



Challenges and future directions in photoelectro-Fenton techniques: A comprehensive review of emerging applications and innovations

Ihsan H. Dakhil ^{a, b}, Ammar S. Abbas ^{a, *}

a Chemical Engineering Department, College of Engineering, University of Baghdad, Baghdad, Iraq

b Chemical Engineering Department, College of Engineering, Al-Muthanna University, Samawa, Iraq

Abstract

This comprehensive review examines the fundamental principles and practical applications of heterogeneous electrochemical wastewater treatment utilizing Fenton's reactions. The fundamental equations involved in generating hydroxyl-free radicals in electro-Fenton and photoelectro-Fenton processes have been reviewed. Photoelectro-Fenton processes have been proven to be the most effective methods for mineralizing and degrading pollutants in wastewater. The primary focus is on understanding the limitations of hybrid Fenton processes and proposing practical solutions to address these challenges. Additionally, the study evaluated the significance of electrode configuration development and light penetration enhancement in promoting hydrogen peroxide production and enhancing hydroxyl radical generation. These improvements contribute to the enhanced degradation and mineralization of contaminants in groundwater. A comparative analysis of electrode materials, novel reactor configurations, and operating conditions demonstrates the relationship between preparation methods and treatment efficiency. Research gaps for improving the photoelectro-Fenton process are identified, with suggestions for future work. Studies have shown that internal light irradiation leads to higher removal efficiency compared to external lighting systems with the same light source power. The most important recommendations were utilizing multi-electrode stacked reactors to reduce energy consumption, enhancing current efficiency, the shape of the electrodes also plays a vital role in increasing light exposure on the photoanode surfaces, and conducting long-term experiments with various contaminants to demonstrate the reactor's stability, efficiency, and efficacy in treating industrial wastewater.

Keywords: Electrode arrangements; Novel reactors; Fenton hybrid; Doping methods; Fenton's challenges.

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1- Introduction

Water pollution represents the most significant environmental and public health challenge facing humanity in the 21st century. Due to the increasing discharge of industrial effluents, agricultural intensification, and rapid urbanization, a broad spectrum of complex pollutants has been released into the aquatic environment globally [1]. These pollutants span a wide range of compounds that include traditional pollutants (organic and inorganic matter, nutrients, pathogens), heavy metals, industrial chemicals, agricultural discharges, pharmaceuticals and personal care products, endocrine-disrupting compounds, microplastics, and a long list of emerging pollutants of concern [2, 3]. The occurrence of these pollutants, even in traces in the aquatic environment, could disrupt marine life and human drinking water quality as well, and present a high risk to human health via bio-accumulation and possible toxicological effects [4].

Traditional wastewater techniques have been applied to the treatment of industrial wastewater pollutants,

including adsorption [5, 6], coagulation [7], sedimentation [8], ion exchange membrane [9], multilayered membrane [10] and biological methods [11]. These methods have been extensively developed to remove organic and inorganic pollutants such as dyes, pharmaceuticals, and heavy metals from industrial wastewater [12, 13]. However, these conventional treatment methods are always inefficient in treating resistant compounds, such as recalcitrant compounds, which have complex molecular structures, high stability, and low biodegradability [14]. To overcome these drawbacks, extensive efforts have been made to develop advanced treatment techniques that can degrade or mineralize recalcitrant contaminants for the protection of water quality, as well as the use of water recycling for agriculture and industrial uses [15, 16].

Advanced oxidation processes (AOPs) have been identified as the most promising technology for overcoming these challenging treatment issues. AOPs are involved in the production of highly reactive oxygen species, hydroxyl radical ($\cdot\text{OH}$) in particular, with



*Corresponding Author: Email: ammarabbas@coeng.uobaghdad.edu.iq

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powerful oxidative strength ($E^\circ = 2.8 \text{ V}$ vs. SHE) and unselective oxidation on organic species [17]. The high oxidation power of the hydroxyl radical allows it to attack most organic molecules, and a succession of oxidative reactions occurs to turn complex pollutants into smaller and more biodegradable molecules or to achieve their complete mineralization to CO_2 , H_2O , and inorganic ions [18]. This versatility makes AOPs particularly valuable for treating wastewater containing biorefractory contaminants that resist conventional wastewater treatment. Different AOPs have already been developed, such as Fenton and Fenton-like oxidation [19], ozonation [20], photocatalytic [21, 22], and electrochemical oxidation [23]. These treatments proved to work effectively against organic pollutants in wastewater [24-27]. The use of combined AOPs for treating wastewater has been researched, showing a high potential for removing organic pollutants [28]. As a result, hybrid AOPs may provide a promising approach for economic and sustainable wastewater treatment.

The Fenton process can be considered the most prominent electrochemical advanced oxidation process that has attracted attention in recent decades [29]. This process gained more attention due to its simplicity and effectiveness for wastewater treatment. The classic Fenton reaction involves catalytic decomposition of hydrogen peroxide by ferrous ion to produce hydroxyl radicals ($\cdot\text{OH}$) in an acidic medium, as shown in Eq. 1 [30].



The production of hydroxyl radicals, a second highly oxidizing agent after Fluorine, will degrade and mineralize organic contaminants [31]. In the classic Fenton process, it performs best under acidic conditions (pH 2-3), allowing iron to remain physically stable and catalytically active [32]. However, the conventional Fenton process has several associated limitations, including pH correction, continuous iron dosing, and sludge disposal [33]. Various modifications and combinations have been investigated to address these limitations and enhance the Fenton treatment process. Among these technologies, electrochemical advanced oxidation processes (EAOPs) are among the most promising, as they combine electrochemical technology with Fenton chemistry to develop treatment systems that are more effective and environmentally friendly [34]. The electro-Fenton (EF) process is a typical example of this process, where H_2O_2 is generated at the cathode. H_2O_2 generation occurs electrochemically; therefore, there is no requirement to add H_2O_2 externally and store it, which adds to the safety factor and potentially lowers operating costs [35].

Several challenges still need to be addressed regarding the EF approach. In EF, hydrogen peroxide generation is restricted by oxygen diffusion through the water. The current efficiency at low pH (pH 2-3) is also reduced [36]. However, it is energy-consuming, and efficiency depends on the cathode material, current density, oxygen

concentration, and solution conductivity. The EF process has been coupled with light irradiation using a PEF technique to address these drawbacks. PEF could be considered a relatively new hybrid approach with both ecological and economic advantages, in conjunction with surface photocatalysis and EF. It is very promising for its applications in larger units [37]. In the PEF system, hydrogen peroxide is produced continuously in situ on the cathode via the oxygen reduction, and iron ion acts as the catalyst of the Fenton reaction [38]. Simultaneously, UV-irradiation promotes the photo-reduction of Fe^{3+} to Fe^{2+} , accelerating the catalytic cycle and potentially leading to the direct photolysis of intermediate compounds and complexes. The superiority of PEF over other AOPs has been demonstrated for a remarkable diversity of water pollutants. Comprehensive studies have shown its effectiveness for treating pharmaceutical compounds (antibiotics, analgesics, anti-inflammatory compounds), personal care products, industrial chemicals (dyes, surfactants, pesticides), phenolic compounds, and various emerging contaminants [39]. Despite the promising aspects of PEF technology, several challenges hinder its widespread implementation in full-scale wastewater treatment facilities. These include energy consumption concerns, complexity of operation, difficulty of light irradiation, electrode stability, optimal reactor configuration, and process scalability for large-scale applications [40]. Addressing these challenges requires innovative approaches in electrode materials, reactor configurations, and process optimization.

Electrode materials constitute another critical aspect of PEF systems, influencing both process efficiency and long-term stability. Anode materials must exhibit high oxygen evolution overpotential, good conductivity, and exceptional resistance to corrosion under oxidizing conditions. While boron-doped diamond (BDD) electrodes have shown excellent performance due to their wide electrochemical window and weak adsorption properties, their high cost has motivated research into alternative materials such as mixed metal oxides, carbon-based electrodes, and dimensionally stable anodes [41]. Similarly, cathode development focuses on materials with high selectivity for the two-electron oxygen reduction to H_2O_2 , with carbon-based materials (such as graphite, carbon felt, and carbon-PTFE composites) and gas-diffusion electrodes showing particular promise [42]. Mass transfer limitations represent another significant challenge in PEF systems. The efficiency of pollutant degradation depends on the effective transport of reactants (O_2 , $\text{Fe}^{2+}/\text{Fe}^{3+}$, and target pollutants) to reaction sites. Innovative reactor designs incorporating features such as turbulence promoters, enhanced mixing mechanisms, and optimized electrode configurations can improve mass transfer characteristics and overall treatment performance [43]. Nanotechnology integration represents yet another frontier in PEF research. Nanomaterials can enhance process efficiency through various mechanisms, including improved catalytic activity, increased surface area for reactions, enhanced light absorption, and superior electron transfer properties.

Nanostructured electrodes and composite nanomaterials have shown promising results in preliminary studies, though questions regarding long-term stability and potential environmental impacts require careful consideration [44].

According to the Scopus database, a search was conducted for scientific papers focusing on removing persistent organic pollutants (POPs) from simulated and real wastewater up to May 2025. The search used the keywords "photoelectron-Fenton" and "persistent organic pollutants within "title, abstract, and keywords." The search found 21 documents, including 14 articles, four reviews, and 2 book chapters. A comprehensive study of the hybrid Fenton processes for degrading wastewater pollutants has become essential. Exploring novel reactor shapes, electrode fabrication materials, electrode arrangement, and optimal doping techniques to enhance electrode specifications. Based on our knowledge, this aspect remains unexplored in detail.

This comprehensive review aims to provide a detailed analysis of the current state of PEF technology, with a particular focus on recent advances in electrode materials, electrode arrangements, reactor configurations, and operational parameters that enhance the process efficiency of degrading refractory organic pollutants. This study examines photoanode modification strategies, including doping methods that improve light absorption. The influence of reactor geometry and electrode arrangement on system performance is critically evaluated, accompanied by a systematic comparison of different doping methods for fabricating photoanodes and the determination of optimum operating conditions to examine electrode performance. Finally, key challenges and promising research directions have been identified that could facilitate the broader application of PEF technology in industrial wastewater treatment.

2- Hybrid Fenton-based processes

2.1. Electro-Fenton processes

EF is a combined electrochemical and Fenton process in which hazardous reagents are detoxified without their handling and addition, and it consumes a low amount of energy with a short process lifetime [45]. This technique could potentially be used to treat wastewater, including industrial and groundwater [46]. In the process of anodic oxidation, H_2O_2 is continuously generated in situ at a suitable cathode through the two-electron reduction of dissolved oxygen as shown in Eq. 2. The pollutants undergo mineralization through either direct electron transfer processes or the action of radical species, specifically hydroxyl radicals, which are produced on the electrode surface, as demonstrated in Eq. 3 [47].



The free hydroxyl radical is a potent oxidizing agent that can react with organic pollutants in wastewater to form dehydrogenated compounds [48]. Anodic oxidation is typically carried out at the anode in electrochemical cells. In this process, a polluted solution is treated using an anode made of inert materials, commonly composed of BDD and Pt, which are electrodeposited with nanoparticles such as TiO_2 , PbO_2 , and SnO_2 . Under these circumstances, most aromatic solutions exhibit limited mineralization because they produce carboxylic acids that are hard to oxidize [33, 49].

The EF method involves the destruction of pollutants through the combined action of Fenton's reagent in the bulk solution and anodic oxidation at the anode surface. The EF process can be divided into four categories based on the addition or formation of Fenton reagents: cathode EF process (EF- H_2O_2), sacrificial anode EF process (EF-Feox), Fe^{2+} cycling EF process (EF-Fere), and the combined cathode and Fe^{2+} cycling EF process (EF- H_2O_2 -Fere). In the EF- H_2O_2 process, externally added Fe^{2+} and in-situ generated H_2O_2 through electrochemical O_2 reduction on the cathode can eliminate the handling, transportation, and storage costs and risks associated with H_2O_2 . The EF-Feox process involves externally added H_2O_2 while electro-generating Fe^{2+} using a sacrificial anode. However, this method has drawbacks like high anode consumption and significant iron sludge production. Regarding the EF-Fere process, both H_2O_2 and Fe^{2+} are added externally; however, Fe^{3+} from the Fenton reaction is reduced to Fe^{2+} at the cathode, thereby decreasing iron sludge production and the initial Fe^{2+} concentration input. The EF- H_2O_2 -Fere process combines the EF- H_2O_2 and EF-Fere processes. Here, in-situ H_2O_2 generation via cathodic O_2 reduction and Fe^{2+} regeneration through Fe^{3+} reduction on the cathode can eliminate the need for external H_2O_2 addition, reduce iron sludge production, and decrease the initial Fe^{2+} concentration input [50]. The mechanism of reactions in the four different EF processes is illustrated in Fig. 1(a-d).

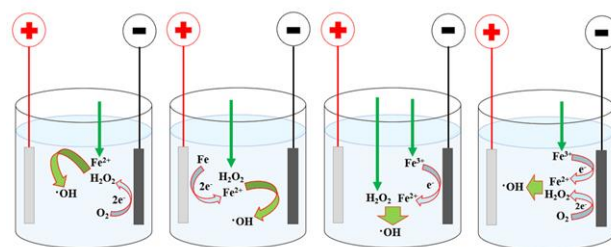


Fig. 1. The reaction mechanisms underlying four different types of EF processes: (a) EF- H_2O_2 , (b) EF-Feox, (c) EF-Fere, and (d) EF- H_2O_2 -Fere

Abbas and Abbas [51] examined various graphite and carbon fiber electrodes in different electrochemical cells using direct, indirect, and EF processes. The best results were obtained with carbon fiber as a cathode via the EF process, resulting in a 55.59% reduction in phenol after 180 minutes. This finding was attributed to the large surface area of the cathode, which increases hydrogen peroxide production.

Abbas and Abbas [52] prepared and characterized MnO₂-graphite electrodes and examined their efficiency in degrading phenolic compounds from wastewater using the EF process under various operating conditions, including temperature, current density, and ferrous concentration. The maximum COD removal was 88% within a reaction time of six hours.

Kuleyin et al. [53] delved into the efficacy of the EF technology in treating biologically processed textile wastewater using graphite electrodes. The EF process was evaluated in removing color, COD and TOC in batch and continuous modes of operation. Different operation parameters, such as pH, current density, reaction time, and Fenton catalyst concentration, were investigated. In a continuous operational mode, removal rates of 89 % color, 93% COD, and 58% TOC were successfully achieved. The results indicated that using the EF process with graphite anodes and cathodes has the potential to enhance the quality of wastewater for reuse.

Al-Khafaji and Mohammed [54] recommended integrating EC and EF to oxidize organic substances in refinery-produced water, thereby reducing COD to meet statutory limitations. The influence of process variables (current density, pH, and reaction time) on COD removal was investigated by the response surface method. The combination of EC and EF efficiently removes oil-contaminated water, and this process is further enhanced by ultraviolet photolysis. The total COD removal reached a high value of 82.9% within 1 hour.

Salazar and Ureta-Zañartu [55] achieved the complete degradation of the triadimefon conazole fungicide in water at pH 3 using EF and PEF processes. A proposed reaction pathway outlines the oxidation of triadimefon by hydroxyl radicals, explaining the formation of nearly all identified intermediates. The electrochemical setup consisted of a one-compartment cell featuring a glassy carbon mesh cathode and an outer steel mesh anode. Throughout the degradation processes, 4-chlorophenol, hydroquinone, carboxylic acids, and inorganic ions were identified as intermediates, ultimately leading to the complete mineralization of triadimefon to CO₂ and H₂O. Nayeibi and Ayati [56] investigated the effectiveness of removing amoxicillin from hospital wastewater using the electrocoagulation (EF) method with an aluminum anode. Through a methodical approach, the optimal values for various process parameters were identified: the antibiotic initial concentration at 100 mg.L⁻¹, reaction time of 90 minutes, initial pH of samples set at neutral, supporting electrolyte concentration of 0.02 M Na₂SO₄, electrode distance of 5.5 cm, and applied current density of 5.5 mA.cm⁻². Using aluminum anodes significantly improved the process efficiency under neutral pH conditions, achieving a removal rate of approximately 95%. This enhancement could potentially address the constraints faced by traditional EF processes when degrading amoxicillin.

Al-Khafaji and Mohammed [57] suggested using the EF process to degrade organic pollutants in produced water,

which is an effluent from an oil refinery. Continuous electrochemical (EF) processes were analyzed using a dimensionally stable anode of Ti-RuO₂/IrO₂ and an activated carbon fiber felt (ACFF) cathode. The effect of operating variables, such as Fenton catalyst concentration, current density, and reaction time, on COD removal efficiency was examined using response surface methodology. Through multiple response optimization for continuous EF experiments, it was revealed that under optimal conditions (Fenton catalyst concentration of 0.306 mM, current density of 156.6 mA, and reaction time of 180 min), the COD removal efficiency reached 73.33% with an electrical energy consumption of 0.901 kWh/kg COD.

Vigil-Castillo et al. [58] conducted a study on degrading the asulam herbicide through the EF process using an undivided electrochemical BDD/carbon felt cell to produce H₂O₂ continuously. A central composite design, in combination with response surface methodology, was employed to determine the optimal operational parameters: a current density of 0.30 A, a Fenton catalyst concentration of 0.3 mM, and a Na₂SO₄ concentration of 0.11 M at pH 3, which ensured complete asulam degradation through electro-Fenton (EF). Aromatic intermediates such as 4-aminobenzenesulfonamide, 4-aminophenol, and 4-benzoquinone were identified, while acetic acid, oxalic acid, and NO₃ ions were characterized as final degradation by-products. This study also conducted a comparison between the EF and PEF using solar irradiation. It was determined that exposure to natural sunlight plays a role in increasing •OH production, and the SPEF process emerges as a viable choice for large-scale water treatment. Table 1 summarizes the materials used for electrodes in EF processes for removing various pollutants in the cited studies.

In summary, the EF processes show promising results in removing various pollutants from different types of wastewaters using diverse electrode materials and configurations. However, its efficiency in removing persistent compounds was relatively low.

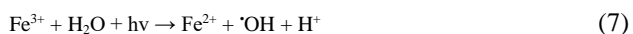
2.2. Photoelectro-Fenton processes

PEF processes are advanced oxidation processes that utilize UV light, fluorescent lamps, or sunlight to generate hydroxyl radicals to degrade various pollutants. These processes utilize photocatalysts, such as TiO₂, ZnO, and CuO, which can enhance the efficiency of the degradation reactions. Combining electro-Fenton (EF) and photocatalytic degradation in a photoelectrochemical (PEF) system effectively eliminates pollutants, including organic and inorganic compounds, achieving high degradation efficiencies. The main operating factors that affect the performance of the PEF process are pH, initial concentration of pollutant, current density, and type of catalyst [59, 68-70].

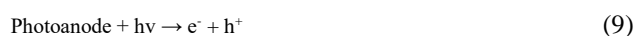
Table 1. Summary of electrode materials in EF processes for the degradation of various pollutants

Pollutant (s)	Electrode		Time, min	Removal Eff., %	Ref.
	Anode	Cathode			
Phenol	Graphite	Carbon fiber	180	55.59%	[51]
Phenol	Graphite	Graphite	180	88.01%	[52]
Textile Wastewater	Graphite	Graphite	30	88%	[53]
Produced Water	Ti-RuO ₂ /IrO ₂	ACFF	60	82.9%	[54]
Pesticides	Steel mesh	Carbon mesh	30	26%	[55]
Amoxicillin	Aluminum	Graphite	90	95%	[56]
Produced Water	Ti-RuO ₂ /IrO ₂	ACFF	180	73.33%	[57]
Erythrosine B dye	BDD	Carbon felt	120	90%	[59]
Pesticides (triclopyr)	IrO ₂	Carbon-PTEF air-diffusion	300	30%	[60]
Pesticides (triclopyr)	BDD	Carbon-PTEF air-diffusion	270	47%	[60]
Ponceau SS diazo dye	Ti/Pt	Carbon-PTEF air diffusion	360	60%	[61]
Ponceau SS diazo dye	BDD	Carbon-PTEF air diffusion	360	70.6%	[61]
Methyl orange dye	Pt	Fe-Activated carbon	30	96%	[62]
Sulfonamide	Pt	Carbon-felt	480	95%	[63]
Sulfonamide	BDD	Carbon-felt	190	50%	[63]
Naproxen	Pt	Carbon-PTEF air diffusion	240	40%	[64]
Naproxen	BDD	Carbon-PTEF air diffusion	240	46%	[64]
Naproxen	IrO ₂	Carbon-PTEF air diffusion	240	49%	[64]
Sulfamethoxazole	RuO ₂ /Ti	ACF felt	360	63%	[65]
Acetaminophen	Ti/RuO ₂ /IrO ₂	SS	120	98%	[66]
Landfill leachate	BDD	Carbon felt	240	66%	[67]

The PEF process utilizes UVC light with a wavelength ranging from 200 to 280 nm. The primary advantage of this process is the continuous generation of hydrogen peroxide (H₂O₂) through the reduction of oxygen (O₂) on a carbon-based cathode, as illustrated in Eq. 4. The procedure involves the production of hydroxyl radical (•OH) by catalytically decomposing H₂O₂ with Fe²⁺, as shown in Eq. 5, in an acidic medium with a pH of 2-3. Furthermore, the simultaneous cathodic regeneration of Fe²⁺ is performed to avoid the accumulation of iron sludge, as shown in Eq. 6 [71]. In addition, the utilization of UV light in the PEF process accelerates the degradation of contaminants through the implementation of the following mechanisms: (i) photochemical regeneration of Fe²⁺ by photoreduction of Fe³⁺ to generate a larger amount of hydroxyl radicals, as shown in Eq. 7, and ii) photolysis of H₂O₂ molecules to generate •OH as shown in Eq. 8 [59].



The PEF process facilitates the degradation of recalcitrant carboxylic acid intermediates that often accumulate during Fenton and EF treatments. The photodecarboxylation pathway contributes significantly to achieving complete mineralization of organic pollutants [72]. When semiconductor photoanodes are employed in PEF systems, additional photocatalytic pathways contribute to pollutant degradation by generating electron-hole pairs and subsequently forming reactive oxygen species, as illustrated in Eqs. 9-12.



One of the primary limitations of PEF processes is the necessity for suitable lamps to generate artificial UV light, resulting in a potential up to 40% increase in operating costs compared to the Fenton process [73]. Babuponnusami and Muthukumar [74] conducted a comparative study on the performance of advanced oxidation processes, including Fenton, EF, sonoelectro-Fenton, and photoelectron-Fenton, for eliminating phenol. This study observed that the degradation efficiency follows the order PEF < SEF < EF < F. The photoelectron-Fenton process achieved complete degradation of phenol and 64.19% mineralization after 30 minutes. Meanwhile, Fenton and EF required more time for the degradation and mineralization of phenol.

A study by Da Costa Soares et al. [60] addressed the challenge of the Fenton-based process at neutral pH for the mineralization of herbicides, utilizing Fe(II)-EDDS as a catalyst. The first scope was a comparison study to evaluate the performance of an anode made from BDD and IrO₂ in the PEF process for degrading triclopyr. The results showed a clear enhancement in the electrolytic mineralization of pollutants with the BDD anode compared to IrO₂, due to the high yield of hydroxyl radicals. On the contrary, the anode based on IrO₂ is more cost-effective. In addition to the original research objective, the effect of PEF on the degradation profile was investigated in comparison with SPEF. Because the photon intensity of sunlight is higher than that of UV, the photolytic reaction rate is enhanced, producing hydroxyl radicals, which in turn improve the EF process. Studies have demonstrated that SPEF is more effective than PEF under identical operating conditions.

Dos Santos et al. [61] studied and compared the effectiveness of various electrochemical methods, including EC-H₂O₂, EF, PEF, and PF-based processes, for a similar electrolyte of Ponceau SS diazo dye in an acidic sulfate medium, employing Pt-air diffusion and BBD-air diffusion anodes to investigate their performance. The study found that BBD anodes increased degradation and mineralization; however, it recommended using Pt anodes for PEF because they were more cost-effective than BBD anodes. This study demonstrated the potential of using iron-recycled moieties grafted to activated carbon electrodes to remove the need for iron salts in EF reactions, thereby overcoming the inherent requirement for iron salts in EF and PEF processes. The effectiveness of the PEF process was assessed based on the decolorization and oxidation of the dye solution following a defined electrolysis time. Results showed that using Fe/AC material in the PEF process significantly improved color removal and total organic carbon (TOC) reduction, confirming the effectiveness of the Fe/AC electrode in the PEC degradation of pollutants.

Al-Khafaji and Mohammed [57] investigated the improvement of the continuous EF process using UVA irradiation. The study demonstrated that the COD removal efficiency in continuous EF increased to 81.1% and 86.0% when one and two UVA lamps (3 Watts each) were introduced, respectively. The study concludes that EF is an efficient method for degrading produced water and suggests that further improvements can be made by incorporating photo-assisted techniques.

Crispim et al. [67] demonstrated that using more powerful UV light enhanced the effective removal of COD in the PEF process. Specifically, employing the UVC lamp led to a 1.35-fold increase compared to using the UVA lamp in PEF. This suggests that a higher amount of oxidants were produced during the PEF UVC process, enabling various oxidation mechanisms such as the direct breakdown of the generated byproducts through photolysis and the indirect oxidation by oxidants.

It can be concluded that BBD, Pt, or Ti were served as anodes due to their superior oxidant power, allowing them to generate hydroxyl radicals ([•]OH) directly, with high chemical stability under harsh reaction media and less significant deterioration, and high oxygen over potential which favors the generation of hydroxyl radicals. In the same context, carbon-based cathodes were selected due to their effective production of hydrogen peroxide, which facilitates two-electron oxygen reduction. They also feature a high surface area that increases the reaction site for H₂O₂ generation, a cost-effective, porous structure that promotes efficient oxygen diffusion, good electrical conductivity while remaining stable in the presence of H₂O₂ and generated radicals.

There are several ways to enhance the photoactivity of the electrode surface, thereby increasing the active area for the reaction. These methods mainly include chemical reduction of metal salts and electrical deposition of the

anode or cathode electrodes [77]. Electrochemical procedures involve the deposition of various materials. Some examples of these deposited materials include:

- 1- Coating surfaces with precious metals such as platinum and ruthenium using the galvanic reduction method of salts of those metals, such as titanium nanotubes or meshes [78].
- 2- Several metals, such as nickel, platinum, tin, and copper alloys, are used in various ways, including immersion, electroless, and cathodic deposition [79].
- 3- Metal oxides, such as lead dioxide and manganese dioxide, are produced by electrolytic deposition on the anode electrode [80].
- 4- Improve electrodes by making a composite of different materials, such as metal, ceramic, and polymer coating, using anodic electrodeposition.

Based on a bibliometric study conducted on the Scopus database, the number of scientific studies dedicated to PEF increased by almost 300% between 2010 and 2025, indicating that the scientific community is recognizing its potential for application in wastewater treatment. This promotion of research interest has led to significant progress in PEF technology and equipment, including electrode materials, reactor types, and operating conditions. Table 2 summarizes the maximum wavelength of UV for degrading different pollutants and types of electrodes in PEF processes.

3- Material and configuration of electrodes

Electrochemical (EC) and photoelectrochemical (PEC) reactors have garnered significant attention as promising techniques for wastewater treatment due to their eco-friendly and operational flexibility. The performance of these reactors is highly dependent on the reactor arrangement, and both divided and undivided cells are often used; divided cells avoid the undesired side reactions but lead to increasing electrical resistance, whereas undivided cells provide a simple design and reduce energy cost [34]. The material of the electrodes is vital in terms of their performance, durability, and dimensionally stable anode to be efficient in the degradation of pollutants [81]. In photoelectrochemical technologies, which combine photocatalysis and electrochemical reactions, the source of light is of extreme importance, where UVA (315 to 400 nm wavelength) has cheaper energy and can efficiently activate the widely types of photocatalyst, whereas UVC (100 to 280 nm wavelength) has higher energy and promotes direct photolysis of resistant compounds and intensifies disinfection, although having higher application costs [82]. The synergetic effect of electrochemical processes and photocatalysis in these hybrid reactors has been proven to improve treatment efficiency due to free radical formation and electron-hole separation [83, 84].

Table 2. Summary of operating conditions for different pollutants eliminated using the PEF process

Pollutant	Anode / Cathode	λ_{\max} UV (nm)	UV Power (W)	UV Position	Time, min	Removal Eff., %	Ref.
Asulam herbicide	BDD / Carbon felt	360	13	External	180	98%	[58]
Erythrosine B dye	BDD / Carbon felt	360	100	External	90	95%	[59]
triclopyr	IrO ₂ / Carbon felt	365	6	External	270	ε ε %	[60]
triclopyr	BBD / Carbon felt	365	6	External	180	66%	[60]
Ponceau SS diazo dye	Pt / Carbon-PTEF air diffusion	360	160	Internal	360	93.4%	[61]
Ponceau SS diazo dye	BBD / Carbon-PTEF air diffusion	360	160	Internal	360	97.6%	[61]
Methyl orange dye	Pt / Fe-Activated carbon	365	75	Internal	30	98%	[62]
Naproxen	Pt / Carbon-PTEF air diffusion	360	160	Internal	120	80%	[64]
Naproxen	BBD / Carbon-PTEF air diffusion	360	160	Internal	120	83%	[64]
Naproxen	IrO ₂ / Carbon-PTEF air diffusion	360	160	Internal	120	78%	[64]
Sulfamethoxazole	RuO ₂ -Ti / ACF felt	365	100	External	360	80%	[65]
Acetaminophen	Ti-RuO ₂ -IrO ₂ / SS	360	48	External	120	97%	[66]
Landfill leachate	BBD / Carbon felt	360	6	External	240	68%	[67]
Landfill leachate	BBD / Carbon felt	254	6	External	240	89%	[67]
Herbicide	Pt / Carbon-air diffusion	300	160	External	120	75%	[75]
Acid Blue 92	BBD / CNTs-PTEF	254	15	External	45	85%	[76]

In this section of the study, we will review innovative electrochemical and photoelectrochemical reactors used for wastewater treatment. This review will address the electrode materials, the arrangement of the electrodes within the reactors, and the types of light irradiation used in the photoelectrochemical reactors.

McQuillan et al. [46] evaluated the capability of inexpensive and readily available graphite electrodes to enhance the path of the EF process. A novel approach of the EF reactor was characterized in the electrolysis system using five graphite rods (diameter of 6.3 mm) arranged vertically in a circular configuration as a cathodic region, with a single carbon rod (diameter of 8 mm) inserted in the reactor as an anodic region. The anode and cathode distance was set at a fixed distance of 10 mm in all experiments. The EF cell layout used in this study is shown in Fig. 2a.

Montenegro-Ayo et al. [85] proposed a new pre-pilot scale reactor to degrade the antibiotic ciprofloxacin in a batch recirculation loop via electrochemical oxidation. The proposed pre-pilot reactor features a circular plate of BDD (10 cm diameter) serving as the reactor's base, in the form of an anode, along with a concave disk of stainless steel (5.5 cm diameter) used as a cathode, positioned 2 cm below the anode. Fig. 2b shows the proposed configuration of a pre-pilot reactor. A mathematical model was conducted using COMSOL Multiphysics software to assess the performance of the proposed reactor.

Lei et al. [86] developed an eccentric column-shaped electrochemical reactor. The reactor consists of the tubular cylindrical cathode (stainless steel) and the Pt-coated Ti anode (economic and high surface area) for precipitation. For uniform current distribution, the cathode (cylindrical) was attached vertically to the inner side of the reactor's wall, and the anode at the center of the reactor, as illustrated in Fig. 2c. In addition, it is possible to automate the buildup of deposits without separating the reactor. First, a study was conducted to verify the viability of this new layout. Furthermore, the formic acid decomposition was evaluated in a continuous flow configuration. Finally, the long-term performance and scalability of the system were assessed.

Abbas and Abbas [52] constructed an undivided electrochemical cell containing a graphite rod with dimensions of (1.5 cm diameter and 6 cm long) which worked as an anode, while the cathode was a hollow-cylindrical graphite with dimensions of (8 cm inner diameter, 10 cm outer diameter, and length 15 cm) as shown in Fig. 2d. This study was concerned with modifying graphite electrodes by electroplating with a phase of γ -MnO₂.

Li et al. [87] discovered a novel type of electrode generated by EF using aeration graphite felt electrodes to remove organic pollutants, which enhanced both the production of H₂O₂ and the cyclic utilization of Fe³⁺/Fe²⁺ ions. Moreover, the H₂O₂ generation achieved in this method was high (152-169 mg.L⁻¹) at a wide working pH (3-10). The new cell consisted of a piece of graphite with dimensions of 3×4×1.7 cm³ as the active dimension, aerated by inserting eleven fiber tubes that functioned as both cathode and anode, with an active dimension of 3×4 cm², used as a Ti/IrO₂/RuO₂ mesh. The oxygen transfer to the GF fiber was significantly improved due to aeration into the graphite felt electrode, resulting in the formation of H₂O₂. The aeration of the electrode also promoted the rapid mass transfer of ions from the bulk solution to the graphite surface through a turbulent electrolyte flow at the interface and a deep penetration of the electrolyte into the graphite felt. Finally, it enhances the degraded performance.

Liang et al. [88] prepared a novel (VO-EF) system consisting of a VO-based cathode and anode by electrodeposition. Different vanadium oxides on carbon paper (CP) were simultaneously used in an ensemble system for the degradation of levofloxacin under neutral pH conditions. A unique system consisted of a Vox@CP cathode and a V₃O₇@CP anode, each with dimensions of 2×1 cm². The benefit of the VO-EF system was demonstrated by its low energy consumption and H₂O₂ residual compared with the Ferrum-EF system under neutral pH conditions.

The main challenge faced by PEC reactors is the limited surface area available for coating electrodes with photocatalysts. Most previous studies have demonstrated that UV light outside electrochemical cells can irritate the electrodes coated with semiconductor photocatalysts [89].

Nevertheless, this approach encounters challenges related to the stability of the electrodes, which are caused by the leaching of the coatings and the loss of photon transport due to light reflection by the cell glass [90]. Internal light irradiation is favored and used when water is highly turbid or colored, preventing light from reaching the photoanodes in the wastewater medium to be treated [91].

Dos Santos et al. [61] constructed a pre-pilot flow reactor within a polycarbonate box measuring 24×24×25 cm³, comprising a 20 cm² BDD or Pt-air diffusion anode and a 20 cm² carbon/PTEF air-diffusion cathode. The scheme compared the performance of AO, PEF, and SPEF in degrading the dye. The electrode gap was 1.2 cm. For photoreactor assessment, a 160 W UV lamp (360 nm wavelength) was centered in the middle of an annular photoreactor, as shown in Fig. 2e. The photoreactor has the following dimensions (11 cm diameter and 21 cm height). Furthermore, the same scheme was previously used to mineralize the herbicide mecoprop [92].

Montenegro-Ayo et al. [90] innovated an annular photo-electrocatalytic reactor with multiple discs coated with TiO₂ nanotubes for the degradation of acetaminophen. A novel reactor has revealed a way to enhance the photoactive surface area in annular reactors, resulting in a reduction in electric energy compared to photocatalysis alone. This reactor involves eight-donut disk electrodes arranged in a perpendicular shape to 14 W of a UV lamp ($\lambda_{\text{max}} = 275$ nm) in the center of the reactor contained in a quartz tube, as shown in Fig. 2f. The photo-anode discs had a total diameter of 71 mm with a defined surface area of 32 cm² per side. In contrast, the cathode discs were 66 mm in diameter with a defined surface area of 30 cm² per side. The electrode configuration provided a uniform current density distribution, contributing to improved light radiation. This photoreactor design addresses the issues in light transport and could offer a new methodology for scaling PEC water treatment. This work proposes the design of a PEC reactor that should be optimized in the future based on enhancing light absorption, as the vertical orientation of the electrodes in the current study may limit light penetration and distribution.

Peralta-Hernández et al. [93] designed and fabricated a batch-recycle reactor to remove direct yellow-52 dye. The novel reactor consists of a cylindrical carbon cloth that serves as the outer cathode, with dimensions of 5.3 cm in diameter and 12 cm in length. A cylindrical Ti mesh electrode, with dimensions of 1 cm in diameter and 12 cm in length, is located within the inner electrochemical cell. The electrochemical cell had an outer diameter of 6.0 cm, which enabled the solution to flow on the exterior of the cathode cloth. The electrochemical experiments were conducted using a membrane with a thickness of 0.36 mm, made from porous polysulfonate, between the two electrodes. The purpose of the membrane was to avoid mixing between the electrodes. For photochemical investigations, a UV mercury lamp with a wavelength of 365 nm was inserted inside the hollow tube of a node electrode on its outer surface. The flow rate of solutions remained constant in the annular reactor through all

experiments at 100 ml/min. The treated solution in the reservoir was saturated with an oxygen sparger. Fig. 2g illustrates the proposed batch-recycle reactor.

De OS Santos et al. [94] tested the enhancement of the anode electrode material by electrodepositing mixed metal oxide (MMO) and BBD on the degradation of herbicide using EO, EF, and PEF. The Batch reactor contains (60 cm²) cylindrical carbon felt, which works as a cathode around the reactor's wall, and a rectangular anode with a dimension of (4 cm²) as shown in Fig. 2h. PEF experiments were performed using 9 W of UVC ($\lambda_{\text{max}} = 254$ nm) immersed in the solution. The results of this study demonstrated that modifying the anode with MMO improved TOC removal. The efficiency of the compared processes follows this sequence: PEF > EF > EO. Furthermore, the PEF-MMO process exhibited the lowest energy consumption among the methods studied.

Banuelos et al. [62] developed a novel design of a rotating disk slurry electrode (RoDSE) in a reactor, featuring an anode composed of a platinum spiral wire. The cathode was a Fe/AC rotating disk. The RoDSE method is a successful process for electrodeposition of iron on the AC. Additionally, Fe/AC can be prepared with relatively homogeneous iron oxide impregnated by applying the RoDSE technique, which will benefit the cathodic production of hydrogen peroxide and the electrocatalytic performance for EF and PEF. This work aimed to assess the effects of EF and PEF on the degradation and mineralization of the MO dye as a model effluent. PEF experiments were conducted using a UVA lamp ($\lambda_{\text{max}} = 365$ nm).

Carbon-based materials are the most common cathode materials used in PEF systems due to their ability to reduce oxygen. Different carbon-based materials, such as graphite, carbon felt, carbon cloth, carbon paper, and carbon-PTFE gas diffusion electrodes (GDEs), have shown a high ability for generating hydrogen peroxide [95]. Carbon felt cathodes (CFCs) offer a high specific surface area, electrical conductivity, and mechanical strength, along with certain advantages. Panizza and Oturan [96] reported that H₂O₂ concentrations of 28-35 mM could be obtained in carbon felt cathodes under optimized conditions (pH 3, 300 mA current density, and an airflow rate of 1 L/min). Although carbon felt electrodes frequently have a mass transfer limitation, their three-dimensional structure can limit the diffusion of oxygen towards the active sites. Carbon-PTFE gas diffusion electrodes have been shown to be better alternatives, offering improved oxygen diffusion properties and consequently higher H₂O₂ generation rates. These electrodes comprise a porous carbon film mixed with polytetrafluoroethylene (PTFE) as a binder, which is typically coated on a metal mesh current collector. A hydrophobic PTFE allows the establishment of a three-phase interface (solid electrode/liquid electrolyte/gaseous oxygen) that allows efficient oxygen reduction [97]. The study of Garcia-Segura et al. [98] reported H₂O₂ production rates between 0.42 and 0.58 mmol/h.cm² when carbon-PTFE GDEs were used, which was at least four times larger than that reported using carbon felt

electrode under the same conditions (0.10-0.15 mmol/h.cm²).

Internal illumination configurations position light sources within the reactor vessel, in direct contact with the solution or separated by transparent protective barriers. These configurations typically employ submersible light sources such as UV-C lamps, UV-LEDs, or fiber optic light distribution systems [99]. The primary advantage of internal illumination is enhanced light utilization efficiency, with minimized losses due to transmission through reactor walls or reflection at interfaces. Additionally, these configurations typically achieve uniform illumination of the photoreactive surfaces and solution volume. However, internal illumination systems face challenges, including potential fouling of light sources, complicated maintenance requirements, and the need for waterproof or protected light source designs [100]. Innovations in internal illumination of PEF reactors include optical photoanode designs, which distribute light throughout the reactor volume. Mousset and Dionysiou [101] reported that PEF reactors incorporating anodes coated with a TiO₂

photocatalyst achieved approximately 70% higher light utilization efficiency and 55% higher pollutant degradation rates compared to conventional external illumination systems of equivalent light source power.

In conclusion, the choice of anode material has a significant impact on the oxidation power of advanced oxidation processes. The most important characteristics in choosing electrodes were their high electrical conductivity, which minimizes ohmic losses and ensures consistent current distribution. Furthermore, efficient electrocatalysts' chemical and mechanical stability will reduce the overpotential required for the desired reaction. When utilizing solar energy as an illumination source, the vertical alignment of the electrodes could potentially restrict the penetration and even distribution of light to the lower electrodes. Conversely, in the case of UV lamps, the electrodes should maintain a minimal distance from the light source to optimize light penetration onto the electrode surface within PEF cells. Table 3 summarizes electrode materials and configurations used in novel electrochemical cells.

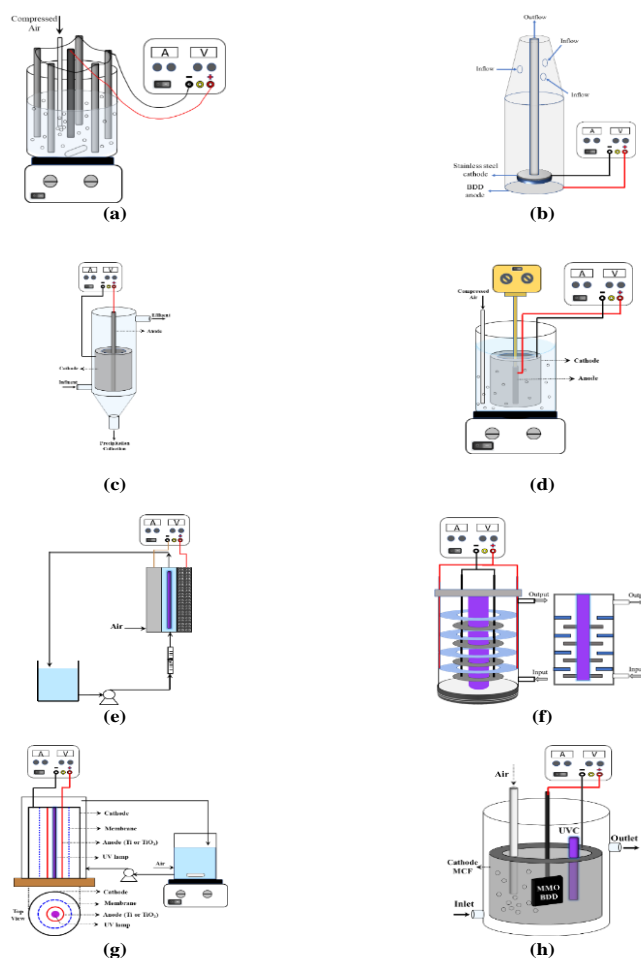


Fig. 2. Novel electrode configurations in electrochemical techniques, (a) McQuillan et al. [46], (b) Montenegro-Ayo et al. [85], (c) Lei et al. [86], (d) Abbas and Abbas [52], (e) Dos Santos et al. [61], (f) Montenegro-Ayo et al. [90], (g) Peralta-Hernández et al. [93], and (h) De OS Santos et al. [94]

Table 3. Summary of novel electrode configurations in electrochemical techniques

Process (s)	Pollutant (s)	Reactor Type	Material of electrodes		Shape of electrodes		Ref.
			Anode	Cathode	Anode	Cathode	
EF	Naphthalene	Batch	Carbon	graphite	Rod in the center	Five rods around the anode	[46]
ECO	ciprofloxacin	Batch	BDD	Stainless steel	Circular disk	Concave disk	[85]
EC	Phosphorus	Continuous	Pt/Ti	Stainless steel	rod	Tubular	[86]
EF	phenol	Batch	graphite	graphite	rod	Hollow cylinder	[52]
EF	Organic pollutants	Batch	Ti/IrO ₂ /RuO ₂ mesh	Graphite felt	rectangular	rectangular	[87]
EF	levofloxacin	Batch	Carbon paper	Carbon paper	rectangular	rectangular	[88]
EF PEF	Ponceau SS diazo dye	Batch	BDD/Pt	Carbon/PTFE-air diffusion	rectangular	rectangular	[61]
PEC	Acetaminophen	Continuous	TiO ₂ nanotube	Ti	donut-shaped discs	donut-shaped discs	[90]
EF PEF	Direct yellow-52	Batch-recycle	Ti mesh semiconductor	Carbon-cloth	cylinder	cylinder	[93]
EF PEF	clopyralid	Batch	MMO/BDD	Carbon felt	rectangular	cylinder	[94]
EF PEF	MO dye	Batch	platinum	Fe/AC	spiral wire	Rotating disk	[62]

PEC reactors play a crucial role in determining their performance for the degradation and mineralization of pollutants from wastewater. Reactor geometry directly influences key operational parameters, including light distribution, mass transfer efficiency, electrode spacing, and fluid flow dynamics, which significantly impact pollutant degradation rates. Literature studies have shown that optimized reactor shapes can enhance light absorption by up to 40% through improved light penetration and reduced shadowing effects [102]. Furthermore, strategic configuration designs that minimize dead zones and promote turbulent flow patterns have demonstrated increased mass transfer coefficients, facilitating faster photocatalyst interactions and reducing treatment times [103]. The arrangement of electrodes within the reactor also affects charge separation efficiency and electron transfer rates, with optimized electrode geometries showing reduced internal resistance and enhanced current densities for oxidation reactions [104]. Recent innovations in reactor design configurations, such as helical and spiral configurations, have improved the reactor performance by 40% compared to conventional rectangular designs. These new configurations maximize the active surface area while maintaining uniform light distribution [105].

4- Types of doping in photoanode synthesis

Developing an efficient method to fabricate photoelectrodes is crucial for advancing wastewater treatment. Photoelectrodes typically consist of semiconductor materials deposited on conductive substrates, with their performance highly dependent on the electronic properties of the semiconductor thin film. Doping semiconductor materials is a fundamental strategy to modify photoanode properties, enhancing charge carrier concentration and improving overall PEC reactor performance [106]. Doping processes are key in determining the physical and chemical properties of photoelectrodes, such as crystallinity, shape, band structure, and carrier transport properties [107].

Consequently, the choice of doping method is crucial for achieving the desired photoelectrode for specific applications. This review examines the primary doping methods employed in photoelectrode preparation, including sol-gel, dip-coating, electrophoretic deposition, and DC sputtering. Knowledge of these doping procedures, their capabilities, and their limitations is crucial for researchers seeking to tailor photoelectrodes to optimal performance and for any potential device application. Doping is a process used to alter the electrical properties of a semiconductor crystal structure by adding controlled amounts of impurity (dopant) atoms into it [108]. Two types of semiconductors exploited for the active layer can be obtained depending on the type of dopant: a) n-type semiconductors (having extra electrons) or b) p-type semiconductors (having missing electrons). The type and concentration of doping elements are well understood to control the charge carrier density, mobility, and recombination rate prerequisites for efficient photoelectrodes [109].

Sol-gel is a colloidal process that produces (open structure) inorganic materials by forming a gel in a solution. For doped photoelectrode fabrication, precursor solutions comprising semiconductor materials and dopant precursors are combined at the molecular level. The sol is hydrolyzed and condensed, forming a gel that is dried and annealed to produce the final doped semiconductor material. This method has several merits, such as a lower temperature process, low cost, adjustable components, and good homogeneity of the dopants [110]. However, the sol-gel suffers from potential cracking during drying, a lack of thickness control, poorer crystallinity compared to other methods, and difficulty obtaining high dopant concentrations [111]. Doped TiO₂, ZnO, Fe₂O₃, and other photoelectrodes can be fabricated using the sol-gel process. For instance, Zhang et al. [112] reported a rapid and cost-effective sol-gel route using polyethylene glycol (PEG) and polyacrylamide (PAM) as co-templates for synthesizing pure and doped mesoporous TiO₂ samples with improved photocatalytic performance.

In the dip-coating process, a substrate is dipped into a precursor solution that contains target semiconductor particles and dopant, after which it is pulled out of the solution at specified moving speeds [113]. During substrate retraction, a thin film solution remains on the surface and then solidifies through evaporation and heat treatment. The submersion process can be repeated several times until the required film thickness is formed. This process has several advantages, including simplicity and low cost, the ability to conformally coat complex features, reasonable control over film thickness from the withdrawal rate, and compatibility with various substrate materials [114]. However, this method still faces some problems, such as a lack of control over microstructure, possible inhomogeneous coating on complex surfaces, its dependence upon the wettability of the substrate, and the need for repeated coatings for thicker films [115]. Xia et al. [116] provided guidance for designing non-metal-doped BiVO₄ photoanodes by developing phosphorus-doped BiVO₄ (P-BiVO₄) photoanodes using dip-coating and thermal treatment. P-BiVO₄ demonstrated reduced interface obstruction compared to undoped BiVO₄. Additionally, P-BiVO₄ exhibited a higher carrier concentration, enhancing its PEC performance.

Electrophoretic deposition (EPD) is the migration of charged particles in suspension under an electric field from one electrode to another, followed by their subsequent deposition as a film [117]. In the preparation of doped photoelectrodes, suspensions of semiconductor particles and their dopant precursors are positioned under the influence of an electric field, which directs the materials to migrate to and deposit on the substrate electrode. This approach offers several benefits, including a rapid deposition rate, well-controlled film thickness, and suitability for complex geometry structures using room-temperature processes [118]. On the other hand, EPD has several limitations, including the requirement for conductive substrates, the need for post-deposition sintering, potential uneven deposition, and a limited particle size range [119]. In this context, self-doped strontium titanate (SrTiO₃) with favorable photocatalytic properties was fabricated by applying the electrophoretic deposition (EPD) method to produce SrTiO₃ thin films on fluorine-doped tin oxide (FTO) conductive substrates [120].

DC-sputtering is a physical vapor deposition technique where a target material (source) is bombarded with high-energy ions, causing atoms to be ejected and deposited onto a substrate [121]. For the preparation of doped photoelectrodes, either a pre-doped target can be used, or co-sputtering from multiple targets can be performed to achieve the desired doping level. This method has several advantages, such as precise control over film thickness and composition, excellent uniformity across large areas, high reproducibility, good adhesion to substrates, the possibility of graded doping profiles, and compatibility

with industrial-scale production [122]. However, several limitations should be considered, including the relatively high equipment cost, the potential for lattice damage due to high-energy deposition, lower deposition rates compared to previous techniques, and the requirement for a vacuum environment. Villamayor et al. [123] synthesized high photocatalytic activity layers by combining a TiO₂ nanostructured coating support with NiO layers using DC-sputtering. The photocatalytic activity of the coatings was evaluated through the degradation of methylene blue under UV light.

4.1. A comparative analysis of doping methods

Different doping methods offer varying levels of control over process parameters. This review examines four doping methods for synthesizing photoanodes: sol-gel, dip-coating, electrophoretic deposition (EPD), and direct current (DC) sputtering. According to the comparative review, it can be concluded that DC-sputtering is a notable method because it allows the film thickness, composition, and microstructure to be controlled relatively precisely by tuning the deposition parameters, including power, pressure, and substrate temperature. This degree of control is essential for the reproducible performance of the photoelectrode.

The effectiveness of doping is contingent upon the ability to control the distribution and concentration of dopants. Both sol-gel and dip coatings offer the advantages of good molecular-level blending, although phase separation may occur during crystallization. As opposed to this, DC sputtering allows for dendritic concentration by the target composition or the co-sputtering rates [124], leading to a larger uniform distribution of dopant atoms. Practical implementations need to be scalable. Sol-gel and dip-coating are standard techniques in laboratory research, and they are generally capable of being scaled up; however, they also have difficulty achieving considerable area uniformity. Although the initial cost of DC sputtering is higher, it is of great practical value, has high scalability, and is of reasonable quality for film, especially for industrial applications [125].

Cost aspects include equipment investments, material usage, energy needs, and processing time. Sol-gel and dip-coating are typically more cost-effective for small production quantities. However, in large-scale production, the DC-sputtering can be more economical [126]. When the equipment cost is not a concern, other deposition systems are not as effective as DC-sputtering equipment due to its high material utilization ratio and formation reproducibility, even though DC-sputtering equipment is more costly compared to the others. Table 4 presents a comparative analysis of the doping methods used to synthesize photoanodes.

Table 4. A comparative analysis of doping methods

Parameter	Sol-Gel	Dip-Coating	EPD	DC-Sputtering
Film uniformity	Moderate	Moderate	Moderate	Excellent
Thickness control	Limited	Moderate	Good	Excellent
Dopant distribution	Good	Moderate	Moderate	Excellent
Dopant concentration control	Moderate	Limited	Limited	Excellent
Crystallinity	Moderate	Moderate	Moderate	Excellent
Scalability	Good	Good	Moderate	Excellent
Equipment cost	Low	Low	Moderate	High
Processing time	Long	Moderate	Short	Moderate
Temperature requirements	High	High	Low	Low
Reproducibility	Moderate	Moderate	Moderate	Excellent

5- Operating parameters of hybrid Fenton processes

The studies on operation parameters that affect the Fenton reaction consist of the pH of wastewater, Fenton catalyst, H_2O_2 concentration, and concentration of organic pollutants, which are extensively focused on in this study to significantly impact the efficiency of Fenton processes in treating refractory organic pollutants. As a result, optimizing the parameters is necessary to achieve the best removal efficiency and minimize economic costs. This section provides an exhaustive literature survey and systematically describes the rules for optimizing the above parameters.

McQuillan et al. [46] assessed the effects of the EF pathway on naphthalene degradation. The influencing parameters on the removal efficiency were evaluated, namely Fe^{2+} concentration (0–2 mM) and current density (0–5 mA). The results reveal the absolute removal of a nearly saturated concentration of naphthalene (20 mg.L^{-1}) within less than 3 h of the reaction time under optimum conditions of operating factors (0.06 mM of iron concentration and 5 mA of time).

Banuelos et al. [62] examined the impact of iron concentration on electrodeposition for MO dye degradation and mineralization in the EF process. The higher electrodeposition iron content is beneficial for the production of H_2O_2 . A series of experiments was conducted to evaluate the degradation and mineralization of the MO dye, employing three different systems to demonstrate the effectiveness of the proposed EF process with iron electrodeposition on AC compared to other systems. The highest efficiency of color removal and total organic carbon (TOC) removal (98%) was observed for an iron concentration of 1973 mg/kg after 30 minutes.

Montenegro-Ayo et al. [85] conducted experiments in a new pilot reactor to investigate the degradation of the antibiotic pollutant from wastewater in a sulfate medium by varying operating variables, including current density, initial ciprofloxacin concentration, and pH. The operating parameters studied were current density (15–60 mA.cm^{-2}), initial ciprofloxacin (5–30 mg.L^{-1}), and pH (3–10) in the presence of 0.05M Na_2SO_4 as supporting electrolyte. Experimental work on COD and average current efficiency (ACE) is conducted. At optimal operating conditions, the maximum removal of COD and average current efficiency (ACE) were 94% and 14.3%, respectively.

Babuponnusami and Muthukumar [74] investigated the operational parameters (current density, H_2O_2

concentration, Fe^{2+} concentration, distance between anode and cathode, pH, and initial phenol concentration) that affect the electrochemical degradation of phenol in their study. The study findings indicated that the optimum operating conditions regarding phenol degradation and COD removal were obtained at 5 cm electrode distance, for the H_2O_2 concentration of 500 mg.L^{-1} , Fe^{2+} dosage of 4 mg.L^{-1} , initial pH of 3, and the current density of 12 mA.cm^{-2} .

Abbas and Abbas [52] studied the effect of Fe^{2+} concentration (0.05–0.4) mM, temperature (30–60) °C, current density (2–8) mA, and reaction time (1–6 h) on the phenol degradation. Maximum COD removal was achieved at optimum operating conditions with 6 h reaction time, current density of 8 mA.cm^{-2} , and Fe^{2+} concentration of 0.4 mM (88.01%). The maximal SPC was 10.22 kW/kg COD.

Liang et al. [88] developed a novel VO-EF system with a VOx@CP cathode and a $\text{V}_3\text{O}_7\text{@CP}$ anode for the degradation of levofloxacin under neutral pH conditions. Effect of pH, reaction time, and initial concentration of levofloxacin. In 600 min at pH 7.30, the VO-EF system could remove 19.19 mg/L TOC with a low electrical conductivity (EC) of 12.3 J/mg/L TOC. The H_2O_2 production rate in the reaction was calculated using the potassium titanium (IV) oxalate method.

Peralta-Hernández et al. [93] investigated the impact of operational factors, including solution flow rate, reaction time, and Fe^{2+} concentration, on the coupled process of EF and PEF for degrading organic pollutants. At the optimum operating conditions, the study showed that H_2O_2 generation increased by 30% in the PEF process compared to the EF process. Similarly, the TOC increased from 24 to 57% in EF and PEF, respectively.

Table 5 presents a summary of operating factors and responses for different AOPs, and Table 6 summarizes the optimal values of operating conditions. Research studies predominantly demonstrate improved outcomes when operating at a pH near 3. This phenomenon likely results from the breakdown of parasitic hydrogen peroxide into water and oxygen as pH levels increase. Additionally, some studies suggest that an acidic medium promotes iron leaching, potentially explaining the enhanced performance observed around pH 3 due to a higher presence of homogeneous reactions. Depending on operating conditions, the Fenton catalyst ($\text{Fe}_2\text{SO}_4 \cdot 7\text{H}_2\text{O}$) concentration in wastewater solution typically ranges from 0.3 to 0.6 mM. The supporting electrolyte (Na_2SO_4) in the test solution should be supplied at a concentration

of 0.05 M to provide the desired conductivity of 10,000 $\mu\text{S}\cdot\text{cm}^{-1}$ [127].

Table 5. Summary of operating parameters and responses for different processes

Process	Pollutant	Operating Conditions	Response	Ref.
EF	naphthalene	Iron concentration, current density	Removal eff.	[46]
EF	Phenol	Temperature, current density, Fe^{2+} concentration, reaction time	COD CE SPC	[52]
EF PEF	MO dye	Reaction time, Iron depositing concentration	Color removal TOC	[62]
F, EF, PEF, SEF	Phenol	Initial Conc., H_2O_2 Conc., Fe^{2+} Conc., Current density, pH, Distance between electrodes	COD EC CE	[74]
ECO	ciprofloxacin	Current density, Initial concentration of ciprofloxacin, and pH	CE ACE	[85]
EF	levofloxacin	pH, reaction time, initial concentration	TOC EC	[88]
EF PEF	Direct yellow-52 dye	Time, flow rate, concentration of Fe^{2+}	H_2O_2 generation TOC	[93]

Table 6. Summary of optimal operating conditions and responses

Pollutant	Optimum conditions	Responses	Ref.
Naphthalene	0.06 mM iron concentration, 5 mA current density	Removal Eff. 100%	[46]
Phenol	8mA/cm ² current density, 0.4 mM Fe^{2+} , 60°C temperature, 6 h reaction time	COD 88.01% CE 46.1% SPC 10.22 kW/kg COD	[52]
MO dye	30 min time, 1973 mg/kg iron concentration	Color 98% TOC 98%	[62]
Phenol	pH 3, 4 mg.L ⁻¹ Fe^{2+} , 500 mg.L ⁻¹ H_2O_2 , 12 mA/cm ² current density, 5 cm distance	COD _{PEF} 64.19% COD _{SEF} 67.93% EC 18.53 kW/kg COD CE 75.46%	[74]
Ciprofloxacin	60 mA/cm ² current density, 5 mg.L ⁻¹ initial ciprofloxacin and 10 pH	CE 94% AEC 14.3%	[85]
Levofloxacin	pH 7.3, 600 min time, 30 mg.L ⁻¹ initial concentration	TOC 80% EC 12.3 J.mg/l	[88]
Organic pollutants	25 min time, 100 ml.min ⁻¹ flow rate, 0.5 mM Fe^{2+}	H_2O_2 EF 70 ppm H_2O_2 PEF 100 ppm TOC _{EF} 24% TOC _{PEF} 57%	[93]

6- Recommendations for further work

- Using multi-electrode stacked reactors, constructed with materials resistant to harsh reaction conditions, reduces energy consumption and improves pollutant removal efficiency. However, in the case of solar energy as a light source, the vertical orientation of the electrodes may have limited light penetration and distribution at the lower electrodes. Cornejo et al. [128] recommended designing multicell stacks that enable the assembly of porous or plate BDD electrodes to enhance energy consumption and current efficiency.
- The electrode shape plays a crucial role in enhancing light irradiation on photoanode surfaces in PEF reactors. Walsh et al. [77] emphasized that surface decoration and modification of electrode supports can improve the active area and positively impact electrochemical cell performance.
- Consider the geometry and suitability of laboratory reactors for industrial applications in treating wastewater from industrial effluents. Peralta-Hernández et al. [93] proposed that the concentration of H_2O_2 depends on cell geometry and flow rate in the proposed reactor.

- Experiments on various pollutants over extended periods are essential to demonstrate the reactor's stability, efficiency, and effectiveness in treating industrial wastewater. Montenegro-Ayo et al. [90] suggested that investigating the scalability of the annular reactor design and its application in larger-scale water treatment systems would be valuable for practical implementation. In addition, they recommended conducting long-term operation studies to assess the stability and durability of the photo electrocatalytic reactor under continuous operation, and varying pollutant concentrations would provide insights into its real-world applicability.

7- Conclusion

The PEF process is highly efficient at degrading resistant pollutants in wastewater systems. The investigations detailed in this assessment show that the main deductions derived from this analysis are as follows:

- The fabrication of photoelectrodes with heterojunction composites by doping nanostructured semiconductor materials enhances the efficiency of

PEF reactors in degrading pollutants, especially in acidic media.

- Using photoanodes in a PEF cell can directly produce $\bullet\text{OH}$ radicals and oxidize pollutants. Similarly, using a carbon-based cathode can efficiently generate H_2O_2 , which participates in the Fenton reaction with Fe^{2+} to produce additional $\bullet\text{OH}$ radicals. UV irradiation enhances the process by photo-reducing Fe^{3+} back to Fe^{2+} and photolyzing H_2O_2 . This electrode combination effectively creates a synergistic system for PEF reactions.
- The vertical arrangement of electrodes in the PEF cells can limit the amount of solar radiation reaching and being dispersed across the lower electrodes. On the other hand, using a UV lamp will provide excellent light penetration into the surface of the electrodes in these cells.
- The electrode surfaces' smoothness, mirroring, and reflectivity significantly influence light acquisition capacity, directly impacting removal efficiency. Consequently, the most effective coating method for removing pollutants from wastewater.
- The performance of the PEF reactor in treating wastewater is significantly enhanced by using a UVC lamp instead of a UVA lamp. This improvement results in the production of a greater amount of oxidants during the PEF-UVC process.
- Internal illumination configurations of light sources within reactor vessels achieve uniform illumination of photoreactive surfaces. Studies have demonstrated enhanced removal efficiency with internal illumination compared to external systems of equivalent light source power.

A comparative analysis of electrode materials reveals that photoanodes fabricated by DC sputtering outperform those prepared by conventional methods in terms of degradation efficiency, light absorption, operational stability, and applicability for industrial wastewater treatment.

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التحديات والتوجهات المستقبلية في تقنيات فينتون-الكهروضوئية: مراجعة شاملة للتطبيقات والابتكارات الناشئة

إحسان حبيب داخل^{١،٢}، عمار صالح عباس^{١*}

^١ قسم الهندسة الكيميائية، كلية الهندسة، جامعة بغداد، بغداد، العراق

^٢ قسم الهندسة الكيميائية، كلية الهندسة، جامعة المثنى، السماوة، العراق

الخلاصة

يتناول هذا الاستعراض الشامل المبادئ الأساسية والتطبيقات العملية لمعالجة مياه الصرف الصحي الكهروكيميائية غير المتجانسة باستخدام تفاعلات فينتون. وقد تمت مراجعة المعادلات الأساسية المتضمنة توليد الجذور الحرة الهيدروكسيلية في عمليتي فينتون الكهربائية والفوتوفينتون الكهربائية. وقد أثبتت عمليات الفوتوفينتون الكهربائية أنها من أكثر الطرق فعالية في تعدين وتحلل الملوثات في مياه الصرف. وينصب التركيز الرئيسي على فهم قيود عمليات فينتون الهجينة واقتراح طرق فعالة لمواجهة هذه التحديات. بالإضافة إلى ذلك، قُيِّمت الدراسة أهمية تطوير تكوين الأقطاب الكهربائية وتعزيز احتراق الضوء في زيادة إنتاج بيروكسيد الهيدروجين وزيادة توليد جذور الهيدروكسيل. تساهم هذه التطورات في تحسين تحلل وتمعدن الملوثات في مياه الصرف الصحي الجوفية. ويُظهر تحليل مقارن لمواد الأقطاب الكهربائية، وتكوينات المفاعلات المبتكرة، وظروف التشغيل، العلاقة بين طرق التحضير وكفاءة المعالجة. كما تم تحديد الثغرات البحثية لتحسين عملية الفوتوفينتون الكهربائية، مع اقتراحات للأعمال المستقبلية. أظهرت الدراسات أن الإشعاع الضوئي الداخلي يؤدي إلى كفاءة إزالة أعلى مقارنةً بأنظمة الإضاءة الخارجية بنفس قوة مصدر الضوء. وكانت أهم التوصيات استخدام مفاعلات متعددة الأقطاب الكهربائية لتقليل استهلاك الطاقة، وتعزيز كفاءة التيار، كما يلعب شكل الأقطاب الكهربائية دورًا حيويًا في زيادة تعرض أسطح الأقطاب الضوئية لأشعة الضوء، وإجراء تجارب طويلة الأمد على ملوثات مختلفة لإثبات استقرار المفاعل وكفاءته وفعاليته في معالجة مياه الصرف الصناعي.

الكلمات الدالة: ترتيبات الأقطاب الكهربائية، المفاعلات المبتكرة، عمليات فنتون الهجينة، طرق الطلاء، تحديات فينتون.