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Review on treating refractory, organics-laden wastewater using three-dimensional electrochemical reactor

Xu Ren[a,c,*], Peixin Tang[a,b,c,†], Linghan Xu[a], Qiaoyun Zhang[a], Kai Song[a], Zhicheng Pan[b,c,**]

[a]*Sichuan City Solid Waste Energy and Building Materials Conversion Technology Engineering Research Center, Chengdu University, Chengdu No. 2025, Chengluo Road, Chengdu, 610106, China, Tel. +86 13438275082; emails: renxu@cdu.edu.cn (X. Ren), 2402180131@qq.com (L. Xu), 1755996734@qq.com (Q. Zhang), songkailw@163.com (K. Song)*
[b]*College of Chemistry and Chemical Engineering, Southwest Petroleum University, Chengdu 610500, Sichuan, China, email: 675498447@qq.com (P. Tang)*
[c]*Postdoctoral Research Station of Haitian Water Group Co., Ltd., Chengdu 610213, Sichuan, China, Tel. +86 13882188111; email: 957668816@qq.com (Z. Pan)*

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ABSTRACT

Electrochemical advanced oxidation processes (EAOPs) can effectively eliminate organic pollutants using the reactive oxygen species (ROS) produced by electrode reactions. Three-dimensional electrochemical reactors (3DERs) are the research frontier of EAOPs. They have higher catalytic activity and lower energy consumption than conventional electrochemical reactors. This mini-review on 3DERs includes their structures, reaction mechanisms, and applications in treating refractory organic wastewater. Developing the preparation techniques for highly efficient particle electrodes (PEs), the core of 3DER, is essential to constructing a high-performance 3DER. This study introduces the characteristics and main preparation methods of carbon-based, metal-based, and composite PEs materials in detail, the applications in refractory organic wastewater treatment are summarized, and the research direction of 3DER in the future is prospected. Our findings could aid new approaches to constructing more efficient 3DERs.

Keywords: Advanced oxidation processes (AOPs); Electrochemical technology; Three-dimensional electrochemical reactor (3DER); Particle electrodes (PEs); Refractory organic wastewater

1. Introduction

The production of refractory organic wastewater, such as landfill leachate, oily wastewater, coking wastewater, pharmaceutical wastewater, and dye wastewater, increases rapidly with the incessant societal development [1,2]. Because refractory organic wastewaters contain numerous highly toxic pollutants, including persistent organic pollutants (POPs) and toxic metals, efficient treatment technology for its control is crucial in environmental science and engineering [3,4]. Biotechnologies, such as anaerobic and aerobic biotechnology, are the most commonly used treatment methods for organic wastewater due to the methods' relatively low cost. However, their removal efficiency toward refractory organics is minimal [5].

In the past 20 y, electrochemical advanced oxidation processes (EAOPs) have been widely used to treat high salinity or refractory organic wastewaters [4,6–8]. Pollutants in wastewater are removed by direct and/or indirect oxidation in electrochemical systems [9,10]. Nevertheless, conventional EAOPs (i.e., 2DER) still have some defects, such as relatively low current efficiency and small treatment capacity, limiting their applications [11]. These limitations are solved by three-dimensional electrochemical reactor (3DER) technology. Compared with conventional electrochemical systems, 3DER increases the electrode reaction area, providing

* Corresponding author.
** Co-corresponding author.
† Joint first author.

more reaction sites and mass transfer efficiency, thereby improving the system's efficiency. Hence, removing the organics improves significantly [12–15].

Herein, we provide a comprehensive summary of the 3DER, including its structure, organics removal mechanism, and the characteristics of carbon-based, metal-based, and composite particle electrodes (PEs) materials. Moreover, applying 3DER to treat refractory organic wastewaters is elucidated.

2. Characteristics of 3DER

2.1. Definition

The 2DER is a conventional electrochemical reactor with a pair of parallel anode and cathode (i.e., 2D electrodes). Similarly, 3DER was established based on 2DER by filling conductive particles between the parallel electrodes. The conductive particles are called PEs, third electrodes, or bed electrodes. In the electric field, PEs are polarized to form several micro-electrodes with different charges at both ends. This process illustrates that electrochemical reactions take place on the electrode surfaces and at the surface of PE ends [16]. Therefore, the mass transfer distance in the reactor is significantly reduced [17]. As a result, contaminants can be removed on the surfaces of 2D electrodes and PEs. Hence, the pollutant removal efficiency of 3DER is much higher than 2DER.

2.2. Mechanism

The most significant advantages of 3DER in refractory wastewater treatment are (i) the high pollutants removal efficiency with relatively low cost, and (ii) stable and simple operation. Refractory organic pollutants are primarily removed by direct and indirect oxidation (Fig. 1). In some cases, adsorption and coagulation contribute to the removal process. Meanwhile, the reduction at the cathode is also relevant to removing pollutants, such as nitrates, and toxic metals (or trace metals).

2.2.1. Direct and indirect oxidation

Like a conventional electrochemical system, the electrooxidation (EO) in 3DER can be categorized into direct and indirect oxidation [18]. Direct oxidation refers to the process whereby pollutants are oxidized on the anode surface through electron transfer. Indirect oxidation is one where reactions at the electrodes promote the production of radical reactive oxygen species (ROS) (e.g., $^{\bullet}OH$, $SO_4^{\bullet-}$, $^{\bullet}Cl$) and non-radical ROS (e.g., ClO^- and Cl_2) in the system. These ROS can oxidize and remove the contaminants in the electrolytes.

The mechanism of direct oxidation mainly depends on the anode materials, which could be active or inactive. Under an electrical current, OH^- and H_2O lose electrons, combining with the metals on the anodes to form M($^{\bullet}OH$) [Eqs. (1) and (2)]. Many active sites are present on the surface of an active electrode, capable of combining with M($^{\bullet}OH$) to form MO with higher oxidation ability [Eq. (3)]. Subsequently, MO could oxidize the organics [Eq. (4)], and the oxygen evolution reaction converts MO to M simultaneously [Eq. (5)]. For the inactive anode, M($^{\bullet}OH$) directly reacts with organics [Eq. (6)], and the oxygen evolution reaction also occurs on the anode surface [Eq. (7)] [19,20].

$$M + H_2O \rightarrow M\left(^{\bullet}OH\right) + H^+ + e^- \quad (1)$$

$$M + OH^- \rightarrow M\left(^{\bullet}OH\right) + e^- \quad (2)$$

$$M\left(^{\bullet}OH\right) \rightarrow MO + H^+ + e^- \quad (3)$$

$$MO + R \rightarrow M + RO \quad (4)$$

$$MO \rightarrow M + 1/2O_2 \quad (5)$$

$$M\left(^{\bullet}OH\right) + R \rightarrow M + mCO_2 + nH_2O + H^+ + e^- \quad (6)$$

$$M\left(^{\bullet}OH\right) \rightarrow M + 1/2O_2 + H^+ + e^- \quad (7)$$

The indirect oxidation mechanism is relatively complicated. Some inorganic ions can be converted into radical and/or non-radical ROS [Eqs. (8)–(17)] with high redox potential under electrical current [21–23]. Meanwhile, the current can promote the circulation between the high and low valence metals (such as Fe(IV)/Fe(III)/Fe(II), Ni(III)/Ni(II), and Co(III)/Co(II)), so that organics can be removed continuously [18]. Additionally, the cathode, such as graphite felt, can produce hydrogen peroxide (H_2O_2) *in-situ* [Eq. (18)],

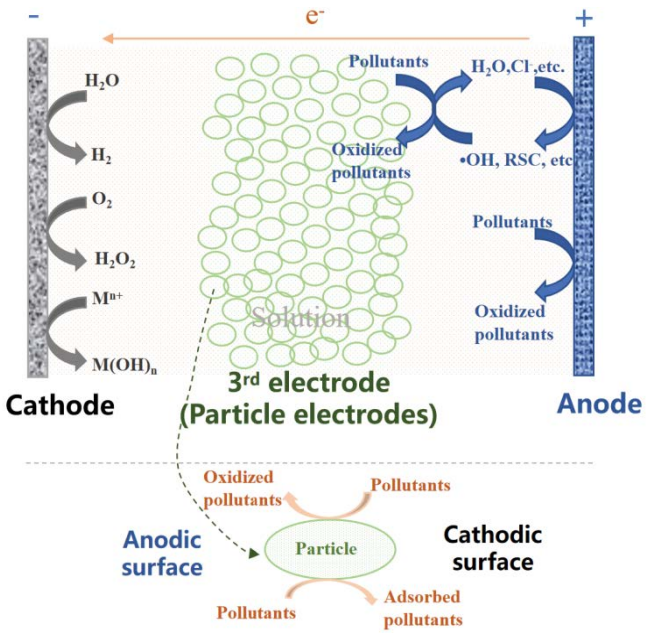

Fig. 1. The main electrochemical reaction in 3DER.

which can be decomposed by metal catalysts (such as Fe(II)) in the system to form •OH [Eq. (19)] [7].

$$Cl^- \xrightarrow{\text{current activation}} {}^{\bullet}Cl \quad (8)$$

$${}^{\bullet}Cl + Cl^- \rightarrow Cl_2^{\bullet-} \quad (9)$$

$$Cl_2^{\bullet-} + H_2O \rightarrow HClO + Cl^- + H^+ + e^- \quad (10)$$

$$2Cl^- \rightarrow Cl_2 + 2e^- \quad (11)$$

$$Cl_2 + H_2O \leftrightarrow 2H^+ + ClO^- + Cl^- \quad (12)$$

$$\left(S_2O_8^{2-} / SO_4^{2-}\right) \xrightarrow{\text{current activation}} SO_4^{-\bullet} \quad (13)$$

$$SO_4^{-\bullet} + OH^- \rightarrow SO_4^{2-} + {}^{\bullet}OH \quad (14)$$

$$SO_4^{-\bullet} + H_2O \rightarrow SO_4^{2-} + {}^{\bullet}OH + H^+ \quad (15)$$

$$SO_4^{-\bullet} + SO_4^{-\bullet} \rightarrow S_2O_8^{2-} \quad (16)$$

$$S_2O_8^{2-} + e^- \rightarrow SO_4^{-\bullet} + SO_4^{2-} \quad (17)$$

$$O_2 + 2H^+ + 2e^- \rightarrow H_2O_2 \quad (18)$$

$$H_2O_2 + Fe^{2+} + H^+ \rightarrow Fe^{3+} + H_2O + {}^{\bullet}OH \quad (19)$$

2.2.2. Adsorption and coagulation

The specific surface area and porosity of PEs are generally large, allowing the removal of contaminants by physical adsorption. Under the optimal conditions, positive and negative charges converge simultaneously at each end of the particles to form anodic and cathodic surfaces, respectively. The ions in the solution migrate toward the surface with an opposite charge under Coulomb force, allowing the pollutants to adsorb on the particles [24–27]. In some cases, the combination of physical and electro-sorption can effectively remove organics [12].

Coagulation or electrocoagulation exists in electrochemical reactors using iron-based and/or aluminum-based PEs. These electrodes are continuously consumed to produce *in-situ* Al(III), Fe(II), or Fe(III) ions under electrical current. At the same time, H_2O is incessantly reduced to gaseous hydrogen and OH^- at the cathode (or cathodic surface on PEs). Hence, $Al_n(OH)_m^{(3n-m)+}$ ($n > 1$, $m \leq 3n$) or $Fe(OH)_n$ ($n = 2,3$) is formed in solutions and finally polymerize to colloids with a positive charge. This occurrence allows the removal of contaminants through coagulation or electrocoagulation [28,29].

2.2.3. Reduction process on the cathode

Removing contaminants (such as NO_x^-–N, metals, fluorides, and chlorinated organics) through cathodic reduction requires some attention [30]. For instance, NO_x–N can be reduced to nitrogen gas or ammonia nitrogen [31], while metal ions are deposited *in-situ* on the cathode or form hydroxide ($M(OH)_n$) near or on the cathode surface under alkaline conditions [30]. Therefore, the cleaning of the cathode surface is vital to the performance of an electrochemical reactor. However, in some cases, chlorinated organics can be removed directly by cathodic reduction or indirectly by catalytic hydrogenation [32].

2.3. Classification and characteristics of 3DER

According to the filling way of PEs, 3DER can be classified into fixed-bed and fluidized-bed 3DER. Fixed-bed 3DER (also called a PEs bed) refers to the fixing PEs in a container located between the anode and cathode. In contrast, the PEs in a fluidized-bed 3DER can move freely [18]. The characteristics of the two 3DER types are summarized in Table 1.

2.3.1. Fixed-bed 3DER

Fixed-bed 3DER is a reactor in which the PEs are fixed inside the reaction tank. Compared with the fluidized-bed 3DER, the fixed-bed 3DER is much easier to operate, and its PEs have a longer lifetime. However, the particles agglomerate more easily, and the reactor is prone to short circuits. Therefore, fixed-bed 3DER has relatively poor stability. Generally, fixed-bed 3DER can be divided into two types

Table 1
Characteristics of fixed-bed 3DER and fluidized-bed 3DER

Items	Fixed-bed 3DER	Fluidized-bed 3DER
Characteristics	Fixed PEs in the container(s)	PEs is free to move
Advantages	Relatively easy to operation	High mass and heat transfer
	Long PEs lifetime	Relative stable
Disadvantages	Prone to short circuit	Difficult to study the mechanism
	Particles are easy to agglomerate	of pollutant removal
	Relatively poor stability	Relatively high-cost

according to the fluid flow patterns, which are continuous flow reactor (Fig. 2) and intermittent flow rector (Fig. 3).

The continuous flow reactor can be divided into three types, (a) horizontal-flow reactor (Fig. 2a), refers to a reactor in which fluid flows horizontally through the PE bed; (b) vertical-flow reactor (Fig. 2b), refers to a reactor where the wastewater flows vertically through the PE bed; and (c) tube type (Fig. 2c), composed of several hollow tubes in parallel, with the particles arranged on the surface of the tubes, fluid heat carrier (such as water) flows through the tube to heat or cool the electrolytes. Notably, there is scarcely any heat exchange between (a), (b), and outside. As for intermittent flow rector, a certain volume of electrolytes electrolyte is introduced into the reactor until the reaction is completed and then discharged them. Mechanical stirring or aeration is used to achieve homogenization rather than liquid flow.

2.3.2. Fluidized-bed 3DER

The particle sizes in fluidized-bed 3DER are much smaller than those in fixed-bed 3DER and are usually powdery or even nanoscale. Fluid flow and mechanical agitation are usually used to homogenize and suspend the particles in the electrolyte. Therefore, fluid always flows from the bottom to the top of the reactor to negate particulate deposition by gravity [33].

According to fluid flow patterns, fluidized-bed 3DER can be divided into two types: (i) continuous flow reactor (Fig. 4a), which has continuous influent and effluent, and (ii) intermittent flow reactor (Fig. 4b) has discontinuous influent and effluent. Fluidized-bed 3DER usually applies mechanical stirring and/or aeration to achieve electrolyte homogenization.

The mass (i.e., contaminates) and energy (i.e., heat) in fluidized-bed 3DER are relatively evenly distributed [34]. The particulate surfaces can make full contact with the electrolytes, making particle agglomeration tedious. Hence, the system is more stable, exhibiting a higher mass transfer efficiency than the fixed-bed 3DER [35,36]. However, the current studies focus on improving the conductivity of PEs in fluidized-bed reactors [33]. Moreover, the randomness in particle movement in the system, resulting in the pollutant mechanism, is also extremely complicated and should be further studied [34,36].

2.4. Particle electrodes

The electrodes are the core component of an electrochemical reactor, while PEs are the most crucial part of 3DER. PEs consist of many particles with conductive and (or) catalytic abilities [13]. These particles significantly shorten the mass transfer distance and increase the system's reaction area, thus, improving the pollutant removal efficiency significantly [37–39]. Previous studies have shown that preeminent PEs in 3DER should be porous materials with excellent conductivity and catalytic activity, relatively large specific surface area, and stable physical and chemical properties. Several materials, including carbon-based materials (e.g., activated carbon (AC) and carbon aerogels), metal-based materials (e.g., metal and metal oxide), and minerals (e.g., kaolin and zeolite), have been studied as PEs to make 3DER. In the Table 2, we instructed the main preparation methods of typical PEs and summarized the characteristics of different types of PEs in Table 3.

2.4.1. Carbon-based materials

The major merit of carbon-based materials is their superior conductivity, including granular activated carbon (GAC) [40,41], carbon nanomaterials [42], carbon aerogels [43], flake graphite [44], biochar [45], etc., are the most widely used and studied materials for preparing PEs. The carbon-based PEs have the advantage of large specific surface area, low cost, and easy manufacturing process. The large specific surface area is usually accompanied by a developed porosity and abundant oxygen-containing surface functional groups, which aid adsorption and electrosorption of pollutants [46,47]. However, they are prone to short-circuit and exhibit poor electro-oxidation capacity.

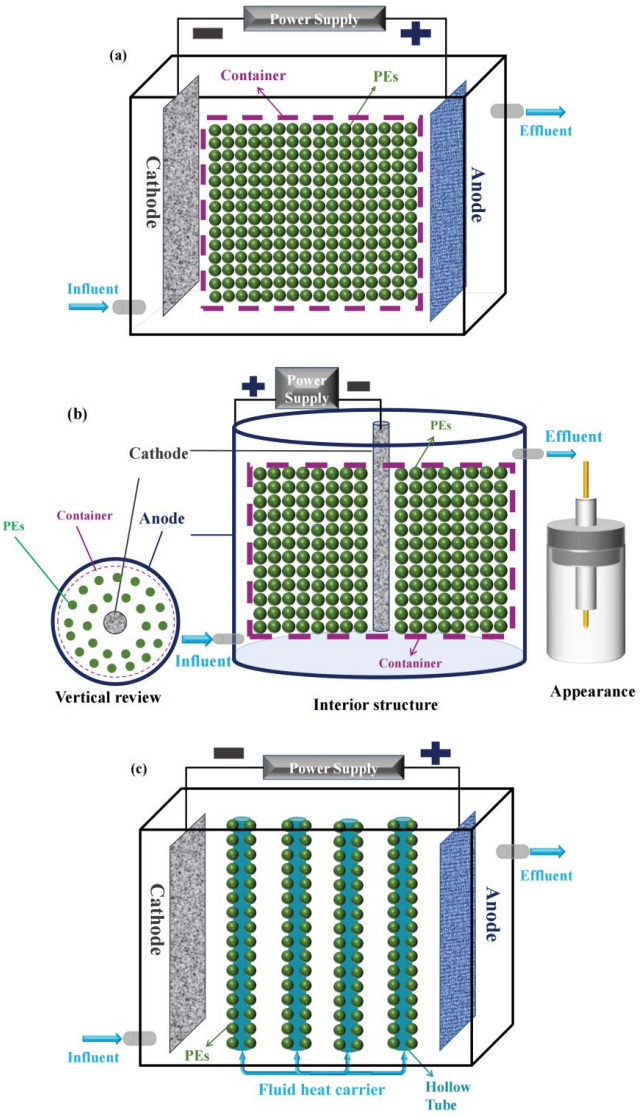

Fig. 2. The continuous flow fixed-bed 3DER. (a) Horizontal flow reactor, (b) vertical flow reactor and (c) tube-type reactor.

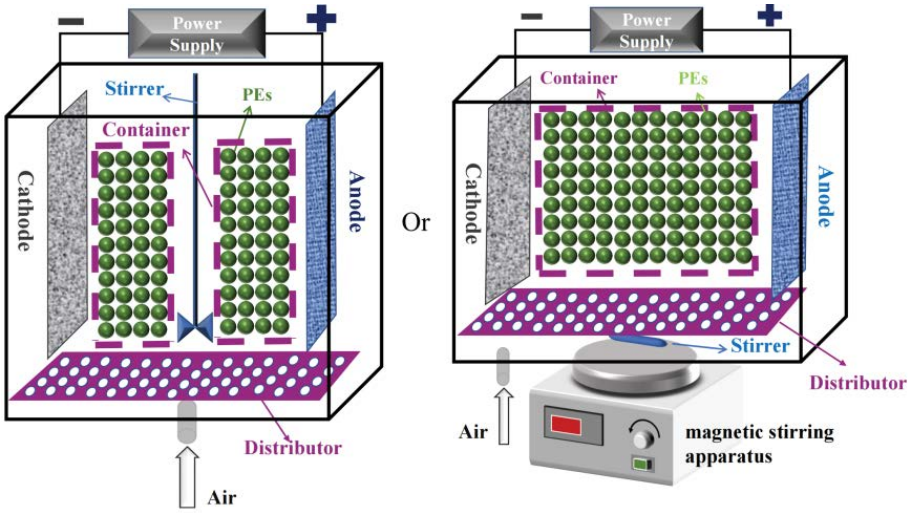

Fig. 3. The intermittent flow fixed-bed 3DER.

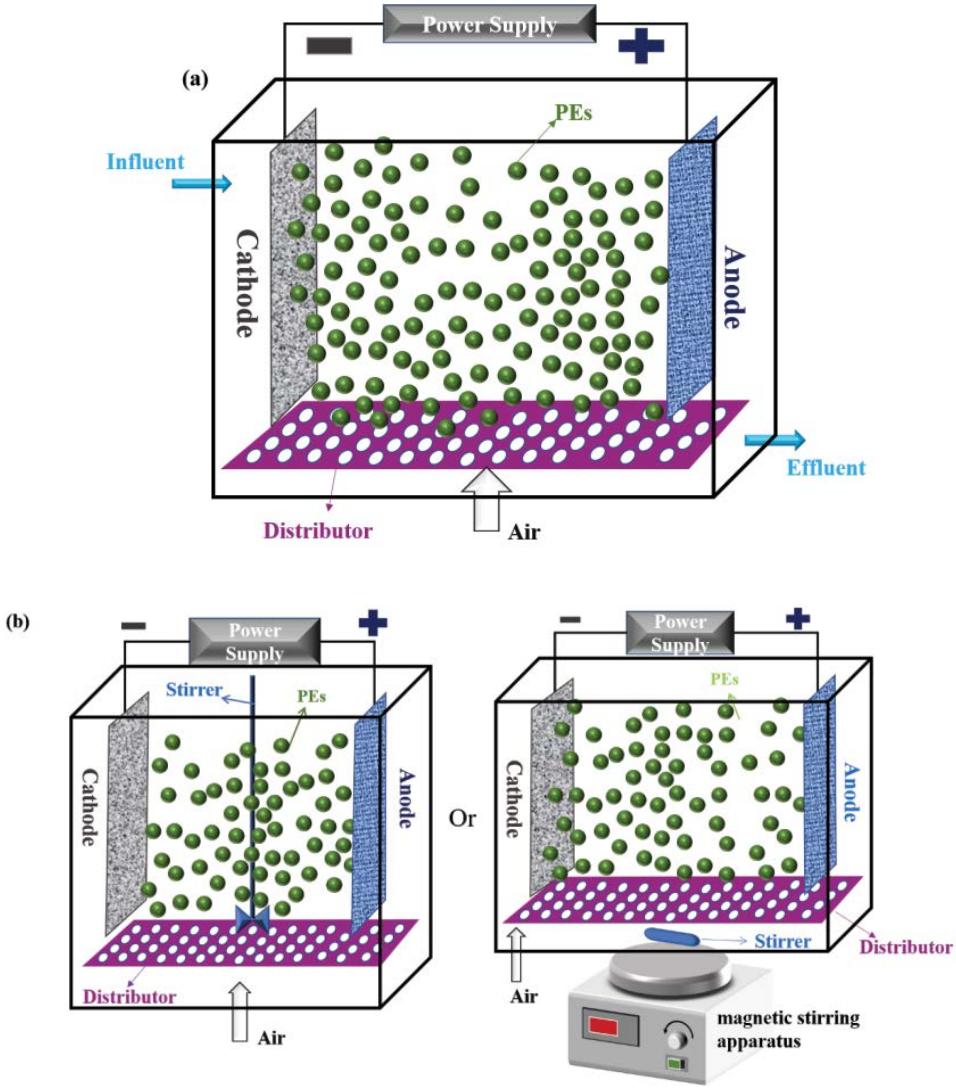

Fig. 4. The fluidized-bed 3DER. (a) Continuous flow reactor and (b) intermittent flow reactor.

Table 2
Main preparation methods of PEs

Categories	PEs	Preparation method	Main raw material	References
Carbon-based materials	GAC	Purchase	GAC	Cho et al. [40]
	GAC	Purchase	GAC	Pedersen et al. [41]
	Modified multi-walled carbon nanotube (MWCNT)	Mixed heating in anhydrous ethylene glycol	MWCNT powder Bismuth pentahydrate Ethylene glycol	Dighole et al. [42]
	Graphene aerogel	Hummer's method Hydrothermal synthesis method	Graphite powder Ethylenediamine	Chen et al. [43]
	Flake graphite	Sol–gel method	Flake graphite $Pr(NO_3)_3 \cdot 6H_2O$ $Co(NO_3)_2 \cdot 6H_2O$ Titanium tetra-n-butoxide Ethanol	Yu et al. [44]
	Granular sludge carbon	High temperature sintering	Sewage sludge $FeSO_4$ $Ca(OH)_2$ $ZnCl_2$ Sodium carboxymethyl cellulose	Ji et al. [45]
	Slag	High temperature sintering	Steel slag Zeolite SnO_2 MnO_2 Starch	Zhang et al. [48]
	Tailing	High temperature sintering	Flotation tailings Soluble starch Garden soil	Yang et al. [49]
Metal-based materials	$CuFe_2O_4$	Sol–gel combustion method	$Cu(NO_3)_2 \cdot 3H_2O$ $Fe(NO_3)_3 \cdot 9H_2O$ Citric acid	Li et al. [50]
	Fe_3O_4-based particle	Two-step hydrothermal method	$FeCl_3 \cdot 6H_2O$ CH_3COONa Natural flake graphite Ethylene glycol Polyethylene glycol	Zhou et al. [51]
Composite materials	Conductive polyurethane/polypyrrole/graphene	Solution blending *In-situ* oxidative polymerization	Graphene Sodium dodecyl benzene sulfonate Polyurethane Citric acid Polypyrrole	Guo et al. [56]
	FeC@nitrogen doped-graphene-iron oxide	High temperature sintering	Ferric chloride $Na_4Fe(CN)_6 \cdot 10H_2O$	Ghanbarlou et al. [57]
	AgPd nanoparticles supported on cyclodextrin polymers	Self-assemble strategy	5,5-dimethyl-1-pyrrolidine N-oxide Na_2PdCl_4 (3-Aminopropyl)trimethoxysilane β-cyclodextrin	Guo et al. [58]

Table 3
Characteristics of different PEs

Categories	Advantages	Disadvantages
Carbon-based materials	Excellent adsorption performance Low-cost Easy manufacture	Easy to short-circuit Poor catalytic activity
Metal-based materials	Good electrical conductivity Low-cost Have many catalytic active sites	Poor adsorption Difficult to fluidize
Composite materials	High stability Excellent catalytic performance	High-cost Complex manufacture

Table 4
Application of 3DER in treating some organic wastewaters

Wastewater	Main pollutant	2D electrodes	PEs	Operating conditions	Removal rate	References
Leachate	Humic acid, fulvic acid and other refractory organics	Ru/Ir coated titanium plate	Fe/C particles	U = 5 V PE = 1 g $Na_2S_2O_8$ = 5 mM	COD = 72.9% NH_4^+–N = 99.9%	Yu et al. [60]
		Stainless steel sheet, graphite	Columnar AC	J = 30 mA/cm^2 Electrode spacing = 6 cm Fe^{2+} = 1.0 mM Initial pH = 3.0	COD = 96.2% NH_4^+–N = 94.3% TP = 99.2% Chroma = 93.6%	Lu [61]
Oily wastewater	Refractory organics, petroleum and so on	Pt electrode, stainless steel	Magnetic steel slag	U = 20 V PE = 75 g HRT = 2 h	TOC = 85%	Wang et al. [62]
		Ir/Ru coated titanium mech	Rod AC, glass beads, quartz sand insulating particles	I = 6 Å Electrode spacing = 40–60 mm V = 4 L	COD (in 15 min) = 55% COD (15–35 min) = 35.8%	Yin et al. [63]
		Porous graphite plate	Iron particle	U = 12 V Initial pH = 6.5	COD = 92.8%	Yan et al. [64]
		$Ti/SnO_2+Sb_2O_3$ DSA type anodes, 304 stainless steel mesh plate	GAC, porous ceramsite particle	J = 30 mA/cm^2 PE = 75% HRT = 100 min	COD = 45.5 TOC = 43.3% Toxic unit = 67.2%	Wei et al. [65]
		Stainless steel, graphite	Metal loaded slag	U = 6 V PE = 15 g V = 200 mL	COD = 88.23%	Liu et al. [66]
Coking wastewater	Toxic inorganics (ammonia nitrogen, cyanide, sulfide, fluoride, etc.), refractory organics (phenol, polycyclic aromatic hydrocarbons, oxygenated heterocyclics, nitrogen containing heterocyclic, etc.)	Ti plate coated with Ru/Ir, Ti plate	Ti-Sn-Ce/BC	J = 30 mA/cm^2 HRT = 150 min	COD = 92.91% DOC = 74.66%	Zhang et al. [67]
		Ti/RuO_2-IrO_2 plates	Metal oxide load GAC	Electrode spacing = 28 cm HRT = 3.53 h Aeration		Liu et al. [68]
		Ti/RuO_2-IrO_2 electrodes	Metal oxide load GAC, AC	I = 150 mA (3DERs) I = 20 mA (3DBERS) CH_3COONa = 0.4 g/L	TN = 70.7% COD = 55.8%	Wu et al. [69]
		Ti/RuO_2-IrO_2 electrodes	Metal oxide load GAC, GAC	U = 8 V 1,000 Hz (3DERs) U = 3 V (3DBERS) I = 10–20 mA (3DBERS) Aeration = 100 L/h	TN = 76.30% COD = 79.63%	Wu et al. [70]

(Continued)

Table 4

Wastewater	Main pollutant	2D electrodes	PEs	Operating conditions	Removal rate	References
Coking wastewater		Ti/RuO_2-IrO_2 electrodes	Metal oxide load GAC	I = 150 mA HRT = 1 h Aeration = 100 L/h	COD = 73.21% NH_4^+–N = 38.2% NO_3^-–N = 91.46%	Wu et al. [71]
Pharmaceutical wastewater	Amoxicillin	Ti/RuO_2 electrodes	GAC	J = 5 mA/cm^2 NaCl = 17 mM Initial pH = 5.56 GAC/quartz sand = 9:1	AMX = 98.8% TOC = 47.6%	Shi et al. [72]
	Antibiotic pharmaceutical	RuO_2/IrO_2 coated Ti plate, stainless steel plate	GAC	Electrode spacing = 3.5 cm PE = 100 g HRT = 6 h O_2 = 0.4 L/min	TOC = 71% Bacterial inhibition rate < 70%	Zhan et al. [73]
	Norfloxacin	Ti/RuO_2/IrO_2 mesh, graphite felt	Sulfur-zinc modified kaolin/steel slag	U = 4 V pH = 3–10	NOR = 100% (acidic) NOR > 90% (neutral) NOR > 80% (alkalinity)	Song et al. [74]
	Antipyrine	Ti mesh coated with SNO_2-TA_2O_5-IRO_2, Ti mesh	Modification Sn-Sb-Bi/γ-Al_2O_3	Na_2SO_4 = 0.1 M HRT = 4 h pH = 7	Antipyrine =94.4%	Liu et al. [75]
	Rhodamine B (RhB)	Stainless steel plate, Ti plate	Electro-biological particle electrode made from steel converter slag	I = 1.00 Å Na_2SO_4 = 0.1 M T = 20°C Aeration = 2.5 L/min Initial pH = 5.86 DO = 3.25 mg/L	RhB = 91.68% COD = 87.63% NH_4^+–N = 90.54%	Feng et al. [76]
Dye wastewater		Ti/RuO_2-IrO_2-TiO_2-SnO_2 electrode, stainless steel plate	GAC coated with SnO_2-Sb doped TiO_2	J = 60 mA/cm^2 Electrode spacing = 3 cm HRT = 3 min Initial pH = 7 Bed height = 8 cm	COD = 60–70%	Li et al. [77]
	Methylene blue (MB)	Ti/RuO_2/IrO_2, graphite rod	Kaolin/slag	U = 11 V Electrode spacing = 4 cm Na_2SO_4 = 0.1 M HRT = 90 min T = 26°C pH = 3.0	MB = 87.05%	Song et al. [78]
		Pb/PbO_2, stainless steel	GAC	J = 23 mA/cm^2 Electrode spacing = 3 cm HRT = 60 min pH = 7.6	MB = 97.4% COD = 87.5% TOC = 83.0%	Shokoohi et al. [79]
	Turquoise blue dye (BT)	Flat Al electrodes	Aluminum microbipolar electrode	J = 180 A/m^2 NaCl = 1 g/L 10 mg dye/g Al particle Rotation speed = 300 rfm	BT = 96%	A. Bakalem et al. [80]
Other	Reverse osmosis concentrates	Ti/IrO_2-RuO_2, stainless steel	γ-Al_2O_3/Sn-Sb oxide	I =600 mA HRT > 2 h	COD = 100%	Liu et al. [81]
	4-Chlorophenol	DSA plate, Ti plate	Biochar-loaded PEs	I = 1 Å Electrode spacing = 3 cm PE = 5 g Na_2SO_4 = 2 g/L	4-Chlorophenol = 99.93%	Xie et al. [82]

(Continued)

Table 4

Wastewater	Main pollutant	2D electrodes	PEs	Operating conditions	Removal rate	References
Other	Wood vinegar wastewater	Stainless steel, graphite	AC and iron powder mixture	U = 5 V HRT = 60 min	COD = 70.8%	Fan et al. [83]
	Atrazine	Ti/RuO_2-IrO_2, stainless steel	$CuFe_2O_4$ magnetite nanoparticle	J = 4 mA/cm^2 PS = 4.0 mM PE = 3 g/L HRT = 35 min Initial pH = 6.3	ATZ = 99% TOC = 22.1%	Li et al. [50]
	Bisphenol A (BPA)	DSA and Ti/IrO_2-RuO_2, gas diffusion electrode	Magnetic nitrogen doped/ reduced graphene oxide	Electrode spacing = 2.5 cm Na_2SO_4 = 0.05 M T = 25°C Initial pH = 3.0 Aeration = 0.5 L/min	BPA = 90.0% TOC = 60.5%	Zhang et al. [84]

2.4.2. Transition metal-based materials

Similar to carbon materials, transition metals-based materials have good electrical conductivity, additionally with several catalytic active sites on their surface. Iron-based materials (such as steel slag [48], tailings [49]) and other iron synthetic materials (e.g., $CuFe_2O_4$ [50] and $Fe_3O_4^-$-based materials [51]) are the most researched due to their easy availability. Moreover, transition metal-based materials can provide some metal ions for electro-Fenton-like reactions. Hence, the organics degradation efficiency can be improved significantly [52]. The slag and tailings are solid wastes produced by mining and industrial metallurgical processes, harmful to the environment. However, they have a high recovery value for metals. Thus, their utilization is of great significance to the sustainable development of the environment.

Some previous studies modified slags and tailings to prepare PEs with a mesoporous structure or multiple active sites. They demonstrated that these PEs had outstanding adsorption and catalytic abilities [53]. However, due to their high density, most transition metal-based materials are relatively difficult to fluidize or homogenize in an electrolyte.

2.4.3. Composite materials

Composite materials mainly refer to combining two or more categories of materials that combine their characteristics to achieve a higher stability and better catalytic performance than a single material. The properties of these raw materials often influence the removal mechanism of contaminants. The composite materials frequently have multitudinous surface functional groups, capable of enhancing their electro-oxidation and electrocatalytic performances. The PEs prepared by composite materials can catalytically produce different ROS, increasing the pollutants' removal by the combined efforts of the ROS [54,55]. Recently, some scholars [56–58] prepared composite electrodes to build 3DER for removing refractory organics. These PEs exhibit large specific surface area and superior catalytic activity. However, the composite materials are still tedious for practical application due to high-cost and complex manufacturing process.

3. Applications in refractory organic wastewater treatment

Generally, the ratio of biochemical oxygen demand (BOD) to chemical oxygen demand (COD) of wastewater (i.e., the value of BOD_5/COD) is lower than 0.3; hence, it can be considered as refractory organic wastewater [59]. Due to its environmental friendliness, no secondary pollution, high efficiency, flexible and easy operation, 3DER has become an important method to treat refractory organic wastewater, such as leachate, oily wastewater, coking wastewater, pharmaceutical wastewater, dyeing wastewater, and their membrane concentrates. Table 4 shows some recent studies on the treatment of refractory organic wastewater by 3DER. Voltage (U), current density (J), current intensity (I), reaction time (HRT), reaction flow rate (RFT), temperature (T), and volume (V) are the key factors affecting the treatment efficiency. The construction of 3DERs, reaction conditions, and contaminate removal mechanisms focus on various 3DER. However, most of these studies are laboratory-based; their practical applications in the field still need further research.

4. Conclusions and prospects

This review summarizes the reaction mechanisms and applications of 3DER, elucidating the characteristics of carbon-based, metal-based, and composite PEs materials. In conclusion, direct and indirect oxidation are the main organics removal pathway. The PEs can significantly increase the current efficiency and shorten the mass transfer distance of the electrochemical system, thus, significantly improving the pollutants removal efficiencies. Due to the excellent ability of 3DER to reduce toxicity and enhance the biodegradability of refractory organic wastewater, we believe that 3DER is the preeminent treatment process for refractory organic wastewaters. However, many studies on the treatment of refractory organic wastewater by 3DER are still in the laboratory stage, as its practical applicability remains to be explored.

Moreover, many previous studies focused on the preparation of composite PEs materials, and the preparation methods and reaction conditions are increasingly complicated. These trends seem to work against the engineering application of 3DER. Therefore, it will be a promising research direction to explore the preparation of the PEs materials with low cost and easy synthetic conditions. In addition, it is important to realize the stable operation of large-scale industrial reactor in the future.

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