Progress and Prospect of Anodic Oxidation for the Remediation of Perfluoroalkyl and Polyfluoroalkyl Substances in Water and Wastewater Using Diamond Electrodes

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Abstract
Although diamond electrodes are widely used in the field of electroanalysis and sensing, their application in the field of environmental engineering has yet to be fully realized. Many research studies have considered their potential application in water and wastewater treatment, where the in-situ electrochemical process can avoid the need for chemical additives by facilitating the oxidation of pollutants on the electrode surface or mediated by electrochemically synthesized oxidants in solution. Diamond-based electro-oxidation can effectively treat a number of organic micropollutants and is now being evaluated for the abatement of perfluorooalkyl and polyfluoroalkyl substances, which pose health concerns and are ubiquitous recalcitrant environmental contaminants. To move implementation of diamond-based electro-oxidation forward, the integration of modifications and codopants to yield more advanced electrode materials needs to be further developed and understood. The progress and current strategies associated with diamond electrode modifications for perfluorooalkyl and polyfluoroalkyl substances abatement as well as future considerations are discussed.

Keywords
Boron-doped diamond, Electro-oxidation, Wastewater, Water treatment, PFAS

Introduction
Perfluorooalkyl and polyfluoroalkyl substances (PFASs) are a wide class of synthetic chemicals, characterized by an aliphatic carbon backbone with fluorine atom substitutions on the alkyl chain. Although PFAS species have been widely manufactured for various industrial applications since the early 1950s, it was not until 2001 that definitive data, published in the literature, documented their adverse health impacts and widespread contamination on a global scale [1]. PFASs are now known to be highly toxic, with many harmful health effects to humans [2, 3, 4], leading many governments across the world to impose strict health-based notification levels [5]. PFASs have an extremely wide range of applications, including as a mist suppressant agent for carcinogenic aerosols [6], aqueous film-forming foams, surfactants, and lubricants [7], and are also present in various household products such as carpets, clothing, and nonstick cookware [8]. PFASs are known to be chemically stable and resistant to biodegradation, as well as recalcitrant to conventional treatment processes [5,9, 10, 11], leading to the accumulation of perfluorinated compounds in the environment, wildlife, and humans [11,12]. The
ubiquity of PFASs in natural waters has rendered them among the most hazardous environmental contaminants and priority substances for monitoring by the European Union Directive 2013/39/EU [13].

PFASs have proven to be recalcitrant toward conventional oxidation processes such as ozone [14], with much research dedicated to other treatment processes such as ultraviolet (UV) photolysis [15, 16, 17, 18, 19, 20] and sonochemical degradation [21, 22, 23], amongst many others [24,25]. In environmentally relevant circumneutral pH conditions, PFAS recalcitrance can be attributed to the presence of fluorine atoms (i.e. \(\ldots\text{–CF}_2\text{–CF}_2\ldots\)). Moreover, in advanced oxidation processes, hydroxyl radicals attack the hydrogen bonds in organics; however, this mechanism cannot proceed with PFASs given the lack of hydrogens to abstract, rendering it relatively inert to hydroxyl radical-mediated oxidation [26].

At present, electrochemical processes are being widely pursued as a viable technology for efficient remediation of PFASs owing to their ability to facilitate direct oxidation at the electrode–electrolyte interface. In particular, diamond electrodes such as boron-doped diamonds (BDDs) are receiving heightened attention owing to their favorable properties for water and wastewater treatment applications [27], enabling a substantially more efficient process owing to their wide potential range of water stability (−1.25–2.3 V_{SHE} [28]). Moreover, BDD has been observed to be resistant to degradation in chemically, mechanically, and thermally adverse conditions [28, 29, 30, 31, 32, 33]. The use of BDD for electro-oxidation has been evaluated for a large number of organic micropollutants in recent years [34, 35, 36, 37], which has captured the attention of many researchers for applications toward PFAS remediation. The following review describes the current state of the art and considers the future research and prospects of diamond electrodes to help address the crisis of environmental PFAS contamination.

State of the art

The efficacy of BDD electrodes for the abatement of PFAS has been widely reported (see summary in Table 1), including investigations of the effects of typical electrochemical process variables, such as current density, supporting electrolyte type and concentration, electrolysis time, hydrodynamic conditions (flowrate/stir speed), and kinetics, amongst others [38, 39, 40, 41, 42]. These studies were conducted in various aqueous media, including surface water [43], groundwater [44, 45, 46], and industrial wastewater [38,39,47, 48, 49]. Similar to BDD electro-oxidation processes targeting other organic micropollutants, its effective oxidative performance is largely attributed to its rapid charge-transfer kinetics, low background current, weak adsorption properties, chemical inertness, and wide electro-potential window [28]. In general, PFAS oxidation via BDD electro-oxidation is a mass transfer limited process, which proceeds primarily through direct oxidation at the electrode surface [50,51], rather than mediated by *OH oxidation. This has been demonstrated previously, whereby perfluorooctanesulfonic acid (PFOS) degradation was unaffected when the *OH scavenger tert-butanol was present during electrolysis [52], indicating the effectiveness of diamond electrodes for PFAS degradation in the presence of co-occurring organic scavengers in the water matrix. The mechanism of degradation via electro-oxidation is dependent on the specific PFAS species; however, general pathways have been observed [53,54] (Figure 1). Owing to its recalcitrance toward *OH attack, the initial decomposition of PFAS is attributed to direct oxidation [50], whereby an electron is transferred
from the carboxyl or sulfonic acid group at the electrode to form a perfluorinated radical
\((\text{CnF}_{2n+1}\text{COO}^*/\text{CnF}_{2n+1}\text{SO}_3^*)\) [55]. Subsequently, this radical
undergoes decarboxylation or desulfonation to form a perfluoroalkyl radical \((\text{CnF}_{2n+1}^*)\), which is then
susceptible to defluorination by reactions with \(^*\text{OH}, \text{O}_2,\) and \(\text{H}_2\text{O}\), as well as via direct oxidation [54].

Figure 1. Mechanism of PFAS degradation using electrochemical oxidation. Cycle I: \(\text{O}_2\), Cycle II: \(^*\text{OH}\), Cycle III:
\(\text{H}_2\text{O}\). PFAS, polyfluoroalkyl substance.
Table 1. Experimental studies concerning the effects of operational parameters on PFAS electrochemical degradation by BDD anode.

<table>
<thead>
<tr>
<th>PFAS</th>
<th>Parameters investigated, unit (levels)</th>
<th>Experimental condition</th>
<th>Results</th>
<th>References</th>
</tr>
</thead>
</table>
| PFOA                        | CD, mAcm$^{-2}$ (2.3, 11.85, 21.4) C, mgL$^{-1}$ (1, 5.5, 10) EC $Na_2SO_4$, gL$^{-1}$ (1.5, 7.85, 14.2) t, h (1, 2.5, 4) | Factorial experiment, Batch mode experiment Anode: Nb/BDD (35.05 cm$^2$) Cathode: Nb/BDD (21.57 cm$^2$) x: 2 cm | • CD and t were the most important factors for PFOA degradation and $F^-$ generation.  
• High EC had negative effect on generation of $F^-$. But it had no significant impact on PFOA degradation.  
• C had slight negative effect on degradation, while it had insignificant effect on $F^-$ generation. | [38]       |
| PFOA                        | P, V(1.5, 2, 2.5, 3, 3.5, 4) CD, mAcm$^{-2}$ (0.12, 0.59, 5.88 17.64, 23.24) pH, (3, 9, 12) C, mgL$^{-1}$ (20, 30, 50) | Batch mode experiments (three-electrode quartz cell) Anode: Si/BDD (8.5 cm$^2$) Cathode: Ti sheet (8.5 cm$^2$) Reference electrode: saturated calomel electrode (SCE) x: 3 cm  
T: 32 °C  
EC ($NaClO_4$): 1.4 gL$^{-1}$ n: 1500 rmin$^{-1}$ | • PFOA decomposition was higher at acidic pH and high anodic P, CD, and C. | [50]       |
| 6:2 chlorinated polyfluoroalkyl ether sulfonate (F–53B) | Anode type $BDD$, $BDD/SnO_2$, $BDD/PbO_2$, $BDD/SnO_2 – F$  
F and Sn ratio (0:1, 1:1, 2:1)  
CD, mAcm$^{-2}$ (5, 15, 20, 30)  
pH (3, 7, 9)  
C, mgL$^{-1}$ (10, 20, 100)  
Electrolyte type $NaCl$, $Na_2SO_4$, $NaClO_4$  
EC ($NaCl$), molL$^{-1}$ (0.5, 1, 2.5) x, cm (0.5, 1, 1.5, 2) | n: 800 rmin$^{-1}$  
T: 40 °C  
Cathode: Pt sheet (2.25 cm$^2$) Reference electrode: SCE | • BDD/SnO2–F had the highest oxidation ability for F–53B.  
• With increasing the ratio of F varying from 0:1 (F:Sn) to 2:1, the removal ratios of F-53 increased.  
• F–53B removal ratio was greater at acidic conditions, and at higher CD and C.  
• The effect of electrolyte on F–53B degradation was in the order of $NaCl > Na_2SO_4 > NaClO_4$.  
• A very high and very small x had negative effect on F–53B degradation. | [65]       |
| Material           | Electrical connectors resistivity (Ti wire), Ω (7, 9) CD, mAcm⁻² (1.8, 20, 27, 40) | Electrolytes: NaCl + Na₂SO₄ Anode: Si/BDD (78.5 cm⁻²) Cathode = Ti sheet (78.5 cm⁻²) x: 3 cm | • Increasing the CD enhanced PFAS removal.  
• Low resistance (7Ω) demonstrated higher removal efficiency for all PFAS with low energy consumption. | [41] |
|--------------------|--------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------|------|
| PFOA PFHxS (perfluorohexanesulfonic acid) PFOS | Standard CD, mAcm⁻² (1, 5, 10, 25, 50) Combined CD, mAcm⁻² (50/1, 50/5, 50/10) | Anode: Nb/BDD (105 cm⁻²) Cathode: Nb/BDD x: 3 mm EC Na₂SO₄: 50 mM | • In standard CD, increasing the CD increased rate constant. However, the higher current was only beneficial in the first 15 min of the process  
• In combined CD, 50/5 mA cm⁻² combination allowed for a 37% reduction in energy usage to obtain 75% defluorination compared to using 50 mA cm⁻² alone.  
• Combined CD of 50/5 mA cm⁻² had great promise in a simulated ion exchange regeneration solution. | [40] |
| PFNA (perfluorononanoic acid) PFDA (perfluorodecanoic acid) | Anode type Ti/SnO₂ − Sb − Ce, Ti/SnO₂ − Sb/ Ce − PbO₂, BDD | Cathode: Ti electrode x: 1.5 cm EC NaClO₄: 10 molL⁻¹ CD: 10 mA cm⁻²: 800 rmin⁻¹ T: 25 °C | BDD had the best performance in the degradation, defluorination and TOC removal of both PFAS compared to modified PbO₂ and SnO₂ electrodes. | [42] |
| PFOA | Cathode type BDD, Pt, Zr, Stainlesssteel | Q: 360 Lh⁻¹ T: 25 °C Anode: Si/BDD (63 cm²) x: 10 mm EC Na₂SO₄: 3.5 molL⁻¹ pH: 4 CD: 7.9 mA cm⁻² | • Among all cathodes tested, Pt acted as an electrocatalyst and improved the defluorination reaction by the reduction reaction of perfluorinated carbonyl intermediates on the cathode. | [56] |

BDD, boron-doped diamond; PFAS, polyfluoroalkyl substance; PFOA, perfluorooctanoic acid; PFOS, perfluorooctanesulfonic acid.  
aStandard CD means using one single CD from the beginning to the end of experiment.  
bCombined CD means using 50 mA/cm² for the first 15 min followed by a lower CD for the rest of experiments.
Owing to their recalcitrance toward •OH, electro-oxidation of PFAS is slow when compared with other organic micropollutants, such as pesticides [34]. This •OH inertness is attributed to the high electronegativity of the fluorine atoms in PFAS species. Dehalogenation of PFAS by electrochemical reduction at the cathode has previously been observed [26, 56, 57, 58], thereby increasing the susceptibility of products to subsequent oxidation at the anode [26]. Reductive activity at the cathode ceases once dehalogenation occurs, whereby electro-oxidation is required for any further degradation. This has also been widely reported with other halogenated chemicals [59, 60, 61, 62, 63, 64].

Although electro-reduction can increase PFAS degradation, substantial research has been dedicated recently to exploring variables to maximize electro-oxidation using diamond anodes via direct electron transfer at the electrode-solution interface. Gomez-Ruiz et al. [66] compared the effect of surface and crystalline features of two commercial BDD anodes on their electrochemical performance for perfluorooctanoic acid (PFOA) electrolysis: ultrananocrystalline diamond (UNCD); and microcrystalline diamond (MCD) electrodes. Results showed that MCD yielded a sharp abatement of PFOA independently of the applied current density (1–5 mA/cm²). In comparison, the UNCD anode provided significantly slower PFOA degradation kinetics. Moreover, the energy requirements for maximum PFOA degradation using the MCD anode were 37-fold lower than that of UNCD. The higher sp³ carbon content, lower boron level, and H-terminated carbon content of the MCD appeared to favor faster PFOA degradation. Alternatively, the ultrananocrystalline surface features and higher conductivity of the UNCD anode limited the electrochemical activity for PFOA electrolysis [66].

Pierpaoli et al. [67] compared the efficacy of two BDD electrodes with different boron contents (0.5k ppm and 10k ppm) in degrading PFOA and PFOS in landfill leachates. Although low BDD showed a greater reduction in chemical oxygen demand, both BDDs performed similarly for the removal of PFOA and PFOS, indicating that boron-doping did not significantly affect PFAS removal efficiency. Moreover, the background current analysis showed that the recorded working potential window was 2.6 and 3.1 VAg/AgCl for 10 k and 0.5 k ppm electrodes, respectively. The slightly narrower potential window was owing to higher carrier concentration in the higher doped electrode [67]. No clear trend regarding the effect of boron-doping levels has been universally established for anodic oxidation processes for water and wastewater treatment applications. Previous research has attributed higher boron-doping concentrations to an increased number of crystallites with a small diameter on the surface and consequently a greater density of active sites, leading to better oxidation performance for azo dyes [68]. In contrast, greater oxidation and degradation of the herbicide tebuthiuron resulted using lower boron-doping (5k ppm) owing to decreased incorporation of impurities in the diamond lattice during chemical vapor deposition [69].

The treatment and selectivity of multiple mixed PFAS species using BDD anodes have been an area of recent research interest. Gomez-Ruiz et al. [70] investigated the electro-oxidation of 6:2 fluorotelomer sulfonamide alkylbetaine and 6:2 fluorotelomer sulfonate, with six other short-chain perfluorocarboxylic acids (PFCAs) in industrial wastewater effluents. The oxidation of the larger chain 6:2 fluorotelomer sulfonamide alkylbetaine molecule was kinetically favorable over the shorter chain 6:2 fluorotelomer sulfonate and PFCAs molecules. Similarly, the simultaneous BDD electro-oxidation of 12 PFCAs, consisting of both short (C3–C6) and long (C7–C18) chains, was investigated in a variety of
water matrices (deionized water, raw surface water, and wastewater treatment effluent) [71]. Once again, PFAS chain length was found to affect oxidation selectivity, whereby larger species were kinetically favored.

To enhance the primarily direct oxidation-mediated process, some researchers have explored the coupling of other processes with that of diamond electrode oxidation. Two recent research studies have investigated the efficacy of BDD electro-oxidation of perfluorohexanoic acid (PFHxA) [72] and hexafluoropropylene oxide dimer acid (HFPO-DA or GenX) [73] in concentrated nanofiltration retentate. Aside from optimizing process parameters (i.e. current density, transport phenomena, reactor design, and so on) and the addition of supplemental treatment technologies such as nanofiltration, other aspects related to diamond electrode materials, such as material modifiers and codopants, are now being explored to further increase their applicability as a viable treatment option for PFAS remediation.

Future outlook

Doped-diamond electrodes are fabricated mainly through chemical vapor deposition or high pressure, high-temperature particle production. Other methods include vacuum annealing and surface transfer doping of undoped diamond [28]. Recently, modifications to BDD have been investigated to improve the electrochemical activity (i.e. electro-oxidation, electrocatalytic performance, and extended solvent window) (see Table 2). Zhuo et al. (2020) [65] used SnO2, PbO2, and SnO2–F as BDD modifiers to investigate the removal of F–53B, a substitute for PFOS. The reported oxygen evolution reaction (OER) potentials for the modified electrodes were 2.35, 2.30, and 2.25 V versus saturated calomel electrode (SCE) for the BDD-SnO2-F, BDD-SnO2, and BDD-PbO2, respectively. When compared with BDD, all modified electrodes exhibited improved removals of F–53B. However, BDD-SnO2-F demonstrated the greatest performance, with a removal of 95.6% after 30 min of electrolysis (30 mA cm−2, pH = 3, 40 °C). In comparison, removal of 91% and 87% was achieved when using the BDD-SnO2 and BDD-PbO2 electrodes, respectively. The authors proposed that a significant level of free •OH exists in solution owing to the repelling effect caused by F electronegativity on the •OH formed on the electrode surface. Consequently, this repelling effect could make more active sites available at the electrode surface to facilitate the direct oxidation of PFAS. In fact, removal of F–53B with a 2:1 F:SnO2 electrode was 1.14 and 1.21 times higher than that observed with a 1:1 and 0:1 F:SnO2 ratio, respectively.

Table 2. Summary of recent modified and codoped BDD PFAS oxidation research.

<table>
<thead>
<tr>
<th>PFAS</th>
<th>Codopant or modification</th>
<th>Oxygen evolution reaction (OER)</th>
<th>Reaction rate constant, k/(s-1)</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td>PFOA</td>
<td>Amount of boron doping (500 and 10 000 ppm)</td>
<td>~2.4 V vs Ag/AgCl (500 ppm) ~2.1 V vs Ag/AgCl (10 000 ppm)</td>
<td>5.55 × 10⁻⁵ (j = 75 mA cm⁻²; 500 ppm) 2.67 × 10⁻⁵ (j = 25 mA cm⁻²; 500 ppm) 5.67 × 10⁻⁵ (j = 75 mA cm⁻²; 10 000 ppm) 1.78 × 10⁻⁵ (j = 25 mA cm⁻²; 10 000 ppm)</td>
<td>[67]</td>
</tr>
<tr>
<td>PFOS</td>
<td></td>
<td></td>
<td>5.85 × 10⁻⁵ (j = 75 mA cm⁻²; 500 ppm)</td>
<td></td>
</tr>
</tbody>
</table>
Liu et al. (2019) [74] observed an increase in PFOA removal when a boron and nitrogen codoped diamond (BND) electrode was used compared with conventional BDD and nitrogen-doped diamond (NDD) electrodes. Although all materials showed similar morphology, oxygen evolution potential, and an identical crystal structure, PFOA reductions of 85.3 and 78.6% were achieved with the BDD and NDD, respectively (4 mA cm\(^{-2}\), 0.05 M Na\(_2\)SO\(_4\), pH\(_0\) = 4.8), although a reduction of 99% was achieved using BND. On average, removal of PFOA with BND electrodes was 1.3 and 1.6 times higher than those observed with BDD and NDD electrodes, respectively. The improved performance of BND over BDD and NDD was attributed to a greater electron transfer rate arising from the boron and nitrogen codoping effect. Moreover, BND also exhibited increased kinetics for PFOA degradation via sulfate activation. Previous research concerning the codoping of boron and nitrogen on graphene and nanodiamond demonstrated that a synergistic coupling enhanced the catalytic performance of the electrode [75, 76, 77]. Boron and nitrogen doping adjusts the electronic structure of carbon materials, which enables the formation of defect-induced active sites for electrochemical activity [76].

In general, the co-oxidation and generation of chemical oxidants for water treatment applications by BDD are a promising area of research [78] and have also been investigated for enhanced PFAS degradation during electro-oxidation. The circum-neutral generation of ferrate from low concentration Fe\(^{2+}\) in synthetic groundwater during a BDD electro-oxidation process was recently observed to
significantly increase PFOS degradation [79]. Moreover, increased oxidation of PFOS was observed in chloride-containing waters owing to the electrosynthesis of Cl\(^{-}\), as well as chlorate (ClO\(_3^{-}\)) and perchlorate (ClO\(_4^{-}\)) by-products [80]. Another study found, that by using hydrogen peroxide as a Cl\(^{-}\) and *OH scavenger, ClO\(_4^{-}\) (an endocrine disruptor) generation could be inhibited without impairing PFOS and PFOA degradation (owing to the primary pathway of direct oxidation) [81]. This process of coupling electrosynthesis and electro-oxidation is worthy of further research investigation.

Other potential areas of interest for future applications of diamond electrodes for the treatment of PFAS contaminants exist beyond the use of codopants, modifiers, and cogeneration of electrolyte-derived oxidant species as described previously. The development of porous and three-dimensional electrodes with larger reactive surface areas more active sites [82,83] should provide for more efficient PFAS degradation. However, the use of porous diamond electrodes has yet to be pursued for PFAS remediation applications, in contrast to investigations of PFAS treatment with Ti\(_4\)O\(_7\)-based porous electrodes [84]. With increased active sites, however, it would be important to also investigate reactor design to maximize PFAS diffusion to the electrode surface. Moreover, integrating nanomaterials into electrodes can lead to a higher surface area, which may improve reaction rates on electrode surfaces [85]. Finally, advances in molecularly imprinted polymer electrodes are key to enhancing the efficiency of electrochemical systems by increasing PFAS selectivity. However, to date, most molecularly imprinted polymer electrode research has been directed toward the field of sensors [86,87], thus their use for PFAS degradation efficacy is mostly unexplored.

**Conclusion**

At present, the use of BDD electrodes for water/wastewater treatment is confined to relatively small-scale applications. Cost reduction [34,88], increased oxidation selectivity, and reactor design [89,90] are important challenges to address to enable their scaleup and application. Further development and application of BDD electro-oxidation technologies could assuage the challenge presented by PFAS contamination of the environment. Although recent scientific and technological advances in this area have been significant, further research opportunities exist to improve process effectiveness. In particular, modifications and codopants for advanced electrode materials can help increase the current efficiency of the electro-oxidation process. Moreover, innovation in the area of porous electrode design may further increase the applicability of the process for PFAS treatment in water and wastewater.

**Declaration of competing interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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**References**


This study presents a thorough investigation of the BDD electro-oxidation of a mixture of 12 PFAS, both short- and long-chains species. In addition to using a variety of water matrices, it provides a comparative analysis of oxidation efficiency toward the various PFAS species.


This paper presents one of the first examples of the use of modified diamond electrodes with co-dopants for the electrochemical degradation of PFAS. The research presented by the authors demonstrated the increased effect boron and nitrogen co-dopants have on the oxidation of PFAS, when compared to conventional BDD electrodes.


This study presents the first example of a simultaneous electro-oxidation and electro-generation process for the circumneutral synthesis of ferrate (Fe(VI)). The relative effect of BDD electro-oxidation, as well as the enhanced affect due to ferrate oxidation is investigated, as well as the effect of co-occurring scavengers (NOM).


