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Progress on the Regulation Mechanism of Free Radical and Non-Free Radical Pathways in the Advanced Oxidation Technology of Peroxyacetic Acid

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Abstract. As a green oxidizing agent, peroxyacetic acid (PAA) has made important progress in the activation mechanism and engineering application research. This review systematically analyzes the radical generation laws in UV light activation, transition metal catalysis, and nanomaterial composite non-homogeneous systems, focusing on revealing the ultrasonic activation mechanism in non-homogeneous catalytic systems where the non-radical pathway accounts for more than 70 %. It was shown that the rate of ·OH generation was enhanced 3.8-fold when the PAA concentration was ≥ 0.5 mM (K_{obs} = 0.42 min^{-1}). By integrating the data of the transition metal/carbon material co-activation system, the dynamic regulation of the active species by environmental factors, such as pH (3.0-8.2), coexisting ions (Cl^-, HCO_3^-, etc), was elucidated. Practical cases demonstrate that the CoFe-LDH/PAA system effectively removes over 90 % of sulfamethoxazole from hospital wastewater, resulting in a 70 % reduction in the cost of treating tons of water compared to the traditional process. In the future, future work should develop machine learning-driven regulation systems to realize the intelligent switching of free radical/non-free radical pathways and break through the bottleneck of complex water quality adaptation.

Keywords: Peroxyacetic acid (PAA), Advanced oxidation processes (AOPs), Free radical pathways, Non-radical pathways, Wastewater treatment

1. Introduction

The threat of emerging pollutants in the aquatic environment has led to innovations in advanced oxidation processes. While the traditional Fenton process suffers from high iron sludge production (120-150 mg/L) and low H_2O_2 utilization (<40 %), the PAA system exhibits unique advantages:

• The O-O bond energy (159 kJ/mol) is significantly lower than that of H_2O_2(213 kJ/mol) and is more easily activated to produce reactive species such as ·OH (1.96 V), $CH_3C(O)OO·$, etc.

• Decomposition products are non-toxic, and THMS generation is 90 % lower than chlorine disinfection

• Broad-spectrum pH adaptability (3.0-11.0), breaking the limit of ozone alkaline deactivation

However, there are three major limitations of the existing studies:

• Insufficient quantification of non-radical pathway ($^1O_2$, electron transfer) contributions

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• Unknown mechanism of interference between NOM (10 mg/L HA decreases efficiency by 30 %) and Cl^-(>1 mM triggers pathway shift) in real water bodies

• Poor stability of transition metal catalysts (>50 % activity decay over 5 cycles)

This review innovatively integrates the literature in the last six years (2019-2025) to study the experimental data and theoretical models of UV activation, transition metal catalysis and non-homogeneous material systems, and elucidates the adaptive mechanism of PAA under different water quality conditions, to provide theoretical support and direction of technological breakthroughs for engineering applications.

2. Activation mechanisms and free radical generation

2.1. Chemical properties of PAA

Peroxyacetic acid (PAA) is an organic peroxyacid with the molecular formula $CH_3C(O)OOH$. The peroxy group (O-O bond) in its molecular structure is highly reactive, with a bond energy (159 kJ/mol) significantly lower than that of H_2O_2(213 kJ/mol) and persulfate (317 kJ/mol), making it easier to be activated by light, heat or catalysis [1].

Oxidizing ability and pH dependence: The standard redox potential of PAA is 1.96 V (vs. standard hydrogen electrode (NHE)), which is higher than that of H_2O_2(1.78 V). Under acidic conditions (pH < 8.2), PAA exists as a neutral molecule that directly oxidizes pollutants, whereas its decomposition rate is accelerated in alkaline environments (pH > 8.2) to produce H_2O_2 and acetic acid (CH_3COOH). It has been shown that PAA is more stable under acidic to neutral conditions (pH 3.0-8.2), while its decomposition accelerates under alkaline conditions (pH >8.2) with a significantly shorter half-life [2].

Decomposition products and environmental friendliness: The final decomposition products of PAA are water and acetic acid, with no halogenated by-products remaining. For example, in humic acid-containing wastewater treatment, the generation of trichloromethane (TCM) in the PAA system is reduced by more than 90 % compared with chlorine disinfection [3], which is a characteristic that gives it a significant advantage in medical wastewater treatment.

2.2. Activation pathways of PAA

The activation mechanisms of PAA can be classified into three categories: homogeneous catalytic, non-homogeneous catalytic, and energy-driven activation, with significant differences in the active species generated by different pathways and their selectivity.

2.2.1. Homogeneous catalytic activation

Homogeneous activation relies on transition metal ions (e.g., Fe^{2+}, Co^{2+}) to cleave the O-O bond of PAA by single-electron transfer (SET).

1. Iron-based activation: Fe^{2+} reacts with PAA as shown in equation (1)

$$Fe^{2+} + CH_3C(O)OOH \rightarrow Fe^{3+} + CH_3C(O)O\cdot + OH^- \quad (1)$$

The secondary rate constant ($K_{Fe^{2+}}$) for this system was 1.10×10^4-1.56×10^4 $M^{-1}\cdot s^{-1}$ (pH 3.0-8.1). The early stage of the reaction is dominated by $CH_3C(O)O\cdot$, while the later stage of Fe^{3+} reduction generates H_2O_2, which contributes to ·OH via a Fenton-like reaction [4].

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2. Cobalt-based activation: Co^{2+}activates PAA more efficiently (k_{Co}^{2+} =1.70× 10^3-6.67×$10^3 M^{-1}s^{-1}$) and selectively generates acetyl peroxyl radicals ($CH_3(O)OO\cdot$)

For example, the rate of degradation of carbamazepine by the Co^{2+}/PAA system at pH=6.0 (k_{obs}=3.04×$10^{-3}s^{-1}$) was 2.1 times higher than that of the Fe^{2+} system [2].

2.2.2. Non-homogeneous catalytic activation

Non-homogeneous catalysts promote electron transfer and reduce metal leaching through surface active sites.

1. Metal oxide catalyst: nano copper oxide (nCuO): The Cu^+/Cu^{2+} redox pair on the surface of CuO can activate PAA efficiently. The nCuO/PAA system has been shown to degrade bisphenol A up to 98 % within 30 min, and the Cu leaching concentration is lower than 0.1 mg/L [5].

2. CoFe layered double hydroxide (LDH): Co^{2+}/Fe^{3+} synergistically leads to free radical concentrations up to 5.27×10^{-13} M and sulfamethoxazole removal of more than 95 % [6].

3. Carbon-based composites: Carbon dots (CDs) modified g-C_3N_4/Sn_2 heterojunction broadened the visible light response range and promoted the generation of superoxide radicals ($\cdot O_2^-$) and holes (h^+), which enhanced the degradation rate of anti-inflammatory pain by 5.6-fold [7].

2.2.3. Energy-driven activation

1. UV activation (UV/PAA): UV light (λ=254 nm) directly cleaves the O-O bond of PAA

$$CH_3C(O)OOH \rightarrow CH_3C(O)O\cdot + \cdot OH \quad (2)$$

The UV/PAA system exhibited maximum ·OH production at pH 3.0 and a carbamazepine degradation efficiency of 90 % [8].

2. Electrochemical activation (EC/PAA): cathodic reduction of PAA to generate ·OH

The EC/PAA system degraded tetracycline at a rate 3.8 times higher than that of PAA alone [9].

2.3. Comparison of revitalization paths

The different activation pathways cleave the peroxygen bond (O-O, bond energy 159 $kJ\cdot mol^{-1}$) of PAA through energy transfer or electron transfer mechanisms, generating reactive species dominated by ·OH, $CH_3C(O)O\cdot$ (acetoxyl radical), and RO·(organo-oxygen radical) [10]. The critical pathway comparison is shown in Table 1:

Table 1. Comparison of radical generation characteristics and operational parameters for PAA activation pathways

activation system	Dominant free radicals	Optimal pH range	Oxidizer consumption	Energy consumption/costs	Secondary pollution risk
UV/PAA	·OH, $CH_3C(O)O\cdot$, RO·	7.0-9.0	Medium (50-300 μM)	High (UV equipment)	No metal residues
Transition Metal/PAA	·OH, RO·, high-value metal oxides	3.0-6.0	Low (<100 μM)	Low (room temperature reaction)	Metal leaching (e.g., Cu^{2+})
Heat/PAA	·OH, $CH_3\cdot$	5.0-8.0	High (>500 μM)	Very high (heating to 60-80 ℃)	Not have

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2.4. Radical generation pathways under different activation conditions

2.4.1. Ultraviolet (UV) activation

UV light (λ = 254 nm) generates free radicals by direct cleavage of the O-O bond of PAA (Eq. 3):

$$CH_3C(O)OOH \rightarrow CH_3C(O)O\cdot + \cdot OH \quad (3)$$

It was shown that the generation of free radicals in the UV/PAA system has a significant pH dependence. Under acidic conditions (pH= 3.0), the steady-state concentration of ·OH reaches up to 3.8×10^{-13}M, with a contribution of more than 80 %, whereas under alkaline conditions (pH= 9.0), $CH_3C(O)OO\cdot$ becomes the dominant species (with a contribution of 65 %) through a chain reaction

Key Features:

pH dependence: Acidic conditions (pH=3.0) were dominated by ·OH (concentration 3.8×10^{-13}M, contribution >80 %), and alkaline conditions (pH=9.0) were dominated by $CH_3C(O)OO\cdot$ through a chain reaction (contribution 65 %).

Efficacy and Limitations: achieved 90 % carbamazepine degradation at pH 3.0 within 30 min, but efficiency decreased by 40 % when turbidity exceeded 5 NTU [8].

2.4.2. Transition metal catalyzed activation

Iron-based catalysts

Homogeneous Fe^{2+} activation.

$$Fe^{2+} + CH_3C(O)OOH \rightarrow Fe^{3+} + CH_3C(O)O\cdot + OH^- \quad (4)$$

The secondary rate constant k = 1.10×10^4-1.56×10^4 $M^{-1}s^{-1}$ (pH = 3.0-8.1), and the bisphenol A removal at pH = 5.0 was 92 %, but Fe^{3+} precipitation (pH>8.0) led to deactivation [4].

Zero-valent iron (ZVI) activation:

$$ZVI + CH_3C(O)OOH \rightarrow Fe^{2+} + CH_3C(O)OO\cdot + H^+ \quad (5)$$

The core-shell structure $SiO_2@Fe^0$ reduces Fe leaching to 0.05 mg/L and enhances free radical yield by 1.8-fold [5].

Cobalt-based catalysts

$$Co^{2+} + CH_3C(O)OOH \rightarrow Co^{3+} + CH_3C(O)OO\cdot + H^+ \quad (6)$$

Free radical quenching experiments showed that 2,4-hexadiene (2,4-HD) inhibited the degradation of sulfamethoxazole by 85-99 %, confirming the dominant role of $CH_3C(O)OO$- [2]. Co^{2+}dissolution of 0.8-1.2 mg/L requires loaded catalysts (e.g., $Co_3O_4@C$) to reduce the risk.

Copper-based catalysts

Nano-CuO activated PAA by surface Cu^+/Cu^{2+} cycling, and the rate of carbamazepine degradation at pH = 6.0-7.0, $k_{obs} = 4.7 \times 10^{-3}s^{-1}$, was 2.3 times higher than that of the homogeneous Cu^{2+} system by a factor of 2.3 [11].

Table 2 shows a comparison of the radical generation characteristics of the transition metal catalysts:

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Table 2. Comparison of free radical generation characteristics of transition metal catalysts

Catalytic performance parameters"	Fe^{2+}	Co^{2+}	nCuO
k ($M^{-1}s^{-1}$)	1.10×10^4	6.67×10^3	3.8×10^2
Dominant free radicals	$CH_3C(O)O^-$	$CH_3C(O)OO^-$	$CH_3C(O)OO^-$
optimal pH	3.0-8.1	6.0-8.0	6.0-7.0
Metal Dissolution (mg/L)	0.8-1.5	0.8-1.2	<0.1

2.4.3. Non-homogeneous catalytic systems

Layered double hydroxide (LDH)

CoFe-LDH activates PAA via Co^{2+}/Fe^{3+}synergy (Eq. 7):

$$Co^{2+} + Fe^{3+} + CH_3C(O)OOH \rightarrow Co^{3+} + Fe^{2+} + CH_3CO)OO\cdot + H^+ \quad (7)$$

The free radical concentration of 5.27×10^{-13}M resulted in >95 % removal of sulfamethoxazole; Co^{2+} dissolution was <0.05 mg/L, and the retention of activity over 5 cycles was >85 %.

Carbon-based composites

Carbon dot modification of g-C_3N_4/SnO_2: broadened visible light response range (400-650 nm), 5.6-fold increase in the rate of anti-inflammatory pain degradation ($k_{obs} = 1.2 \times 10^{-2}s^{-1}$), no metal leaching [7].

Co@NC catalyst: 3.2-fold enhancement of ciprofloxacin degradation selectivity at a steady-state $^1O_2$ concentration of 2.4×10^{-12}M [12].

3. Mechanisms of regulation of free radical generation by environmental factors

3.1. pH regulation of free radical generation pathways

3.1.1. pH dependence of homogeneous systems

The reaction pathways and radical generation characteristics of the homogeneous Fe^{2+}/PAA system exhibit significant pH-dependent features:

1. Acidic conditions (pH = 3.0-5.0)

Fe^{2+}, which exists in the free hydrated ionic form of ferric ion hexahydrate ($[Fe(H_2O)_6]^{2+}$), preferentially activates PAA via single-electron transfer to generate acetoxyl radicals ($CH_3C(O)O\cdot$), which corresponds to a rate constant for the second-order reaction as high as 1.10×10^4 $M^{-1}s^{-1}$, contributing >80 % to sulfamethoxazole degradation under these conditions [4]. Hydroxyl radical (·OH) generation was inhibited at this time.

2. Neutral to weakly basic conditions (pH 5.5-8.2)

·OH became the dominant reactive species, and the contribution was elevated to 60-75 % [6]. However, the promotion of the reaction rate was weaker than that of the acetoxy radical, and the $k_{Fe^{2+}}$ value decreased to 70 % of the initial activity.

3. Alkaline conditions (pH> 8.2)

Fe^{2+} completely precipitates as $Fe(OH)_3$, catalyst deactivation leads to termination of the reaction, and sulfamethoxazole removal is reduced to less than 32 % of the initial activity.

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3.1.2. pH adaptation of non-homogeneous catalysts

The non-homogeneous system represented by CoFe-LDH exhibits wider pH adaptability characteristics:

1. Synergistic activation mechanisms

The valence synergism of Co^{2+}/Fe^{3+} in layered bimetallic hydroxides (redox potential difference up to 0.84 V) maintains an efficient electron transfer efficiency in the pH range of 6.0-7.0, with a steady-state radical concentration up to 5.27 × 10-¹³ M [6].

2. Surface-limited domain effect

The catalyst surface microenvironment effectively buffered pH fluctuations (ΔpH ≤ 1.5) and maintained 92 % sulfamethoxazole removal at an initial pH of 8.0, which was significantly higher than that of 36 % in the homogeneous system.

3.2. Effects of co-existing substances

3.2.1. Natural Organic Matter (NOM)

NOMs such as humic acid (HA) inhibit degradation efficiency through adsorption site competition and free radical quenching. Example:

Adsorption competition: 10 mg/L HA reduced the removal of sulfamethoxazole (SMX) by 30 % in the CoFe-LDH/PAA system [10].

Quenching effect: The quenching rate constant (k_{HA}) of HA with ·OH is 1.2×10^{10}L/(mol·s), which significantly shortens the radical lifetime [8].

3.2.2. Inorganic anions

Cl^-: Cl^- reacts with $CH_3C(O)OO\cdot$ to form low-activity Cl· (Eq. 8), and the degradation efficiency of SMX decreases by 40 % when the Cl^- concentration is >1 mM [13] :

$$CH_3C(O)OO\cdot + Cl^- \rightarrow CH_3C(O)O^- + Cl\cdot \quad (8)$$

HCO_3^-: HCO_3^- inhibits the reaction by neutralizing H^+(Eq. 9) and quenching -OH ($k = 8.5 \times 10^6 M^{-1}s^{-1}$):

$$HCO^{3-} + H^+ \rightarrow CO_2 + H_2O \quad (9)$$

5 mM HCO_3^- reduces the rate of carbamazepine degradation in the Co^{2+}/PAA system by 55 % (Eq. 10) [2].

$$Co^{2+} + HCO_3^- \rightarrow Co - HCO_3^+ \quad (10)$$

3.3. Effects of temperature

Elevated temperature accelerates PAA activation reaction kinetics but may change the radical-dominant pathway. Take the CoFe-LDH/PAA system as an example:

1. Activation energy: The apparent activation energy (Ea) is 32.5 kJ/mol, which is consistent with the Arrhenius equation

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2. Temperature effect: the SMX degradation rate increased from 0.12 min^{-1} to 0.34 min^{-1} (2.8-fold increase) when the temperature was increased from 20°C to 40°C, but the $CH_3C(O)OO\cdot$ percentage decreased from 75 % to 60 % [6].

3.4. Catalyst stability and reusability

1. Homogeneous catalyst: Fe^{2+} needs to be continuously dosed due to oxidative deactivation, with >50 % decrease in activity after 5 cycles [4].

2. Non-homogeneous catalyst: CoFe-LDH >85 % activity retention after 5 cycles, metal leaching (Co^{2+}<0.05 mg/L, Fe^{3+}<0.1 mg/L) under the emission standards [6].

4. Practical applications and challenges

4.1. Hospital wastewater treatment cases

The CoFe-LDH/PAA system has been realized in hospital wastewater treatment [6] :

Process parameters: 50 mg/L Co_2Fe_1-LDH synergized with 0.2-1.0 mM PAA (pH 6.4, 25°C), the removal of sulfamethoxazole (SMX) was enhanced from 69.6 % (0.2 mM PAA) to >90 % (1.0 mM PAA), and TOC, COD, and BOD were respectively were reduced by 35.8 %, 41.2 % and 22.1 %, and the biocompatibility index (B/C) was optimized from 0.332 to 0.636, confirming the broad-spectrum degradation ability for humic acid and aromatic pollutants.

Economic advantages: CoFe-LDH synthesis cost (15-20 /kg) is 70-80 % lower than precious metal catalysts (RuO_2/IrO_2,2800-3200 /kg), and the cost of tonne of water treatment (<0.8 USD) is significantly lower than that of ozonation (1.2-2.5 /tonne) and the Fenton method (1.8-3.0 /tonne). PAA dosing costs increase non-linearly with concentration (only 0.23 increase in cost per ton of water at 1.0 mM), and operating costs account for <15 % (manageable by annual fluctuations in feedstock of ≤25 % (Luukkonen and Pehkonen 2017).

4.2. Existing challenges

Aqueous matrix interference: HCO3-, Cl- (>1 mM) and humic acid (HA) inhibited the reaction rate (30 % decrease in SMX removal), and HA acted via a dual action of free radical quenching (k = 1.2 × 1010$M^{-1}s^{-1}$) and active site shielding [14].

Mechanistic complexity: the stability of mechanistic radicals ($CH_3C(O)O\cdot$) is pH-regulated, and the catalyst interfacial microenvironment (e.g., the density of surface hydroxyls) affects metal valence cycling, leading to fluctuations in efficiency.

Catalyst deactivation: continuous Co2+/Fe3+depletion and surface passivation lead to a reduction in LDH cycling efficiency, with a 15 %-20 % decrease in SMX removal after 5 treatments [15].

The improved scheme can realize the efficiency enhancement through the following integrated strategies - Electrochemically assisted regeneration: Apply- 0.8 V to regenerate Co^{2+}/$Fe^{2+}$with LDH cycle life exceeding 15 cycles and metal dissolution <0.1 mg/L. Ozone pre-oxidation coupling: Prioritize the cleavage of HA (≥50 % removal) to restore PAA degradation efficiency to 86 % for SMX. degradation efficiency of PAA on SMX to over 86 %. Non-radical pathway design: Fe-N-C catalyst reduces HCO_3^-interference via $^1O_2$ and electron transfer pathways, and improves the degradation rate of target pollutants by 2.1 times.

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5. Conclusion and outlook

Peroxyacetic acid advanced oxidation technologies (PAA-AOPs) can efficiently generate free radicals (·OH, $CH_3C(O)OOO·$) and non-radical species ($^1O_2$, high-valent metals) through the activation pathways such as light, heat, transition metals and carbon materials, which are outstanding in the broad-spectrum degradation of new pollutants (antibiotics, endocrine disrupters) and the enhancement of water biochemical quality. Compared with H_2O_2 or persulfate systems, PAA-AOPs have the advantages of mild activation conditions and few disinfection by-products, especially in complex water quality. However, the practical application of PAA-AOPs still faces the challenges of transition metal leaching, high purity PAA preparation cost, generation of toxic small molecule by-products, inhibition of water quality interferences, and insufficient analysis of non-radical mechanisms.

Future research should focus on the following directions: (1) material design: develop interference-resistant catalytic materials (e.g., metal-nitrogen doped carbon-based catalysts), and directionally regulate the pathways of non-radicals ($^1O_2$, electron transfer); (2) mechanistic analysis: combine with in-situ characterization techniques (e.g., X-ray absorption spectroscopy, time-resolved fluorescence) to reveal the dynamics of the generation of non-radical species; (3) process optimization: integrate (3) process optimization: integrating electrochemical regeneration, pre-oxidation coupling and other strategies to reduce energy consumption and inhibit metal leaching; (4) engineering validation: promoting the pilot test and large-scale application of UV/PAA and non-homogeneous catalytic systems, and perfecting the database of process parameters and dynamic efficacy. Through multi-dimensional innovation and interdisciplinary integration, PAA-AOPs are expected to become a new, efficient, and green pollutant control technology in the field of water treatment and contribute to the sustainable development of the water environment.

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