

Research Progress of Persulfate Activation Methods

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Abstract

With the emergence of emerging pollutants in water, water pollution becomes more and more serious. Under this background, persulfate based advanced oxidation process has attracted wide attention because of its high treatment efficiency and low secondary pollution. In this paper, the research progresses of persulfate activation by heat, light, electricity, microwave, ultrasound, transition metals, alkali, carbon materials, organic matter, etc. were reviewed. Specifically, the mechanisms of different activation methods have been introduced, the treatment result of each activation system and the influencing factors in the reaction process were expounded, and the future development of persulfate activation technology was prospected for reference.

Keywords

Persulfate; Activation Method; Advanced Oxidation Process; Radical; Research Progress.

1. Introduction

With the rapid development of global economy, environmental problems are increasingly serious. Water resources as an essential resource for human production and living, its pollution and governance has always been the focus. In recent years, (water) emerging pollutants, represented by drugs and personal care products, have posed a great threat to ecological security and human health [1]. Traditional water treatment processes usually fail to effectively remove these emerging pollutants, so new clean and efficient water treatment technologies are urgently needed.

Advanced Oxidation Process, (AOPs) based on sulfate radical ($\text{SO}_4^{\cdot-}$) is an efficient water purification technology that has attracted much attention in recent years. It mainly generates strong oxidative active species dominated by $\text{SO}_4^{\cdot-}$ through the activation of Peroxymonosulfate, (PMS) or persulfate (PDS) [2]. Compared with hydroxyl radical ($\cdot\text{OH}$), the primary active species in traditional AOPs system, $\text{SO}_4^{\cdot-}$ has a higher oxidation-reduction potential (2.5~3.1 V) and a wider pH application range, giving it a better water treatment capacity [3][4]. In view of the fact that different activation methods can significantly affect the reaction mechanism and the yield of active species, and ultimately determine the organic degradation efficiency and treatment cost, this paper systematically introduced the research progress of various persulfate (PS) activation systems based on energy and electron transfer activation methods, revealed the action mechanism and characteristics of different activation methods, and prospected the research direction in the future for reference.

2. Energy-based activation

2.1 Thermal activation

Thermal activation of PS is the use of heat to break the peroxide bond in the PS molecular structure to produce $\text{SO}_4^{\cdot-}$ [5]. Zhang et al. [6] found in the thermal activation of PDS to degrade 1-Amino-8-hydroxynaphthalene-3,6-disulphonic acid (H acid) that increasing temperature, increasing PDS dosage and increasing initial pH can significantly increase the degradation rate of H acid. Under the

condition of initial pH = 3–9, the highest degradation rate (77%) of H acid was observed when PDS concentration was 6.0 mmol/L, temperature was 50 °C and pH was 9. Zhang et al. [7] found in their research on the treatment of metal cutting waste liquid by thermal activation PDS that the COD value of the waste liquid after coagulation and demulsification decreased 96.89% after reacting for 3 h under the condition of PDS:12COD₀ = 2.4, pH = 5 and temperature of 100 °C. Through infrared spectrum analysis, the concentration of benzene, amide, carboxylic acid, alcohol, amine, halide, aromatic hydrocarbon and other organics in the waste liquid is decreased; through cation chromatography analysis, it is found that Ca²⁺, Mg²⁺ and Cl⁻ in the waste liquid are also removed to a certain extent.

2.2 Electrochemical activation

Electrochemical activation realizes energy transfer through the electric field of electrochemical reaction, exciting the chemical bonds in PS to break and form SO₄⁻ [8] [9], or accepting electrons generated by electrochemical reaction to undergo oxidation-reduction reaction, generating SO₄⁻ and then degrading organic matters [8][9][10].

Guo et al. [11] used porous carbon felt electrodes to activate PDS to degrade perfluorooctanoic acid (PFOA) by potentiostatic method. The results showed that PFOA was almost completely degraded and the removal rate of TOC was about 55% after reacting for about 8 h at a constant potential of 1.8 V. When the electrolysis time was further extended to 24 h, the removal rate of TOC in the system was about 62.5%. Through the analysis of gas phase products by gas chromatography-mass spectrometry (GC-MS), the authors concluded that the possible degradation pathways of PFOA included SO₄⁻-mediated Kolbe decarboxylation and hydrogenation-reduction of carboxylic acid.

Zheng et al. [12] used electrochemical activation PDS to remove *Microcystis aeruginosa* from the solution. The degradation rates of algae cells and chlorophyll a reached 90.80% and 98.41% respectively after treatment for 60 min at the initial algae density of 1.24 × 10⁷–1.30 × 10⁷ cells/mL, voltage of 7 V, initial pH of 6, and PDS dosage of 4 mmol/L. SO₄⁻ and [•]OH generated in the EC/PDS system were the main active species for algal removal, and they interacted with electrochemistry on algal cells and algal organics, improving the efficiency of algal removal.

Feng et al. [13] found in the research experiment on degradation of phenol-containing wastewater by electrochemical activation of PDS with graphene electrode that PDS was activated with graphene electrode under the energization condition, when n(PDS):n (phenol) = 50:1, pH = 11, and current density of 30 mA/cm², the removal rate of phenol reached 98.91% after 90 min of reaction, significantly better than that of the single graphene system (47.17%).

2.3 Microwave activation

Microwave activation is different from thermal activation. It achieves uniform heating through heat transfer at the molecular level, and reduces the reaction activation energy, thus improving the efficiency of SO₄⁻ production by PS [2]. Zhang et al. [14] studied the degradation of butyl xanthate by PDS activated by microwave (Microwave, MW) and found that when the dosage of PDS was 10 mmol/L, the MW power was set at 250 W and the reaction time was 13 min, the removal rate of butyl xanthate could reach 92.75% (the initial mass concentration of butyl xanthate was 150 mg/L). The experiment leads to a conclusion that the more PDS is added, the larger the MW power is and the longer the activation time is, the higher the degradation efficiency of butyl xanthate is.

Hou et al. [15] found in the MW activated PDS coupled coagulation treatment of dinitrodiazophenol (DDNP) industrial wastewater experiment that when the initial pH of the system was 3, PDS concentration was 8 g/L, MW power was 600 W, and n(Fe²⁺/PDS) was 0.04, the degradation rates of COD and CN were 60.40% and 88.45% respectively after 8 min reaction, significantly higher than those of other processes. Based on the UV-Vis full-band scanning (240–600 nm) results, it could be speculated that the MW-Fe²⁺/PDS system could destroy the conjugated system of benzene ring, as well as the azo group and nitro group structures in DDNP, and generate simple and small molecules such as amides and alcohols. Gu et al. [16] systematically studied the COD removal of DDNP

industrial wastewater under different treatment processes. The results showed that in the MW-Fe²⁺/PDS system, there was a significant synergistic effect between MW, Fe²⁺ and PDS. After treatment, the biodegradability of DDNP wastewater was increased by about 12 times. In addition, the MW-Fe²⁺/PDS process had lower energy consumption than the heat-Fe²⁺/PDS process, and it was a highly efficient pretreatment method suitable for DDNP industrial wastewater.

2.4 Photoactivation

Photoactivation refers to the use of ultraviolet light or visible light to provide energy to activate PS, so that the peroxide bond therein is broken to produce SO₄^{•-}, thereby degrading pollutants [17].

Wang Zhenyu et al. [18] studied the degradation of methylene blue (MB) in dye wastewater and found that the oxidative active substances generated by visible light activation of PDS were SO₄^{•-} and [•]OH, and SO₄^{•-} was the main active species that degraded MB. Increasing the dosage of PDS and decreasing the pH value of the system could both effectively improve the removal rate of MB, while increasing the initial concentration of MB would reduce the removal rate.

Jin et al. [19] concluded in the degradation of tri (1,3-dichloroisopropyl) phosphate (TDCPP) by PDS activated by ultraviolet (UV) that when the initial pH was 7.0, the dosage concentration of PDS was 50 mg/L, and the reaction time was 30 min, the removal rate of TDCPP was the highest (97.9%). In addition, the presence of Cl⁻, CO₃²⁻ and NO₃⁻ ions in the solution inhibited the degradation of TDCPP by UV/PDS to a certain extent, and the inhibition was in the order of CO₃²⁻ > NO₃⁻ > Cl⁻.

2.5 Ultrasonic activation

Ultrasonic activation of PS mainly relies on the local high temperature generated by the collapse of cavitation bubbles formed by ultrasonic wave to activate PS and generate SO₄^{•-}, which is similar to the thermal activation mechanism [20].

Yang et al. [21] studied the factors affecting the degradation of methyl orange by ultrasonic activation of PDS and found that the removal rate of methyl orange was positively correlated with the sound intensity and PDS concentration. When the sound intensity is 0.54 W·cm⁻², the removal rate of methyl orange is 87.38%, which is 45.25% higher than that when the sound intensity is 0.22 W·cm⁻². When the concentration of PDS is 0.5–4 mmol/L, the removal rate of methyl orange in 60 min is 33.5%–54.38% and increases with the increase of PDS concentration.

Li et al. [22] have found in the degradation of perfluorooctane sulfonate (PFOS) in solution by ultrasonic activation of PDS that the removal efficiency of PFOS by ultrasonic activation of PDS system was higher than that by ultrasonic and PDS oxidation systems alone. When the temperature was 30 °C, the ultrasonic frequency was 400 kHz, the power was 100 W, and the density was 2.67 W·cm⁻², under the condition of n(PDS):n(PFOS) being 50:1, and the reaction time being 8 h with argon as the cavitation gas, the removal rate of PFOS in the solution reached 99.5%.

3. Activation based on electron transfer

3.1 Transition metal activation

Transition metal-based catalysts can usually activate PS at room temperature, mainly including homogeneous catalysts represented by transition metal ions (Fe²⁺, Co²⁺, Ti³⁺, Cu²⁺, Ag⁺, and Mn²⁺, etc.), and heterogeneous catalysts represented by transition metal-based solid-phase materials [2]. Among them, Fe-based materials have attracted more attention and research due to their low cost.

Xiong et al. [23] conducted the degradation of RhB by pyrite-activated PDS. When the dosage of pyrite was 1.0 g/L, the dosage of PDS was 0.5 mmol/L, and pH was 5, the degradation rate of RhB (20 mg/L) reached 100% after reaction for 3 h. The free radical species mainly including SO₄^{•-} formed in the system dominated the degradation of RhB. Anions (Cl⁻, NO₃⁻ and HCO₃⁻) in the system can inhibit the efficiency of removing RhB by pyrite activation PDS, and the inhibition effect is in the order of HCO₃⁻ > Cl⁻ > NO₃⁻.

Yao et al. [24] studied the degradation of organochlorine pesticides in water by ball-milling activated PDS with zero-valent iron sulfide (S-mZVI). They found that compared with the mZVI/PDS system, the S-mZVI/PDS system exhibited a higher degradation efficiency for organochlorine pesticides. The larger the S/Fe molar ratio was, the better the activation effect of PDS would be, and the resulting $\text{SO}_4^{\cdot-}$ and $\cdot\text{OH}$ would also increase. When S/Fe = 0.10 and the dosage ratio of PDS/Fe was 1/2, the removal efficiency of 4,4-DCBP and β -HCH was the best, and the degradation rates reached 95.0% and 91.3% after 5 h reaction, respectively, indicating that S-mZVI/PDS system could repair the water polluted by organochlorine pesticides.

In the study on the degradation of active black 5 by PDS activated by Fe_3O_4 , Rebecca et al. [25] first passed through the Fe_3O_4 catalyst prepared by hydrothermal synthesis and characterized. Under the optimal conditions of initial pH = 6, PDS concentration of 6 mmol/L, and Fe_3O_4 concentration of 2.0 g/L, the removal rate of Active Black 5 (50 mg/L) reached 80.2% within 180 min. Moreover, the catalyst exhibited good reusability, and the removal rate was still above 77% after five times of use.

Using potassium ferricyanide as the iron source and copper nitrate trihydrate as the copper source, Wang et al. [26] adopted the two-step method of coprecipitation and then calcining the intermediate product to obtain $\text{CuO}/\text{Fe}_3\text{O}_4$ magnetic nanoparticles. Iron ions and copper ions have a synergistic effect, which is beneficial to accelerating the cycle of Fe(III)/Fe(II), and Cu(II)/Cu(I) and accelerating the generation of free radicals, thereby efficiently treating organic pollutants. The catalytic reaction can be quickly carried out at room temperature, is environment-friendly, and is suitable for treating dye-containing wastewater.

3.2 Alkaline activation

pH was an important factor in the activation process of PS. Basic condition could quickly and effectively activate PS to produce $\text{SO}_4^{\cdot-}$. At the same time, when pH > 8.5, $\text{SO}_4^{\cdot-}$ oxidized H_2O or OH^- to produce $\cdot\text{OH}$, and the system degraded organic matters through the interaction of $\text{SO}_4^{\cdot-}$ and $\cdot\text{OH}$ [27].

Li et al. [28] have found that alkali activation of PDS can effectively treat chlorinated hydrocarbon pollutants and use it for groundwater remediation of chlorinated hydrocarbon contaminated site. The degradation rate of chlorinated hydrocarbon increases with the increase of PDS dosage. The dosage ratio of oxidizing agent in the pilot test stage is 1%. After eight months of construction test, the concentration of chlorinated hydrocarbon in groundwater in the experimental area can reach the remediation target.

Ge Yong et al. [29] found in the degradation of ciprofloxacin (CIP) in water by alkali-activated PMS that with the increase of the concentration of NaOH and PMS, the degradation of CIP tended to increase first and then decrease. The increase of reaction temperature was beneficial to the degradation of CIP; The removal of CIP by different anions showed different effects in the system. Among them, Cl^- , SO_4^{2-} and NO_3^- had little effect on the system. H_2PO_4^- inhibited the degradation of CIP, while CO_3^{2-} promoted the reaction.

3.3 Carbon material activation

Carbon materials have the characteristics of abundant microporous structure, large specific surface area and excellent adsorption performance. Carbon materials such as carbon nanotubes and graphene rely on the developed covalent carbon network and edge-defective oxygen-containing groups to transfer electrons to PS so that it can be activated to produce $\text{SO}_4^{\cdot-}$ [30].

Cao et al. [31] studied the activation of PDS by nitrogen-doped graphite carbon-embedded Fe^0 composite ($\text{Fe}@\text{HC}$). $\text{Fe}@\text{HC}$ material has a core-shell structure with external carbon and internal iron. Its large surface area enhanced the activation of PDS and contributed to the oxidative degradation of pollutants. The co-existence of $\text{Fe}@\text{HC}$ and PDS system could remove azo dye Reactive Black 5 (RB5) more efficiently than $\text{Fe}@\text{HC}$ and PDS system alone. When the initial

concentration of RB5 was 20 mg/L, the dosage of Fe@HC was 0.5 g/L and the concentration of PS was 5 mmol/L, the decolorization rate was 99.24% and the naphthalene ring damage rate was 61.76%. Zhang Liming et al. [32] studied the degradation of Au-Orange G (OG) system by PMS activated by carbon nanotubes (CNT) and they confirmed that the free radical mainly generated by PMS activated by CNT was $\text{SO}_4^{\cdot-}$. CNT/PMS system showed better decolorization effect on OG, because the azo bond and naphthalene ring were damaged to a certain extent during the degradation of OG. In addition, the mineralization rate of OG molecule in the CNT/PMS system was high.

In their study on the degradation of phenol by PDS activated by Co-doped ZIF-67-based mesoporous carbon material (Co-NC), Hsu et al. [33] found that the synthesized Co-NC activated PDS best when Co was doped with 2 mmol, and the synthesis temperature was 30 °C and 600 °C. When the mass ratio of Co-NC to PDS was 1:4 and pH = 7, the mineralization of phenol was the highest, and the mineralization rate of phenol reached 88.3% after 90 min reaction. Under the same conditions, the removal efficiency of activated PDS was better than that of PMS. Based on the experimental results of free radical trapping, it was found that $\cdot\text{OH}$ and $\text{SO}_4^{\cdot-}$ were both involved in the reaction, but $\cdot\text{OH}$ played the leading role.

3.4 Organic matter activation

Liu et al. [34] studied the degradation of organic matter in soil by PDS activated by natural organic matter and found that the highest degradation efficiency of PAHs was close to 80% when humic acid combined with PDS activated by ferrous ion was used to treat soil. The degradation efficiency of polysaccharide-activated PDS to total petroleum hydrocarbon and high-cyclic polycyclic aromatic hydrocarbons is the highest, reaching 84.37% and 87.56%, respectively. The highest soil oxidation-reduction potential (Eh) was 676.27 mV after treatment with humic acid combined with ferrous ion activated PDS, followed by Eh after treatment with citric acid chelated iron and polysaccharide activation, indicating that the oxidation of PDS system activated by macromolecular organic compounds was stronger. The soil treated with humic acid combined with PDS activated by ferrous ions and sugars was less acidic than the soil treated with PDS activated by citric acid chelated iron, and had higher safety.

4. Conclusion

Advanced oxidation technology based on PS, as an efficient means to degrade organic compounds, is being widely used to purify various types of water. With the deepening of research, the activation mode of PS is also constantly optimized. For example, many new catalysts replace the original activation mode with high energy consumption and large secondary pollution. However, there is still room for improvement in PS activation technology. The development direction in the future can be focused on the points below. First, to explore the applicability of each activation method to different treatment objects and select the activation means with high efficiency, low cost and no secondary pollution in practical application. Second, design the reactor with low cost, long service life and the ability of improving the reaction efficiency. Third, realize that high coupling between the PS activation technology and the traditional process, improving the effluent quality and reducing the cost-benefit ratio.

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