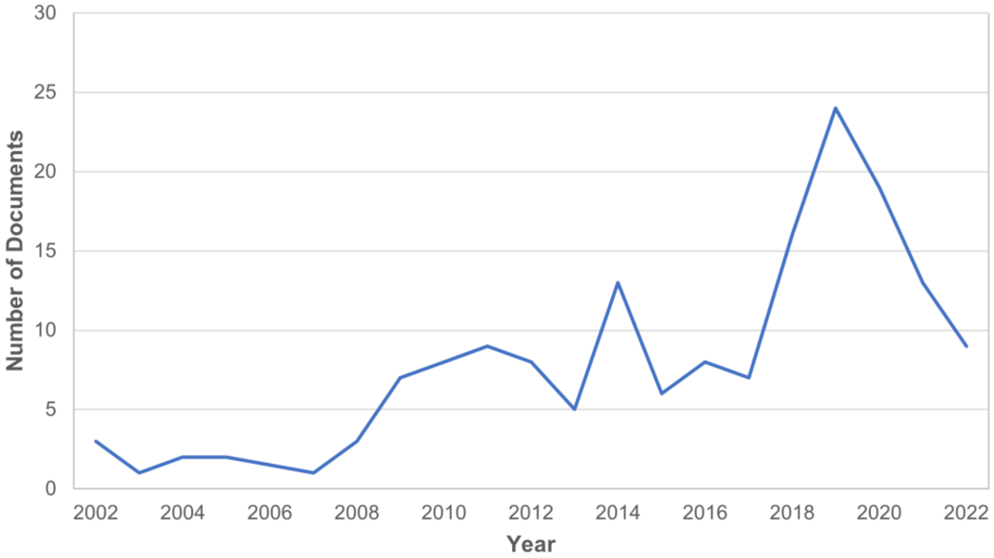

Figure 3. Number of publications per year on EAOPs using diamond technology for water treatment, based on the Scopus database (source: the authors).

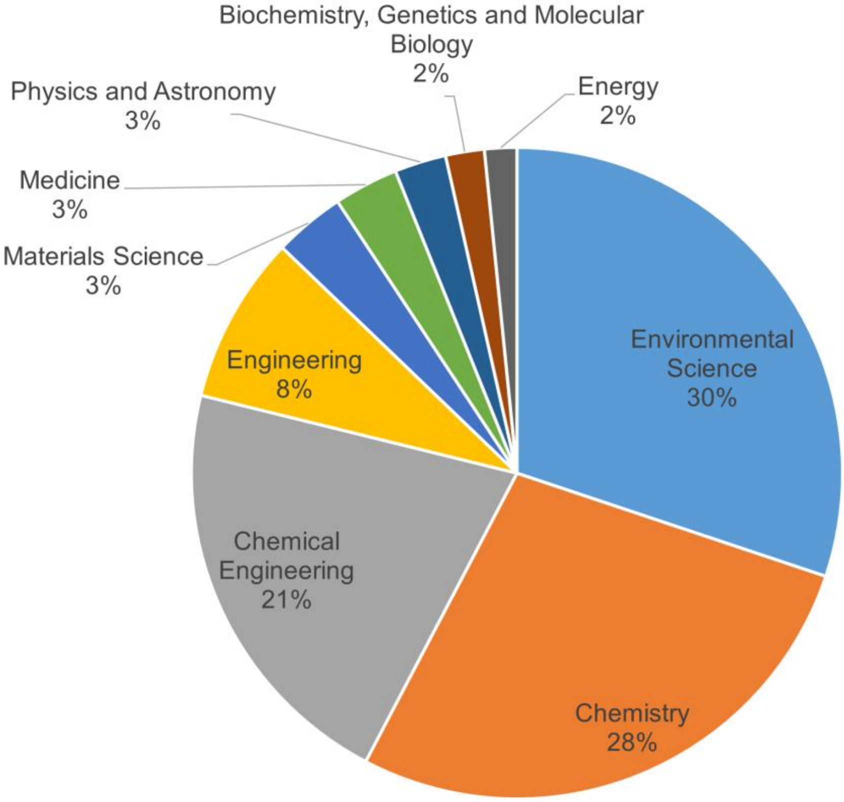

Figure 4. Distribution of the scientific publications according to subject area, based on the SLR article selection performed in the Scopus database. Considered period: 2002–2022. Source: the authors.

In terms of dissemination, articles can be found in more than 60 different journals. Nevertheless, half of those publications are found in only nine of these journals, with more than 30% of the articles published either in Chemosphere, Water Research, Journal Of Electroanalytical Chemistry, or the Chemical Engineering Journal. Figure 6 shows all the journals with at least three publications on the topic.

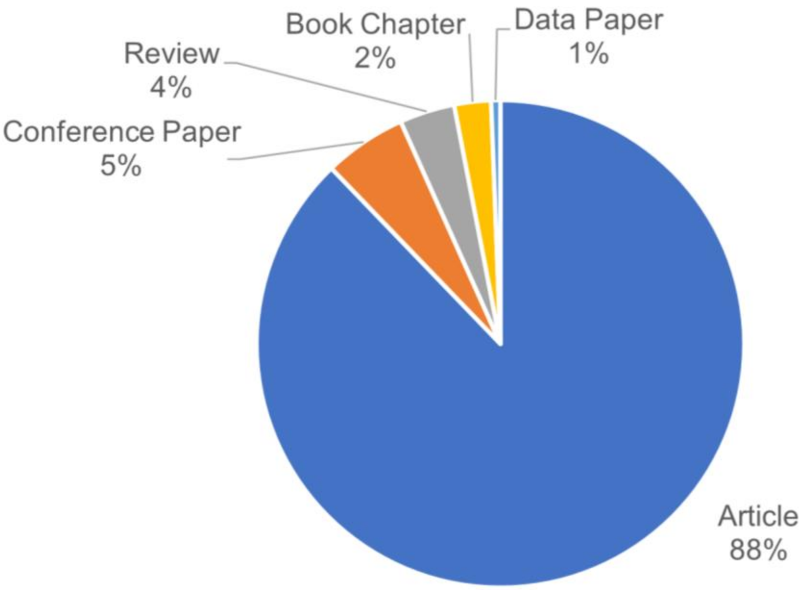

Figure 5. Distribution of publications according to document type, based on the SLR article selection performed in the Scopus database. Considered period: 2002–2022. Source: the authors.

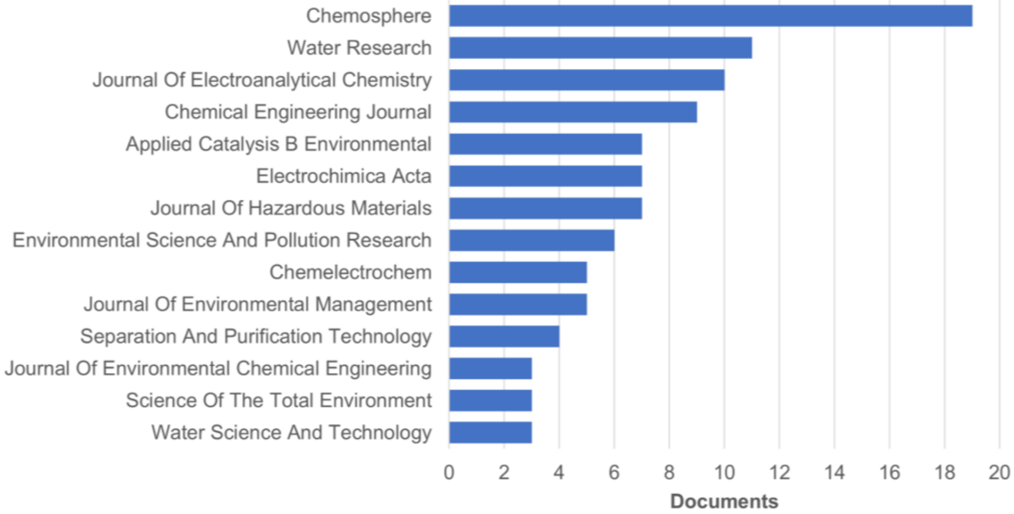

Figure 6. Number of publications per journal with more than three publications on EAOPs using diamond technology for water treatment, based on the SLR article selection performed in the Scopus database. Considered period: 2002–2022. Source: the authors.

These results point to widespread research on diamond technology applied in EAOPs for water treatment across the world, with articles published by universities and research centers from all continents. Figure 7a shows the distribution of articles by country.

The country that stands out in the number of papers on the subject is Spain, having a large share of 35% of publications, followed by Brazil (13%), France (12%), China (10%), and the United States (10%). Together, these five countries account for 80% of the articles on the selected theme. Bibliometric mapping using the VOSviewer tool [55] enables the evaluation of co-authorship of the documents in relation to the countries of publication, as shown in Figure 7b. The countries that collaborated the most were Spain and Brazil, and we can also highlight France, the United States of America, and Italy for their high link strengths, in other words, their elevated number of collaborations with other countries.

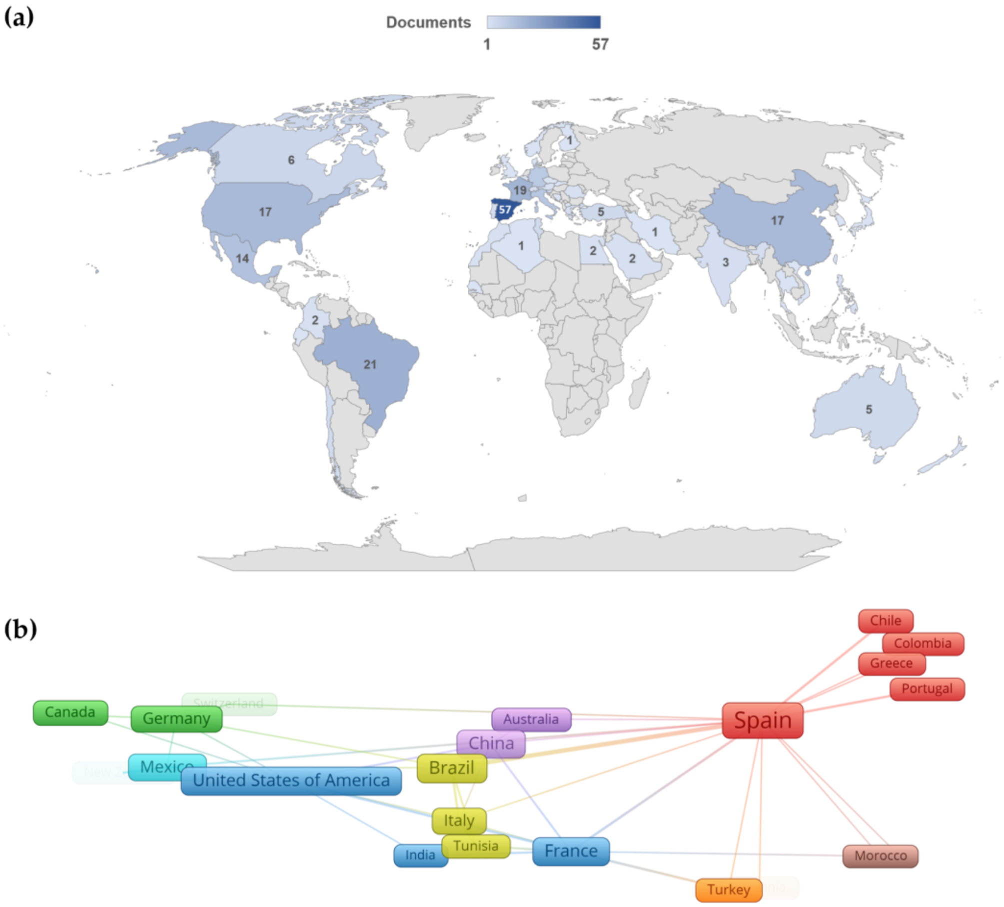

Figure 7. (**a**) Number of publications per country on EAOPs using diamond technology for water treatment; (**b**) countries' co-authorship network (minimum of two occurrences) representation on the application of EAOPs using diamond technology for water treatment, based on the VOSviewer bibliometric mapping software [55]. All results were based on the SLR article selection performed in the Scopus database. Considered period: 2002–2022. Source: the authors.

As expected, due to the high number of articles from Spain, when we evaluate publications by universities and research centers, the Spanish institutions also stand out in number. Notably, the Universitat de Barcelona (Spain) is accountable for 35% of all publications on the topic, in addition to participating in 100% of Spanish publications. Figure 8 presents the distribution of published articles according to universities and research centers that have at least four publications on the subject.

Other outstanding institutions are also worth mentioning in the contribution to the theme, such as Université Paris-Est (France), Universidad de Castilla-La Mancha (Spain), Universidad de Guanajuato (Mexico), and Universidade de São Paulo (Brazil).

A keyword network graph (Figure 9) was built to identify the trends in the use of diamond technology in water treatment through EAOPs, and to verify the relationship among the major topics. In order to build this map, a dictionary of keywords was created, and a pre-treatment of the data was carried out so that there were no repetitions of keywords with the same meaning (e.g.: boron-doped diamond, boron doped diamond, doped diamond, and BDD) but written in a slightly different manner.

There are three major clusters visible in the keywords network graph. As expected, the largest one (red color) represents mostly the use of BDD electrodes in EAOPs for water treatment. This cluster also shows add-ons (complementary techniques) used in different EAOPs, such as UV light or the generation of hydrogen peroxide. Furthermore, the red cluster also presents the most applied pollutants in degradation studies, such as organics, dyes, and pharmaceuticals. The blue cluster mainly represents the most applied EAOP methods: anodic oxidation (AO), electro-Fenton (EF), and photoelectron-Fenton (PEF), as well as the hydroxyl radicals. Their electrogeneration at the diamond surface is the basic requirement for effectively treating water through anodic oxidation since they are the main actors in the mineralization of the pollutants. Finally, the green cluster shows other wastewater treatment methods (photocatalysis, ozonation, Fenton process, and electrocoagulation) that were compared to EAOPs using diamond technology or combined with BDD electrodes to improve efficiency. The keywords: wastewater, real wastewater, and azo dyes also appear, indicating that the group of articles represented by this cluster mostly applied the above-mentioned methods to treat real wastewater and water contaminated with azo dyes.

Figure 8. Number of publications per university and research center with more than four publications on EAOPs using diamond technology for water treatment, based on the SLR article selection performed in the Scopus database. Considered period: 2002–2022. Source: the authors.

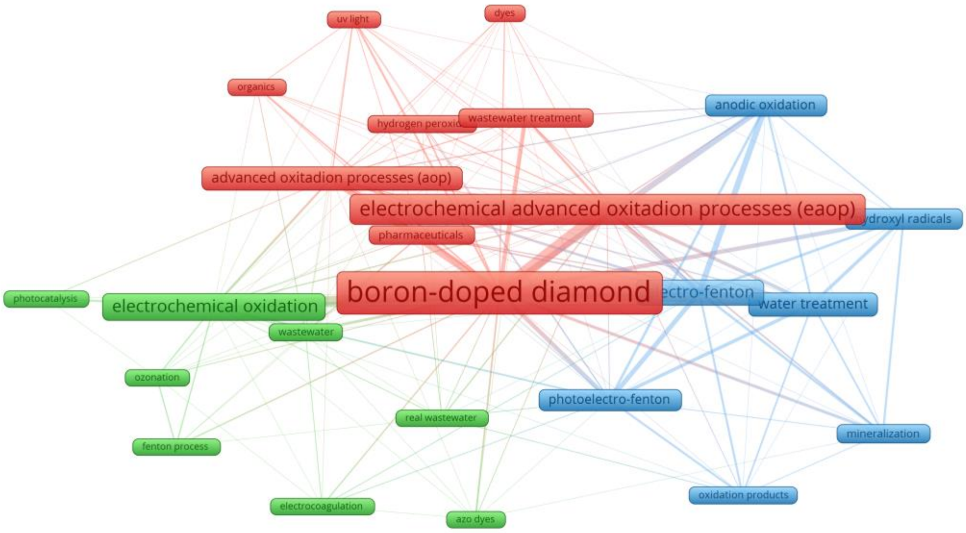

Figure 9. Keywords co-occurrence network (minimum of five occurrences) representation of the trends on the application of EAOPs using diamond technology for water treatment, based on the Scopus database and VOSviewer as the bibliometric mapping software [55,56] (source: the authors).

After careful analysis of the methods applied in water treatment mentioned in each of the SLR-selected articles, we built a network map (Figure 10a), along with a chart containing data on the distribution and frequency of the methods (Figure 10b).

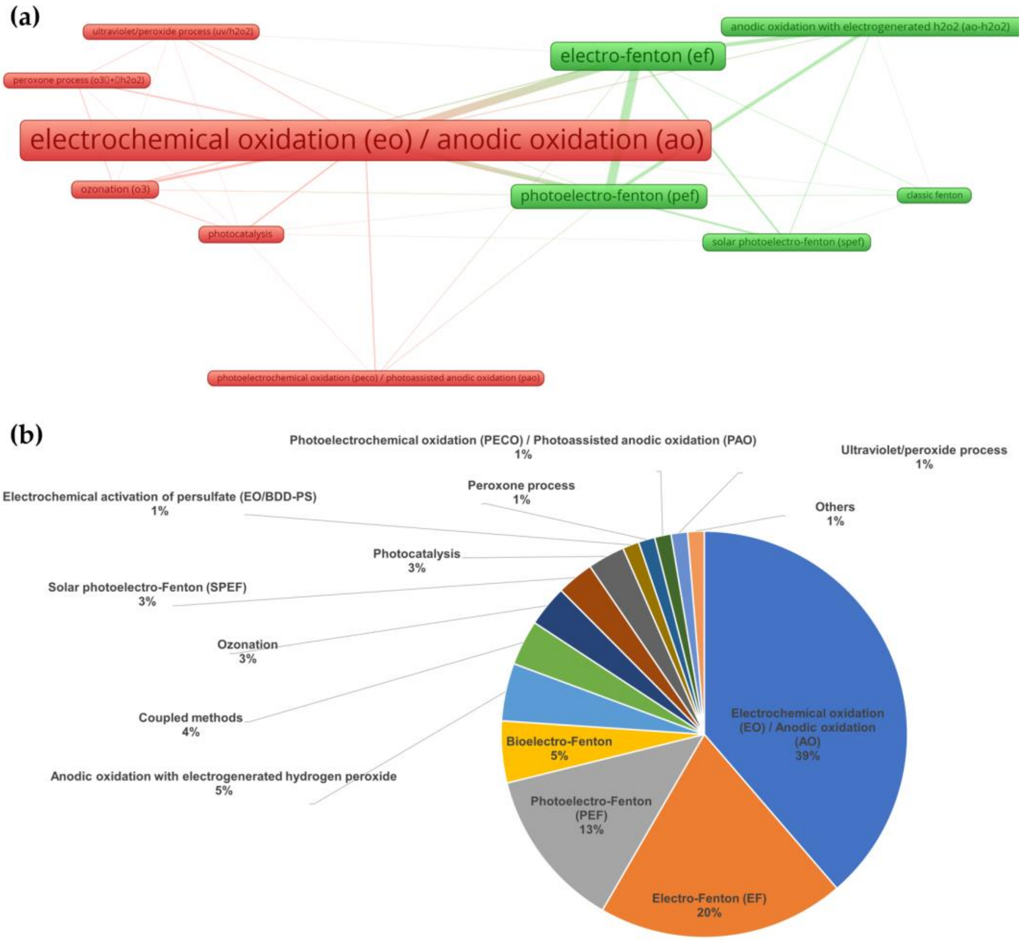

Figure 10. (**a**) EAOPs co-occurrence network (minimum of two occurrences) representation on the application of diamond electrodes for water treatment, based on the VOSviewer bibliometric mapping software [55,56]; (**b**) Distribution data on EAOPs applied in the publications selected by the SLR, based on the Scopus database (source: the authors).

The most widely applied method was anodic oxidation (AO), also frequently referred to as electrochemical oxidation, mentioned in 39% of the articles. Secondly, it was electro-Fenton (EF) (20%), which is based on the electrochemical production of H_2O_2 with the addition of Fe^{2+} in the bulk solution to produce additional hydroxyl radicals as a by-product of the Fenton reaction [57]. The third most applied method was photoelectro-Fenton (PEF) (13%), which is based on the combination of EF with UV light irradiation to enhance the degradation efficiency [58]. In addition, other identified relevant methods were bioelectro-Fenton, anodic oxidation with electrogenerated H_2O_2, ozonation, solar photoelectro-Fenton, photocatalysis, electrochemical activation of persulfate, peroxone process, photoassisted anodic oxidation, and ultraviolet/peroxide process. The methods network graph allowed us to identify which techniques co-occur in the articles, highlighting those that were frequently compared with each other, namely AO, EF, and PEF. A high number of articles comparing these processes with electrogenerated H_2O_2 was also observed, agreeing with the previous indications given by the Venn diagram in Figure 1.

SLR analysis equally revealed the importance of the keywords adopted by each author. Despite applying a systematic method like SLR, we noticed that many articles had not been identified due to the use of non-uniformized keywords or different descriptions for the same EAOP throughout the literature, complicating the identification and proper selection of searched articles. Adopting such practice would only benefit the scientific community and the authors themselves. It would most probably enable the authors to reach out to a higher number of researchers and, consequently, obtain more citations only by adopting standard terms in their titles, abstracts, and keywords. It is certainly important for an author carrying out an SLR to previously identify the maximum possible number of keywords related to the researched topic. However, when there is a large variation between terms representing the same subject, it is likely that some of them will be unknown and/or unidentified. Furthermore, by applying the SLR method using simpler and more comprehensive keywords (such as just "diamond", "EAOP", and "AOP"), we were able to analyze a small but representative sample of hundreds of publications concerning diamond technology applied to EAOPs and extract important insights and relevant information on the theme.

The next sections of this manuscript are organized following the main indications given by the SLR analysis.

3. Doped Diamond Technology Add-Ons

This section focuses on the main explored combination of other techniques (add-ons) to the AO process applying BDD technology: indirect electrolysis to generate additional strong oxidant agents (persulfates, perphosphates, perchlorates or hypochlorite, ozone) and photocatalysis, which enhances the anodic oxidation by the addition of photocatalysts to the AO process.

3.1. Indirect Electrolysis

As previously stated, EAOPs are essentially methods based on the electrooxidation reaction between strong and specific oxidative species and organic water pollutants. Ideally, such chemical reactions should be nonselective and lead to the complete mineralization of the pollutants (R) into carbon dioxide, water, and innocuous inorganic compounds. The efficiency of these eco-friendly processes relies on the on-site generation of the reactive species and their oxidative strength, which, in turn, highly depends on the aqueous media's chemical nature. The hydroxyl radical ($^{\bullet}$OH) is probably the most reported oxidizing agent, since it rapidly interacts with the organic pollutants in a radical oxidation chain reaction [22]. BDD anodes electrogenerate a considerable amount of these radicals (Equations (1) and (2)), but their short lifetime is restricted to the surrounding area of the electrode surface [22].

$$\mathrm{BDD} + \mathrm{H_2O} \rightarrow \mathrm{BDD}(^{\bullet}\mathrm{OH}) + \mathrm{H^+} + \mathrm{e^-} \tag{1}$$

$$\mathrm{BDD}(^{\bullet}\mathrm{OH}) + \mathrm{R} \rightarrow \mathrm{BDD} + \mathrm{CO_2} + \mathrm{H_2O} \tag{2}$$

Another approach to oxidize water pollutants is in situ generation of additional oxidizing agents through indirect electrolysis. On the other hand, BDD electrodes have also been used in the electrochemical synthesis of strong oxidants such as persulfates, perphosphates, perchlorates, or hypochlorite [59]. Anodic oxidation of sulfate, carbonate, or phosphate ions present in the solution produces these mediators according to Equations (3)–(5) [22].

$$2\mathrm{SO_4}^{2-} \rightarrow \mathrm{S_2O_8}^{2-} + 2\mathrm{e^-} \tag{3}$$

$$2\mathrm{CO_3}^{2-} \rightarrow \mathrm{C_2O_6}^{2-} + 2\mathrm{e^-} \tag{4}$$

$$2\mathrm{PO_4}^{3-} \rightarrow \mathrm{P_2O_8}^{4-} + 2\mathrm{e^-} \tag{5}$$

Thus, conductive diamond anodes simultaneously produce hydroxyl radicals as well as other strong oxidizing agents from supporting electrolytes present in wastewaters or added to those with low conductance. Reportedly, these mediated oxidation processes demonstrate higher overall oxidation efficiency when compared to that of conventional BDD electrooxidation [22,59–72]. The success of EAOPs in basic or neutral media has only been recently evaluated, and it appears to be slightly limited. The limitations can be overcome if persulfate radicals are generated at BDD anodes by the electro-activation of sulfate. When competing with water oxidation, these mediators contribute to the inhibition of oxygen evolution and deter mass-transfer limitations. Moreover, EAOP's drawbacks are compensated, and their efficiency and cost reduction are enhanced [73–75].

Ozone is a clean and strong oxidant thoroughly used in pathogenic and bacterial disinfection and in the oxidation of inorganic or organic compounds. It was one of the first oxidants used in the disinfection of public swimming pools and municipal water treatment plants due to its high oxidation standard potential ($E^\circ = 2.1$ V) [76]. According to Equations (6) and (7), the production efficiency of electrogenerated ozone at the anode is limited by the thermodynamically favored oxygen evolution, which takes place at a lower potential [22].

$$3H_2O \rightarrow O_3 + 6e^- + 6H^+ \ (E^\circ = 1.51 \text{ V vs. SHE}) \tag{6}$$

$$2H_2O \rightarrow O_2 + 4e^- + 4H^+ \ (E^\circ = 1.23 \text{ V vs. SHE}) \tag{7}$$

In water disinfection and remediation, ozone production has been focused on reaching higher electrochemical ozone production efficiency [77]. This is primarily attained when performing electrolysis at low temperatures, low interfacial pH (higher degradation efficiency in basic medium compared to the acidic one [78,79]), and using electrodes with high oxygen overpotential such as metallic oxides (MOx, M = Pb, Ti, Ir, Sn, Sb, Ru, Pd, Rh) [79–81]. Electrogenerated ozone using BDD anodes was first reported (to the best of our knowledge) by Katsuki et al., concluding that the percentage of current efficiency was low and dependent on the current density and operating temperature [82]. Since then, several efforts have been made to maximize the current efficiency as well as the amount of ozone produced. Michaud et al. studied the electrolysis in aqueous $HClO_4$ and H_2SO_4 solutions using BDD electrodes and found that small amounts of O_3 and H_2O_2 are formed in both electrolytes [83]. Based on these results, they proposed a simple mechanism for the electrogeneration of hydroxyl radicals, hydrogen peroxide, and ozone, and it is globally adopted. Park demonstrated that ozone was stably generated at BDD anodes for long periods of time [84,85], and Arihara et al. innovated the design of such electrodes and used a freestanding perforated doped diamond electrode which achieved a current efficiency of about 29% at the low applied current of 1 A [86]. The design of the electrodes and/or of the electrochemical cell was further explored by Kraft et al. using a sandwich with a diamond anode, Nafion® polymer electrolyte, and diamond cathode, concluding that ozone production is favored by increasing flow rate and current, but it decreases with increasing conductivity of the electrolysed waters [87]. Sekido and Kitaori developed a small-sized generator driven by a dry-cell (later improved) for use in the average household, obtaining an ozone concentration of over 4 mg/L, which is sufficient for water disinfection [88,89]. A few years later, Honda et al. followed the trend and used a free-electrolyte system including a couple of BDD electrodes as the anode and cathode in combination with Nafion® N117/H^+ as the separating membrane, reaching around 40% of current efficiency [90]. At this point, and side-by-side with nickel-antimony-doped tin dioxide, doped diamond is a promising technology for electrogenerated ozone with a current efficiency of over 30% under mild experimental conditions [81]. Choi et al. studied the influence of inert supporting electrolytes on a solid polymer electrolyte/BDD system and provided a better mechanistic understanding of such an effect [91], and Park et al. tried to find a correlation between the physical and chemical properties of BDD anodes and the ozone concentration found in the water [92]. Kanfra et al. explored the viability of replacing harmful pesticides with short-lived oxidants, such as ozone produced by low operating voltages diamond-based generators, with possible application in agricultural outdoor operation [93]. The geometry and electrochemically active surface area of the BDD electrodes for ozone generation was further investigated by Liu et al. using a microporous BDD electrode etched by oxygen plasma [94]. The latter showed 3.76-times improved ozone generation capacity (under the same unit energy consumption) when compared to the bare BDD. Wood et al. reported the synthesis of nitrogen-doped diamond microparticles under high pressure and high temperature (HPHT), which were then compacted to a freestanding solid electrode and then laser micromachined to give a perforated electrode [95]. These anodes yielded 2.23 ± 0.07 mg/L of ozone per ampere of current, at consistent levels for a continuous 20 h period. Recently, Li et al. explored a solid polymer electrolyte electrolyzer and found that ozone production is favored by the medium current density (125 mA/cm^2) and higher water flow rate (63 L/h) [96]. An ozone concentration of 4.86 mg/L was achieved at the highest current efficiency of 51.29% and the lowest specific power consumption of 37.95 Wh/g.

Photo-assisted electrooxidation using BDD anodes has also been explored as an alternative process for wastewater treatment. Predominantly, the UV/in situ electrochemical oxidation of contaminants is more effective if compared to direct UV irradiation (photolysis) or electrochemical oxidation by BDD technology [97–106]. This is mainly due to the production of homogeneous $^\bullet$OH radicals from in situ generated oxidants such as H_2O_2, O_3, and $S_2O_8^{2-}$ which results in a unique synergistic degradation mechanism with enhanced oxidation and mineralization conditions [98,98–101]. Moreover, electricity is the only required input sustaining the combined UV/AO, which can easily be replaced by the use of solar energy alone [107].

3.2. Photoelectrocatalysis

Conventional chemical advanced oxidation processes (AOPs) usually involve oxidizing species such as hydrogen peroxide (H_2O_2) and a catalyst such as the ferrous ion (Fe^{2+}), known as Fenton or Fenton-like processes, further addressed in Section 4. Photocatalysis is another traditional AOP that requires light irradiation/photons to photolyze the oxidant agent with resulting $^\bullet$OH radicals. Generally, the photocatalyst is a semiconductor and must be excited with higher energy (irradiation with a specific wavelength) than that of its bandgap. The excited electrons are then promoted into the conduction band (e_{cb}^-), leaving positive vacancies or holes (h_{vb}^+) that oxidize water or hydroxide ions, and $^\bullet$OH radicals are then generated, as illustrated in Equations (8)–(10) [108].

$$\text{Photocatalyst} + hv \rightarrow e_{cb}^- + h_{vb}^+ \tag{8}$$

$$h_{vb}^+ + H_2O \rightarrow {}^\bullet OH + H^+ \tag{9}$$

$$h_{vb}^+ + OH^- \rightarrow {}^\bullet OH + H^+ \tag{10}$$

Thus, photocatalysis has been widely adopted as an AOP. Among several semiconductors tested in photocatalysis, titanium dioxide (TiO_2) is the most frequently used. It is a UV-based photocatalyst (crystalline anatase bandgap of 3.2 eV) but is also energy-consuming [108]. Therefore, numerous visible light active photocatalysts have also been described as well as modified TiO_2 catalysts [109,110]. Considering the process application, the photocatalysts can be directly added to the medium enabling a large contact surface between the photons and the catalyst and assuring that the quantum yield is high enough for oxidation of the pollutants. Nevertheless, the suspended catalyst needs to be recovered in a further separation stage. The alternative is to immobilize it onto a carefully chosen substrate and guarantee strong adhesion between the photocatalyst and the support and stability during photocatalysis [109,111,112].

Photocatalysis also has some drawbacks, such as applicability at a limited pH range and the constant need to add H_2O_2 to the media [19,20]. Consequently, EAOPs became the efficient alternative that provides in situ and constant generation of oxidizing agents during electrolysis solely using clean electrons. Furthermore, immobilization of the photocatalysts onto different supports led to different configurations for photocatalytic reactors, and the possible synergy resulting from the combination of photocatalysis and electrochemical anodic oxidation shortly started to be explored, including BDD technology [113]. The main findings for photoelectrocatalytic studies are summarized in Table 4.

Table 4. Examples of reported studies on photoelectrocatalysis using BDD technology.

Support	Catalyst	Pollutant	EAOP	* TOC %	* COD %	** ED %	Ref.
Thin film	TiO_2	Red X-3B	TiO_2 + UV BDD + UV TiO_2@BDD + UV	29.8 67.6 74.2			[114]
Thin film	$ZnWO_4$	Red X-3B	$ZnWO_4$ + UV BDD + UV $ZnWO_4$@BDD + UV	-	-	-	[115]
Thin film	Ce-TiO_2	Red X-3B	Ce-TiO_2 + UV BDD Ce-TiO_2@BDD + UV			0.067 (h^{-1}) 0.137 (h^{-1}) 0.445 (h^{-1})	[116]
Thin film	ZnO	Yellow 15	ZnO@BDD + UV B-ZnO@BDD + UV			35 73	[117]
Film	ZnO	Methyl orange	ZnO@BDD + UV			~85	[118]
Thin film	TiO_2/Sb-doped SnO_2	Bisphenol A	BDD TiO_2/Sb-doped SnO_2@BDD + UV			58.9 67.3	[119]
Film	TiO_2	-	TiO_2@BDD + UV	-	-	-	[120]
Thin film	TiO_2	Methyl orange	TiO_2 + simulated solar light BDD TiO_2@BDD + simulated solar light			45 56 100 (4 h)	[121]
Thin film	TiO_2	Methylene blue	BDD TiO_2@BDD + UV			~20 ~55	[122]
Thin film	TiO_2	Glyphosate	BDD TiO_2@BDD + UV	67 85.3	81 95.2		[123]
Nanoparticles	TiO_2	Herbicides	TiO_2 + simulated solar light BDD TiO_2@BDD + simulated solar light			90 (18.05 h) 90 (1.98 h) 90 (1.15 h)	[124]
Thin film	TiO_2	Diclofenac	BDD TiO_2@BDD + UV	76.1 80.1	83.6 84.7	85.6 98.5	[125]
Thin film	$BiVO_4$	Tetracycline hydrochloride	$BiVO_4$@BDD + simulated solar light			45.1 (10 min)	[126]

* Mineralization parameters: TOC—Total Organic Carbon; COD—Chemical Oxygen Demand. ** ED—Efficiency in pollutant degradation into broken-down products.

Most of the reported works are successful efforts using BDD as the physical support for the combined photocatalysts (mainly TiO_2), which avoids an additional removal step of the catalysts after the process, and enables their recycling and reuse [115–120,125]. Moreover, these studies also demonstrate that photoelectrocatalysis improves the degradation and mineralization of the pollutants when compared to conventional BDD anodic oxidation [114,122,124]. Thus, interesting synergistic effects in the composite photoanode with a 3D framework structure are observed, although, in both oxidation processes, the adopted experimental conditions largely define the degradation and mineralization kinetics of the contaminants. The n/p-type heterojunction established between most of the photocatalysts and BDD has also been further explored, and it is concluded that the nanoparticles properties and the interface structure are highly critical to optimize the accelerated production rate of $^{\bullet}OH$ radicals [121,126]. Lastly, some works also include the determination of energy efficiencies which are equally significant parameters to be taken into account when evaluating EAOPs [125].

In this section, a brief overview of additional strong oxidants simultaneously formed during the electrooxidation of water pollutants was carried out, particularly persulfate radicals and ozone. The electrogeneration of persulfate radicals is a sustainable synergetic effect but is strongly pH dependent which presents a real limitation. On the other hand, anodically activated persulfate radicals through naturally occurring electrolysis processes (resistive heating or combination with UV photolysis as a post-treatment step) further improve the electrocatalytic performance of BDD anodes [69]. Diamond-based clean technology also clearly proved to be one of a few able to efficiently electrogenerate ozone water for disinfection, sterilization, and treatment purposes. The stability of BDD anodes allied with their performance in terms of high-yielding hydroxyl and ozone concentration, with no harmful NO_X formation, greatly benefits EAOPs used in water remediation.

Photoelectrocatalysis offers another add-on to BDD anodic oxidation. In this case, the amount of hydroxyl radicals produced at the diamond anode surface (water electrolysis) is enhanced by additional hydroxyl radicals generated by irradiation of a photocatalyst which, in turn, oxidizes water or hydroxide ions. This process shows good potential, but further development of the design and fabrication of the photoanodes is still required to improve the efficiency of wastewater treatment using BDD technology, and additional costs must also be assessed.

4. EAOPs Synergism with Doped Diamond Technology

In this section, Fenton-based EAOPs applying BDD technology are discussed in detail. The first subsection is related to the literature found on electro-Fenton (EF), and the second one to photo-enhanced electro-Fenton processes.

4.1. Electro-Fenton

The era of Fenton chemistry began in 1894 when Henry J. Fenton first discovered that Fe(II) salts could activate H_2O_2 to oxidize tartaric acid [127]. Peroxides (often H_2O_2) and iron ions undergo reactions known as Fenton reactions (Equation (11)), which result in active oxygen species (primarily hydroxyl radicals) that oxidize organic or inorganic compounds [20].

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + {}^{\bullet}OH + OH^{-} \tag{11}$$

The electro-Fenton (EF) process is an indirect electrochemical process as opposed to anodic oxidation (AO), which is a direct electrooxidation method. It involves the electrogeneration of H_2O_2 at the cathode with the addition of an iron catalyst (Fe^{2+}, Fe^{3+}, or iron oxides) to the solution [128], mostly used among the Fenton-based advanced oxidation processes. Electrogeneration of H_2O_2 is performed by either directly infusing oxygen into Gas Diffusion Electrodes (GDEs) or dissolving O_2 or air into the solution to then be reduced at suitable cathode materials (Equation (12)) [129].

$$O_2 + 2H^{+} + 2e^{-} \rightarrow H_2O_2 \tag{12}$$

Examples of cathodes usually applied in EF are carbon-polytetrafluoroethylene (carbon-PTFE) [130], graphite [131], carbon felt [132], reticulated vitreous carbon [133], carbon nanotubes [134], activated carbon fiber [135], and carbon sponge [136]. The process is known as anodic oxidation with electrogenerated H_2O_2 (AO-H_2O_2) if no catalyst is added to the solution [137]. Furthermore, when H_2O_2 is added to the solution or indirectly produced, the process is often referred to as Fered-Fenton [129]. As many authors do not distinguish between EF and Fered-Fenton, and the latter is often referred to as EF, from this point forward, we will adopt the same notation for both.

Further development and effective application of Fenton-based reactions have been delayed by flaws such as excessive chemical use, resulting in Fe-rich sludge, generation of refractory Fe(III)-carboxylate complexes, and low efficiency as a result of parasitic reactions [137,138]. However, its combination with electrochemistry allowed the research community to get around most of the above-mentioned drawbacks. In EF processes, the commonly used large amounts of iron salts (Fe^{3+}) are reduced at the cathode ($E^{o} = 0.77$ V vs. SHE) and then converted back to Fe^{2+} (Equation (13)) [129].

$$Fe^{3+} + e^{-} \rightarrow Fe^{2+} \tag{13}$$

The cost of the chemical reagents, the sludge generation, and the dangers related to the transport and storage of peroxides are all avoided by the continuous production of H_2O_2 and electro-regeneration of Fe^{2+} at the optimal operational pH value (3.0) [139]. In EF water treatment, organic compounds are oxidized by hydroxyl radicals produced through water oxidation at the reaction layer of the anodes and in the bulk solution by the additional hydroxyl radicals resulting from the Fenton reaction between H_2O_2 and

Fe^{2+} (Equation (11)) [128]. Other reactive oxygen-based species, such as H_2O_2 and the hydroperoxyl radical ($^{\bullet}HO_2$), also contribute to the oxidation of organics in EF-based processes in undivided electrochemical cells [57]. The oxidation of H_2O_2 to O_2 at the anode surface produces $^{\bullet}HO_2$ radicals as intermediate products (Equation (14)) [140].

$$H_2O_2 \rightarrow {}^{\bullet}HO_2 + H^+ + e^- \tag{14}$$

Some researchers also suggest that high-valent oxo-iron complexes may participate in Fenton-based oxidation processes, usually referred to as the non-classical Fenton pathway [141]. There is some consensus among researchers that classical and non-classical mechanisms coexist and predominate in different ways depending on the operating conditions [142].

The first mention (to the best of our knowledge) of an EF process dates back to 1986 when Sudoh et al. applied the method to mineralize phenol in aqueous solutions [139]. Different anode materials (PbO_2, doped SnO_2, IrO_2, RuO_2, and Pt) were tested throughout the earlier publications on EF organics mineralization [128,139,143–145]. Nearly two decades later, BDD began to be applied as the anode in EF methods based on the demonstrated indication that BDD technology is the most suitable anode material for EF water treatment [146]. According to a study by Flox et al., a PbO_2 anode, compared with BDD, was able to completely eliminate m-cresol from aqueous solutions in a shorter amount of time [147]. However, the mineralization process of m-cresol with PbO_2 was significantly less effective under comparable conditions. The BDD anode achieved a quasi-complete mineralization rate of 98%, with an energy consumption of 139 kWh/m^3, while the PbO_2 attained a much lower mineralization rate (68%) and required higher energy consumption (165 kWh/m^3) to achieve the total disappearance of aromatic intermediates. Ganiyu et al. studied the EF mineralization of the analgesic antipyretic 4-aminophenazone with BDD and sub-stoichiometric TiO_2 anodes [148]. After 8 h of treatment with a 30 mA/cm^2 applied current density, the EF/BDD process achieved a Total Organic Carbon (TOC) removal of 96%, while the EF/sub-stoichiometric TiO_2 only removed 51% of TOC. Bocos et al. studied a sequential combination of electrocoagulation (EC) with EF as a post-treatment process to treat solutions spiked with bronopol, a common antiseptic and preservative used in cosmetics and hygiene products to prevent bacterial growth [130]. Four types of anode material were compared in the EF treatment of electrocoagulated solutions: Pt, BDD, IrO_2-based and RuO_2-based dimensionally stable anodes (DSAs). The Pt, IrO_2-based and RuO_2-based anodes were not able to significantly remove TOC from the electrocoagulated solution containing very refractory organic compounds. In contrast, the BDD electrode could eliminate TOC up to 85% from the same solution after 420 min of global treatment (60 min of electrocoagulation and 360 min of EF/BDD). Klidi et al. compared a commercial BDD electrode with a DSA anode (Ti/IrO_2-Ta_2O_5) for the treatment of real paper-mill wastewater samples, and the superior performance of BDD over DSA electrodes was again demonstrated [149]. After 5 h of continuous EF treatment, BDD could remove 85% of TOC from the samples, with a 22% current efficiency (CE), while the DSA anode only removed 40% of TOC, with a 12% CE. In another study, Olvera-Vargas et al. applied the EF process to remediate artificial solutions containing five drugs and one industrial chemical detected in a real pharmaceutical company effluent [135]. Again, BDD achieved better results in terms of TOC removal and CE, promoting almost complete mineralization (97.30 %) after 6 h of EF under optimized conditions (compared to a DSA electrode (Ti/IrO_2-RuO_2)). Despite the optimum solution pH value for EF being considered as 3.0 [139], Olvera-Vargas et al. were able to notably achieve good results treating solutions with near-neutral pH (6.0) [135]. There is also extensive literature comparing BDD with Pt anodes in EF processes. The TOC removal ability and current efficiency of BDD have proved to be superior in the EF degradation of diverse pollutants such as the antibiotic sulfamethoxazole [150], the herbicide mesotrione [151], the dye azure B [152], the herbicide atrazine [153], the pharmaceutical paracetamol [154], and the biocide chloroxylenol [155]. The set of previously mentioned publications confirms that the oxidative action of hydroxyl radicals is substantially more efficient when BDD is used as the anode in EF processes instead of traditional electrodes.

In addition to higher efficiency, the durability and inertness of BDD technology are strong advantages over other materials. Although BDD anodes are expensive to produce, this disadvantage is easily overcome due to their long operating life. According to Muddemann et al., it is possible to tune the BDD electrode for a service life of up to 18 years [156]. Furthermore, BDD is less prone to fouling compared to other electrode materials [41]. Even when fouling occurs, simple polarization inversion can easily clean their surfaces [41]. In addition, they do not release harmful agents, as in the case of the unstable PbO_2 anode, which tends to release significant amounts of lead into the solution turning it even more harmful than in its pretreatment state [128].

The mineralization efficiency of EF processes highly depends on the H_2O_2 electrogeneration and the Fe^{2+} regeneration rate. Therefore, it is crucial to select the most appropriate cathode material to work with BDD anodes to achieve the highest possible process efficiency. Pérez et al. tested three types of cathodes coupled with BDD anodes to improve efficiency and reduce EF's energy consumption and costs [157]. They employed cathodes of stainless-steel, reticulated vitreous carbon (RVC) electrode covered with a carbon black (CB), and polytetrafluoroethylene (PTFE) mixture to act as the active phase for the generation of H_2O_2, referred to as the CB/PTFE-RVC cathode, and an aluminum foam modified with CB and PTFE, referred to as the CB/PTFE-Al. The tests were performed in Na_2SO_4 solutions containing the herbicide clopyralid as the model biorefractory organic pollutant in a microfluidic-flow cell coupled with a jet aerator to reduce ohmic resistance and improve mass transfer. Both BDD + CB/PTFE-RVC and BDD + CB/PTFE-Al combinations could completely remove clopyralid in less than 2 h of EF treatment, showing far superior results compared to the BDD-stainless steel couple (56%). The CB/PTFE-Al and BDD electrode pair had the most impressive results, promoting a 64% reduction in cell voltage compared to CB/PTFE-RVC for the same current density. Through this electrode combination and reactor configuration, degradation of clopyralid was fast, efficient, and completed in less than 1 h, with total energy consumption as low as 20 kWh/kg [157]. Klidi et al. combined BDD anodes with carbon felt, modified carbon felt, and GDE cathodes for paper-mill wastewater treatment [149]. The oxidation efficiency after 5 h presented the following order as a function of the cathode material: GDE (40% TOC removal) > modified carbon felt (29% TOC removal) > carbon felt (16% TOC removal). Özcan et al. verified whether the efficiency of the EF process in the removal of propham was improved if using Pt or carbon sponge cathodes paired with BDD electrodes [136]. The TOC removal efficiency after 6 h was similar for the Pt and carbon sponge cathodes. However, the BDD/carbon sponge configuration was the most energy-effective system, presenting a 21.6% lower energy consumption (4.72 kWh/m^3) compared to that of Pt (6.02 kWh/m^3). BDD technology has also been applied as cathodes [158–162]. Cruz-González et al. studied the ability to electrochemically produce H_2O_2 through O_2 reduction on a BDD cathode in EF. After 40 min of electrooxidation at 23 mA/cm^2, 88 mg/L of H_2O_2 were accumulated at the BDD cathode [158]. Espinoza-Montero et al. reported 250.1 mg/L H_2O_2 electrogeneration after 540 min of electrolysis at 1.5 mA/cm^2 in acidic media using a BDD cathode [159]. The addition of phenol required a higher current density of the system (2.5 mA/cm^2) to obtain 153.0 mg/L of H_2O_2 and achieve total mineralization within the same 540 min. Garcia et al. observed that, with increased current densities (from 7.8 to 31 mA/cm^2), H_2O_2 generation at the BDD cathode is inhibited by the larger acceleration of reduction of O_2 to OH^- and protons to H_2 [160]. According to Oturan, BDD cathodes in EF processes do not offer advantages once equivalent or superior outcomes could be obtained by employing other cathodes that are more reasonably priced (carbon felt, gas diffusion cathode, or carbon sponge) [138]. Nevertheless, studies on the use of BDD as a cathode are still scarce. In addition, due to the limited number of publications combining BDD anodes with different cathode materials and BDD itself, it is not possible to clearly state which configuration is the most suitable for EF processes. BDD technology, paired with different cathodes under the same experimental conditions, requires further investigation to achieve the highest EF efficiencies possible.

Regarding degradation efficiency, the advantages of adopting the EF/BDD process for wastewater treatment over AO/BDD are not consensual in the found literature. Oturan et al. compared both EAOPs for the atrazine herbicide mineralization in Na_2SO_4 solutions [153]. Electrolysis at a current intensity of 1000 mA for 8 h led to 93% TOC removal for AO/BDD and 97% for EF/BDD. The superiority of EF was associated with a higher amount of hydroxyl radicals present in the bulk solution than that generated by BDD, promoting additional organics oxidation. Pinheiro et al. also obtained better mineralization rates for EF/BDD (90.2% TOC) compared to AO/BDD (62.4% TOC) for paracetamol degradation, and the energy consumption was substantially lower for the EF/BDD process (2080 kWh/kg_{TOC}) than that of AO/BDD (5141 kWh/kg_{TOC}) [154]. For bronopol [130] and 4-Aminophenazone [148] degradation, EF/BDD again presented higher TOC removal rates than AO with electrogenerated H_2O_2 (AO-H_2O_2). However, both achieved mineralization rates higher than 90%, and their differences were fairly similar. Thus, these are a few studies pointing out that EF/BDD has higher relative oxidation power than AO/BDD. In contrast, Murati et al. compared mesotrione herbicide mineralization rates applying BDD technology to both AO and EF processes, and both EAOPs resulted in similar TOC removal rates such as 87% and 85% for AO/BDD and EF/BDD, respectively [151]. The performance of AO/BDD was also found to be higher than that of EF/BDD for the biocide chloroxylenol in a study by Skoumal et al., with TOC removal of 94.6% for AO/BDD and 82.5% for EF/BDD [155]. Results from the degradation of some water pollutants applying EF based on BDD technology are summarized in Table 5.

Table 5. Examples of studies that applied BDD anodes to treat pollutants by electro-Fenton processes.

Pollutant	EAOP	Anode	Cathode	Fe^{2+} conc. (mM)	pH	Initial conc. (mM)	Initial TOC (mg/L)	% TOC Removal	Time (min)	Ref.
bronopol	AO-H_2O_2	BDD	carbon-PTFE	-	3	2.78	100	95%	480	[130]
	AO-H_2O_2	BDD	carbon-PTFE	-	-	2.78	100	simulated water: 90%	480	
	EF	BDD	carbon-PTFE	0.5	3	2.78	100	>99%	480	
	EF	BDD	carbon-PTFE	0.5	-	2.78	100	simulated water: >99%	480	
sulfamethoxazole	EF	BDD	carbon-felt	0.2	3	1.3	150	88%	600	[150]
	EF	Pt	carbon-felt	0.2	3	1.3	150	84%	600	
4-Aminophenazone	AO-H_2O_2	BDD	carbon-felt cathode	-	3	0.192	30	98%	480	[148]
	AO-H_2O_2	Pt	carbon-felt cathode	-	3	0.192	30	82 %	480	
	EF	BDD	carbon-felt cathode	0.2	3	0.192	30	99%	480	
	EF	Pt	carbon-felt cathode	0.2	3	0.192	30	94%	480	
paper mill wastewater	EF	BDD	modified carbon felt	0.5	3	-	115	85%	300	[149]
	EF	DSA	modified carbon felt	0.5	3	-	115	40%	300	
mesotrione	AO	BDD	carbon-felt	-	3	0.1	17	95%	480	[151]
	EF	BDD	carbon-felt	0.1	3	0.1	17	94%	480	
	EF	Pt	carbon-felt	0.1	3	0.1	17	91%	480	
azure B	AO-H_2O_2	BDD	carbon-felt piece	-	3	0.1	18	96%	480	[152]
	EF	BDD	carbon-felt piece	0.1	3	0.1	18	95%	480	[135]
	EF	Pt	carbon-felt piece	0.1	3	0.1	18	85%	480	
pharmaceuticals mixture	EF	BDD	carbon-fiber brush	3.33 * mg/L	6	-	40	97.3%	360	[135]
	EF	Ti/IrO_2-RuO_2	carbon-fiber brush	3.33 * mg/L	6	-	40	~91%	360	
atrazine	AO	BDD	carbon felt	-	6.7	0.1	10	93%	480	[153]
	EF	BDD	carbon felt	0.1	3	0.1	10	97%	480	
	EF	Pt	carbon felt	0.1	3	0.1	10	81%	480	
propham	EF	BDD	carbon sponge	0.2	3	0.5	62.3	78%	120	[136]
	EF	BDD	Pt	0.2	3	0.50	62.3	68%	120	
	EF	Pt	carbon sponge	0.2	3	0.50	62.3	61%	120	

* TOC—Total Organic Carbon.

This section shows that BDD is the most promising anode material for EF applications. It was also noted that there is no definition of which type of cathode is best suited to be used in conjunction with BDD anodes in EF processes. To reach such a conclusion, comparing different cathode materials will be necessary, considering the costs, durability, H_2O_2 generation rates, and energy consumption for each electrode pair. EF/BDD presented, in general, higher mineralization rates compared to AO/BDD due to the more significant presence of hydroxyl radicals, especially in the bulk of the solution, promoting indirect oxidation in addition to direct anodic oxidation at the BDD's surface. However, AO/BDD presented mineralization rates similar to or even superior to EF/BDD for certain types of pollutants. Thus, to conclude which process is the most suitable for real large-scale application, it is also necessary to consider the nature of the pollutants and the additional costs and maintenance that EF can generate due to the use of catalysts, pH correction to the optimal values during the process, post-treatment (pH neutralization), or even when GDEs are used, adding gas costs.

4.2. Photo-Enhanced Electro-Fenton Processes

A few years after the first studies in EF [139], Sun and Pignatello suggested irradiating solutions with UV light to improve the oxidizing power of Fenton-based systems [163]. A faster generation of hydroxyl radicals was observed due to the photo-reduction of $Fe(OH)^{2+}$ species at wavelength values higher than 300 nm (Equation (15)) [141,164]. Additionally, Fe(III)-carboxylate complexes undergo photo-decarboxylation by the incident photons (Equation (16)), promoting Fe^{2+} regeneration, which improves the mineralization efficiency [164,165].

$$Fe(OH)^{2+} + h\upsilon \rightarrow Fe^{2+} + {}^{\bullet}OH \tag{15}$$

$$Fe(COOR)^{2+} + h\upsilon \rightarrow Fe^{2+} + CO_2 + {}^{\bullet}R \tag{16}$$

These findings led to the development of the so-called photoelectro-Fenton (PEF) process. These combine EF with either UVA (λ = 315–400 nm), UVB (λ = 285–315 nm), or UVC (λ < 285 nm) radiation emitted from artificial sources [155].

Mineralization of paracetamol [154] and chloroxylenol [166] was compared employing AO/BDD, EF/BDD, and PEF/BDD, with PEF/BDD performing better and achieving nearly complete mineralization (>97%) after 6 h of electrolysis. Alcaide et al. observed extremely low TOC removal rates (<3%) for bentazon herbicide after 240 min when using AO-H_2O_2 with BDD anodes [167]. On the other hand, by applying PEF/BDD, around 77% was removed from the solution. Fernandes et al. also achieved superior performances while applying PEF instead of AO and photoassisted AO for imidacloprid oxidation using BDD technology [168]. Although UV irradiation of the AO/BDD system enhanced TOC removal rates from 42% to 49%, PEF was more efficient, eliminating 66% of TOC. In contrast, Tirado et al. could remediate real cheese whey wastewaters more efficiently with AO photo-assisted using a UVA lamp, demonstrating that PEF does not necessarily have a better performance than photo-assisted AO [169]. In a recent study, Bravo-Yumi et al. compared AO with EF and PEF for the degradation of synthetic solutions containing tannery dyes Violet RL and Green A and using BDD anode/stainless steel cathode [170]. Experiments were carried out under three current densities: 25, 35, and 50 mA/cm^2. For the lowest current density, AO/BDD could remove approximately 64%, 44%, and 63% of color from the solutions containing Green A (80 mg/L), Violet RL (80 mg/L), and a mixture of both dyes (35 mg/L), respectively, after 60 min of electrolysis. After the same time of electrolysis, EF/BDD treatment exhibited approximately 86%, 82%, and 91% of color removal for the solutions contaminated with Green A, Violet RL, and a mixture of both dyes, respectively. The PEF/BDD yielded similar results but slightly superior removal rates: 86% for Green A, 88% for Violet RL, and 93% for the mixture of both dyes. Despite removal efficiencies pointing to PEF/BDD as the best solution compared to AO/BDD and EF/BDD, the conclusion of the aforementioned study points to another finding. Bravo-Yumi et al. also performed a statistical analysis to determine optimum conditions under the lowest operational costs, higher kinetics, and higher degradation efficiency. Thus, the authors considered factors such as current density, EAOP type, initial dye concentration, and dye type. In contrast with the previously mentioned results, the statistical analysis pointed out that AO/BDD has a better performance. This clearly demonstrates that unequivocal conclusions may be drawn from very different results in relation to which EAOPs performs better in real wastewater remediation. Looking closely at the actual panorama, determining the most cost-effective EAOP strongly depends on several outcomes and independent variables but certainly on the main goal to be achieved. One such variable is, for example, the degradation medium itself. Studies comparing EF-based processes applied to synthetic solutions and real water samples (real wastewater [171,172] and reuse, tap and mineral water [173]), showed that degradation of pollutants is more efficient in real samples.

The recent method of solar photo-electro-Fenton (SPEF) was proposed by Casado et al. to tackle the problem of the high cost of electrical energy associated with artificial UV lamps employed in PEF processes [165]. The SPEF technique involves EF oxidation whilst exposing the solution to sunlight and making use of an inexpensive and renewable energy source with wavelengths higher than 300 nm [57]. Solar light provides superior UV radiation intensity as well as extra absorption at wavelengths greater than 400 nm. For example, it is believed that SPEF's mineralization rates are higher for the photolysis of Fe(III)-carboxylate complexes when compared to those by EF or PEF processes [58,155]. As demonstrated in the previous subsection regarding EF processes, BDD technology also features prominently in PEF and SPEF. Pinheiro et al. studied the mineralization of paracetamol by PEF with BDD and Pt anodes [154]. PEF/BDD reached a higher performance in terms of energy consumption (1947 kWh/kg_{TOC}), mineralization efficiency (8.1% MCE), and TOC removal (98.4%) after 6 h of electrolysis at constant applied potential (−2.7 V). Under the same conditions, PEF/Pt achieved values of 2076 kWh/kg_{TOC}, 5.2% MCE, and 86.2% for energy consumption, mineralization efficiency, and TOC removal, respectively. Skoumal et al. also observed faster TOC removal rates and higher mineralization efficiencies for PEF/BDD compared with PEF/Pt in the oxidation of the biocide chloroxylenol [166]. While the PEF/BDD process eliminated around 97% of TOC from the solution after 5 h of treatment, the PEF/Pt process required 6 h to eliminate approximately 93% of TOC. In another study by Skoumal et al., ibuprofen mineralization by EF, PEF, and SPEF was also enhanced when BDD technology was employed instead of Pt. This is easily explained by the fact that hydroxyl radicals generated at BDD show higher oxidizing ability than those produced over Pt [155]. Thiam et al. tested four different anode materials to compare their performances in the degradation of the herbicide chloramben using PEF (BDD, PbO_2, IrO_2-based, and RuO_2-based anodes) [137]. The obtained current efficiency was around 4–5% for all the tested anodes, but the highest mineralization power was that of the BDD anode, particularly in the early stages of the PEF process. These results are similar to those from other authors who established the following oxidation power sequence for PEF and SPEF systems: BDD > PbO_2~IrO_2-based > RuO_2-based [172,174].

SPEF consistently stands out in terms of mineralization performance and energy consumption when compared to other EAOPs. Salmerón et al. compared AO and SPEF in the degradation of pyrimethanil and methomyl pesticides at a pilot plant scale fitted with filter-press-type electrochemical cells equipped with BDD technology [175]. Treating 75 L samples containing 71 mg/L of dissolved organic carbon by SPEF during 120 min eliminated 76% and 70% of pyrimethanil and methomyl, respectively. AO only removed 19% and 33% of pyrimethanil and methomyl, respectively, under the same conditions. Steter et al. treated aqueous mixtures of methyl, ethyl, and propylparaben in real urban wastewater with low conductivity by AO-H_2O_2, EF, and SPEF [172]. AO-H_2O_2 and EF reached similar TOC removal rates within the 35–36% range, whereas SPEF eliminated 51% of TOC. Salazar et al. observed higher mineralization rates for the SPEF treatment of simulated wastewater containing the industrial textile dye Disperse Blue 3 compared with solar photoassisted AO-H_2O_2 and EF [176]. Furthermore, the SPEF process consumed less energy (around 64 kWh/kg_{TOC}) to achieve similar TOC removal rates (90–96%). The superior performance of SPEF in relation to EF was also demonstrated by Flox et al. in the oxidation of o-cresol (128 mg/L) in 0.05 M Na_2SO_4 solutions containing 0.25 mM Fe^{2+} [147]. After 180 min of treatment under 50 mA/cm^2, EF removed around 53% of TOC, whereas SPEF achieved almost complete mineralization (around 95%). Many authors have compared SPEF with PEF using BDD technology in water remediation containing pollutants such as erythrosine B dye [177], pesticide triclopyr [178], bronopol [179], landfill leachate [180,181], Evans blue diazo dye [182], ibuprofen pharmaceutical [155], and herbicide mecoprop [183]. Although most of these studies were carried out under very different operating conditions (aqueous medium, pollutant concentration, Fe^{2+} concentration, applied current density, irradiation intensity, and cell configuration), SPEF was always pointed out as the best in terms of mineralization if compared to PEF. Skoumal et al. demonstrated that irradiating solutions with either UV or sunlight promotes a significant improvement in the kinetic rate, making the process more efficient due to the faster hydroxyl radical production and Fe^{2+} regeneration due to the enhanced photodecomposition of Fe(III)-complexes with acidic intermediates [155]. Under solar irradiation, this phenomenon becomes even more accelerated compared to artificial UV sources [155], explaining the SPEF superiority over PEF and other EAOPs. Nevertheless, the temperature influence over time should also be assessed in SPEF processes.

Experiments conducted without the application of electrical current indicate that the effect of direct photolysis in degradation is negligible [177], demonstrating the excellent synergism between direct oxidation at the BDD surface and photo-enhanced Fenton reactions to achieve faster and more efficient wastewater treatment. However, the degradation mechanisms become slower with time, and in extended electrolysis, oxidation is mainly driven by the hydroxyl radicals formed at the reaction layer of the BDD anode [155,177]. In addition, energy consumption is again an important factor in the EAOP selection. In the study by Clematis et al., PEF and SPEF consumed 515.6 kWh/m^3 and 20.9 kWh/m^3, respectively, to achieve similar degradation performances. In other words, PEF is not entirely suitable for real applications due to its high energy consumption. However, SPEF is limited by weather conditions and the availability of sunlight in many countries throughout the year. Using inexpensive sunlight as a photon source certainly reduces the energy demand, but supplying sufficient power to the electrode set increases the overall cost. Nevertheless, this can easily be overcome by coupling photovoltaic panels or other renewable energy sources to directly power the electrochemical reactor. Thus, the electrical demand for operating the process might be lowered if one turns the process to an entirely self-sufficient system. Furthermore, recent discussions point out that mineralization efficiencies and cost-effectiveness can be further improved by optimizing the design of the electrochemical reactors [184,185].

A few cost studies of different advanced oxidation processes (AOPs) are found in the literature and, for example, three AOPs—AO using BDD electrodes, ozonation, and Fenton oxidation were evaluated by Cañizares et al. [186]. For all wastes, only AO was able to mineralize the contaminants completely. This study also showed that the degradation efficiency fluctuated with the pollutant concentration due to mass transfer control. It was observed that the nature of the contaminants affected the results obtained in the oxidation by ozonation (at pH 12) or by Fenton oxidation and that large amounts of refractory chemicals were typically accumulated during these treatments. Economic analysis revealed that Fenton oxidation has lower operating costs than AO or ozonation, despite the fact that AO may successfully compete with the Fenton process in the treatment of a variety of pollutants. Regardless of the nature of the pollutant, the investment cost for the ozonation process appeared to be higher than that of either AO or Fenton oxidation. In a more recent study, Mousset et al. evaluated the operational cost efficiency of six distinct types of AOPs, including ozonation, Fenton oxidation, H_2O_2 photolysis, photo-Fenton, electro-Fenton, and photoelectro-Fenton [187]. Unfortunately, AO and SPEF were not considered in this study. The most economical AOP overall was electro-Fenton (108–125 €/m^3), considering the use of chemicals, power, and sludge management in the operational cost calculations.

Table 6 summarizes some of the results obtained in studies applying BDD technology in photo-enhanced EF processes for the degradation of water pollutants.

Table 6. Examples of pollutants treated with BDD anodes in photo-enhanced electro-Fenton processes.

Pollutant	EAOP	Fe^{2+} conc. (mM)	pH	Initial conc. (mg/L)	Initial TOC (mg/L)	%TOC Removal	EC (kWh/kg_{TOC})	Time (min)	Ref.
bentazon	AO-H_2O_2	-	3	20	-	<3%	-	240	[167]
	PEF	0.5	3	20	-	77.0%	* 10.1 kWh/m^3	240	
4-Aminoantipyrine	PEF	* 47.75 mg/L	3	62.5	40.6	pure water: 64.6%	-	170	[171]
	PEF	* 47.75 mg/L	3	62.5	51.1	municipal secondary wastewater: 65.8%	-	170	
Disperse Blue 3 dye	EF	0.5	3	* 200 mM	~427	90%	~69	360	[176]
	Solar AO-H_2O_2	-	3	* 200 mM	~328	93%	~87	360	
	SPEF	0.5	3	* 200 mM	~427	96%	~64	360	
paracetamol	AO	-	2.9–3.0	157	100	62.4%	5141	360	[154]
	EF	1	2.9–3.0	157	100	90.2%	2080	360	
	PEF	1	2.9–3.0	157	100	98.4%	1967	360	
chloroxylenol	AO	-	3	100	61.5	94.6%	-	360	[166]
	EF	1	3	100	61.5	82.5%	-	360	
	PEF	1	3	100	61.5	97%	-	300	
imidacloprid	AO	-	3	50	-	42%	-	360	[168]
	PEF	1	3	50	-	66%	-	360	
	AO + UV	-	3	50	-	49%	-	360	
	Photochemical oxidation	-	3	50	-	−4% (increased)	-	360	
	SPEF	1	3	50	-	89%	-	360	
chloramben	PEF	0.5	3.4	* 1.19 mM	100	96.3%	-	300	[137]
o-cresol	EF	0.25	3	128	100	~53%	-	180	[147]
	SPEF	0.25	3	128	100	~95%	-	180	
	SPEF	1	3	128	100	>98%	155	180	
m-cresol	SPEF	0.25	3	128	100	~96%	-	180	
	SPEF	1	3	128	100	>98%	155	180	
p-cresol	SPEF	0.25	3	128	100	~94%	-	180	
	SPEF	1	3	128	100	>98%	155	180	
mixtures of methyl, ethyl, and propyl paraben	AO-H_2O_2	-	3	* 0.3 mM each	-	35%	-	240	[172]
	EF	0.2	3	* 0.3 mM each	-	36%	-	240	
	SPEF	0.2	3	* 0.3 mM each	-	real wastewater: 66%	84	240	
	SPEF	0.2	3	* 0.3 mM each	-	51%	-	240	

Table 6. *Cont.*

Pollutant	EAOP	Fe^{2+} conc. (mM)	pH	Initial conc. (mg/L)	Initial TOC (mg/L)	%TOC Removal	EC (kWh/kg$_{TOC}$)	Time (min)	Ref.
cheese whey wastewater	AO	-	3	-	21.6	34.7%	-	420	[169]
	AO + UV	-	3	-	21.6	48.8%	-	420	
	PEF	-	3	-	21.6	41.8%	-	420	
	UVA light alone	-	3	-	21.6	32.2%	-	420	
17α-Ethinylestradiol (EE2)	PEF	0.5	3	20	-	deionized water: 74.5%	-	180	[173]
	PEF	0.5	3	20	-	reuse water: 88.4%	8598	180	
	PEF	0.5	3	20	-	tap water: 88.3%	8952	180	
	PEF	0.5	3	20	-	mineral water: 93.6%	7764	180	
erythrosine B dye	EF	0.1	3	100	115	~90%	* 21 kWh/m^3	120	[177]
	PEF	0.1	3	100	115	>99%	* 515.6 kWh/m^3	120	
	SPEF	0.1	3	100	115	>99%	* 20.9 kWh/m^3	120	
triclopyr	AO-H_2O_2	-	7	* 12 mM	17.2	38%	* 2.34 kWh/g$_{TOC}$	300	[178]
	EF	0.06 mM Fe(III)–EDDS (1:1)	7	* 12 mM	17.2	47%	* 1.81 kWh/g$_{TOC}$	300	
	PEF	0.06 mM Fe(III)–EDDS (1:1)	7	* 12 mM	17.2	65%	* 19.2 kWh/g$_{TOC}$	300	
	SPEF	0.06 mM Fe(III)–EDDS (1:1)	7	* 12 mM	17.2	62%	* 1.33 kWh/g$_{TOC}$	300	
bronopol	AO-H_2O_2	-	3	* 0.28 mM	10	58%	-	360	[179]
	PEF	0.50	3	* 0.28 mM	10	~91%	-	360	
	SPEF	0.50	3	* 0.28 mM	10	94%	* 4 kWh/g$_{TOC}$	360	
landfill leachate	EF	* [TDI]$_0$ = 60 mg//L	2.8	-	* 337–430 mg/L DOC	~43% DOC	-	300	[180, 181]
	PEF	* [TDI]$_0$ = 60 mg//L	2.8	-	* 337–430 mg/L DOC	~72% DOC	-	300	
	SPEF	* [TDI]$_0$ = 60 mg//L	2.8	-	* 337–430 mg/L DOC	~78% DOC	* 137 kWh/kg$_{DOC}$	300	
Evans Blue diazo dye	EF	0.5	3	* 0.245 mM	* 100 mg/L DOC	~85% DOC	-	360	[182]

Table 6. *Cont.*

Pollutant	EAOP	Fe^{2+} conc. (mM)	pH	Initial conc. (mg/L)	Initial TOC (mg/L)	%TOC Removal	EC (kWh/kg_{TOC})	Time (min)	Ref.
ibuprofen	PEF	0.5	3	* 0.245 mM	* 100 mg/L DOC	>98% DOC	-	360	[155]
	SPEF	0.5	3	* 0.245 mM	* 100 mg/L DOC	>96% DOC	-	150	
	EF	0.5	3	41	* 31 mg/L DOC	81% DOC	-	360	
	PEF	0.5	3	41	* 31 mg/L DOC	94% DOC	-	360	
	SPEF	0.5	3	41	* 31 mg/L DOC	92% DOC	-	240	
mecoprop	AO	-	3	100	56	49%	-	540	[183]
	EF	0.5	3	100	56	69%	-	540	
	PEF	0.5	3	100	56	>96%	-	540	
	SPEF	0.5	3	100	56	>96%	-	540	

* TOC—Total Organic Carbon; MCE—Mineralization Current Efficiency; EC—Energy consumption; $[TDI]_0$—Initial Total Dissolved Iron concentration; DOC—Dissolved Organic Carbon; EDDS—ethylenediamine-N,N′-disuccinic.

In this subsection, it was demonstrated that diamond technology still stands out in photo-enhanced EF processes, as equally found in the previous subsection concerning EF processes. It was also observed that a combination of a radiation source, either artificial (UV lamps) or natural (sunlight), improves the efficiency of the Fenton-based process and the overall mineralization efficiency. The reported works equally point out the benefits of solar irradiation when compared to artificial UV sources, particularly in terms of energy consumption, mineralization rates, and general costs. Combining SPEF with renewable power supplies may be a more cost-effective solution. Nevertheless, SPEF is limited by weather conditions and geolocation. Furthermore, PEF and SPEF are enhanced EF processes with extra costs to be added due to particular requirements such as the use of catalysts, pH correction during and after the process, and gas sources when gas diffusion cathodes are used. Unfortunately, the cost of the latter is often not considered in the overall calculations presented in the literature. Therefore, we reiterate that for real wastewater treatment applications, all costs and life-cycles involved in each EAOP process must be evaluated in order to determine which one is the most cost-effective without compromising long-term quality and efficiency.

5. Final Considerations and Future Prospects

Anodic electrooxidation (AO) carried out using boron-doped diamond (BDD) technology is an electrochemical advanced oxidation process (EAOP) by itself, and these thin films are excellent electrode materials for the previously stated well-known reasons. It is a green process that only requires an electrical current input. DDs are indeed expensive, but their long operating life clearly redeems any required initial investment. Similarly to any other EAOP, there are also a few weaknesses in AO that require further consideration if one intends to apply BDD technology in wastewater treatment effectively. Consequently, it is not surprising that many researchers have started to explore possible synergism between different EAOPs. In this work, we aimed to evaluate the literature concerning BDD technology as the common denominator applied in enhanced AO strategies, including different EAOPs.

SLR analysis demonstrated the importance of using adequate keywords in any scientific report since it incessantly introduces a degree of discrepancy to this systematic methodology. Authors will certainly benefit if standard terms are employed, and this action alone will certainly have clear repercussions on the future number of citations as well as on the effortless finding of publications when using search mechanisms such as Scopus, Web of Science, Google Scholar, PubMed, amongst others. The application of SLR enabled us to review a representative sample of work among hundreds of publications concerning doped diamond technology applied in EAOPs. We strongly believe that SLR methodology will always be an updated, useful tool and, most probably, a mandatory one in the future.

In this review, it was possible to generally conclude that add-ons such as persulfate radicals (amongst others) clearly show a synergistic effect, improving the overall AO process using BDD technology. These in situ electrogenerated radicals compensate for the weaker electrooxidation in the bulk of the wastewater since the production of hydroxyl radicals by the BDD anodes is restricted to the surrounding area of the electrode. Adding extra chemicals to generate persulfate or other radicals is not by all means sustainable, and it may even point to an additional treatment step in the whole process. Electrogenerated ozone add-on enhances the amount of hydroxyl radicals present in the wastewater and overcomes the pH constriction effect on the production of persulfate radicals. Ozone is a powerful oxidant, and it mass-balances the localized production of hydroxyl radicals at the BDD electrodes. On the other hand, associated costs are yet unaccounted for in order to properly assess the success of this add-on. Photoelectrocatalysis shows good potential as an add-on, but, unfortunately, the design and fabrication of the photoanodes is time-consuming and most possibly not a cost-effective add-on with effective large-scale application in wastewater treatment.

Considering the Fenton-based processes using BDD technology, it is generally accepted that the electro-Fenton (EF) method shows higher mineralization rates if compared to simple AO. Among them, mineralization efficiency typically occurs in the following order: solar photo-electro-Fenton (SPEF) > photo-electro-Fenton (PEF) > electro-Fenton (EF). An obvious drawback of Fenton-based processes is that they always involve a required amount of iron compounds to be added to the wastewater, and consequently, a post-treatment step to remove the residual iron is necessary. These steps certainly have associated costs but may be reduced or overcome if the iron source is deposited onto the BDD electrode, as previously observed for the photoanodes in the photoelectrocatalysis add-on. Nevertheless, one needs to ensure that fouling does not occur and that the extension of the Fenton reaction is not restricted to the surrounding areas of the composite anode. Regarding the cathode material, literature shows that a consensus has not yet been reached for EF-based methods. In addition, pH correction of wastewaters with chemicals is nearly mandatory, generating additional expenses and complications when implemented in large-scale facilities. For extended treatment periods of time, the oxidation mechanisms promoted by EF-based methods slowly decrease with time and mineralization becomes dominated by direct AO. Ultimately, PEF is an effective process but requires the use of artificial UV lamps, and again, it means additional expenses and an increase in energy consumption. The use of solar radiation easily overcomes this problem, but it is limited to weather conditions and the intensity of sunlight depending on geolocation and the season of the year.

EAOPs are definitely the future for sustainable wastewater treatment. Nevertheless, EAOPs are also of great complexity with many accountable variables and extended difficult implementation and/or substitution of the conventional treatment methods. A recent study evaluated the operational cost efficiency of six distinct types of AOPs, including ozonation, Fenton, oxidation, H_2O_2 photolysis, photo-Fenton, electro-Fenton, and photoelectro-Fenton (unfortunately, AO and SPEF were not considered). The detailed study included the use of chemicals, power, and sludge management. It was found that electro-Fenton was the most economical AOP, with a cost of around 108–125 €/m^3. One should note that the future of all EAOPs includes upgrading the power supplies to renewable energy sources enabling self-sufficiency, low maintenance requirements, and consequent applicability.

One of the aspects that we consider relevant, from a performance point of view, is the generality of EAOPs processes as solutions for multiple pollutants and types of wastewater. If the mineralization process is incomplete, complex samples with different organic compounds may generate possible by-products with some toxicity level. Currently, it is not possible to generalize EAOPs processes without optimizing them for each type of water. Most publications do not provide enough detailed studies concerning the chemistry of treated water, which is a critical requirement for future practical applications of EAOPs.

EAOPs largely achieve excellent degradation and mineralization efficiencies when combined with doped diamond technology. On the other hand, these efficiencies are restricted by the common limits of AO using BDD anodes. Overall, further research is still needed to enhance its technology readiness level and allow effective acceptance and application in wastewater remediation.

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