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Oxidative Efficiency of Ozonation Coupled with Electrolysis for Treatment of Acid Wastewater

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Abstract: Establishment of an ozone-based advanced oxidation process (AOPs-O₃) for effective treatment of acid wastewater is an important and difficult task. The process of ozonation coupled with electrolysis (electrolysis-ozonation, E-O₃) has been reported to effectively degrade pollutants in neutral solution. We studied the efficiency of E-O₃ for degradation of acetic acid (HAc, an ozone inert chemical) in acid solution and found that E-O₃ had high oxidative efficiency at pH less than 3. For example, 52.2% of 100 mg·L⁻¹ HAc could be removed by E-O₃ in 120 min at pH 1.0, but only 2.2% and 3.5% by electrolysis and ozonation, respectively. Although the efficiency of E-O₃ decreased with the increase of acidity of solution, it still remained relatively high even at pH 0. An aromatic compound of acetophenone could also be effectively degraded by E-O₃ at pH 1.0. The results indicate that electrons can transfer from cathode to dissolved ozone or oxygen in acidic solution, thus resulting in generation of reactive species, e.g. hydroxyl radicals. A real acidic wastewater was also effectively pretreated by E-O₃. This study provides a promising AOPs-O₃ for treatment of acid wastewaters.

Key words: electrolysis; ozone; acetic acid; efficiency; acid wastewater

1 Introduction

Acid wastewaters from some chemical factories are difficult to treat via conventional treatments. Also, some of concentrated acid wastewater is a potential resource after purification^[1]. Advanced oxidation process (AOP) is well-known for the generation of hydroxyl radicals ($\cdot\text{OH}$), which can mineralize organics at high rate constants ($10^6 \sim 10^9 \text{ mol} \cdot \text{L}^{-1} \cdot \text{s}^{-1}$)^[2]. Although conventional Fenton process is effective for the treatment of weakly acidic wastewater, its application is always limited by the narrow working pH range and large sludge yielded^[3,4]. Of course, some new heterogeneous Fenton processes have been developed in recent years^[5]. It is more environment friendly, e.g., working at neutral pH, but there is a lot of work to be

done before application in practice. Therefore, establishing a method for treatment of acid wastewaters is of great significance to sustainable development of chemical industry.

Ozone, a strong oxidizing agent, has been extensively applied in water treatment for many years^[6, 7]. However, ozonation alone is difficult to completely remove organic pollutants owing to the high selectivity of its oxidative power. Various ozone-based advanced oxidation processes (AOPs-O₃), such as H₂O₂/O₃ (peroxone), UV/O₃, and catalytic ozonation, have been developed to solve the aforementioned issue^[8]. Nevertheless, few studies on AOPs-O₃ for the treatment of acid wastewaters at a pH less than 3 have been reported. Our previous work suggested that the working

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pH range of peroxone process could be broadened by adding catalysts containing Ti ion or the solid super acid of $\text{SO}_4^{2-}/\text{ZrO}_2\text{-Fe}_2\text{O}_3$. However, the stability of the catalysts is unsatisfactory because of rapid dissolution of metal ion leaching at pH less than 3^[9].

Goyal et al reported electrochemical oxidative treatment of acid wastewater (pH < 2) by Ti/RuO₂ electrode, and found that the maximum COD removal was only 48.83% under the optimized conditions^[10]. Some reports indicated that the combination of electrolysis and ozonation could significantly improve the degradation efficiency of organic pollutants^[11-13]. Electrolysis at an anode and a cathode could promote the decomposition rate of ozone to produce hydroxyl radicals^[14, 15]. Therefore, E-O₃ has gained widespread concern for its high efficiency^[16-18]. Electric power is only needed in E-O₃ process without addition of any other chemicals. However, to our best knowledge, no study has been done to test the efficiency of E-O₃ for the treatment of acidic wastewater so far.

Herein, the efficiency of E-O₃ at pH less than 3 was investigated in this work. Acetic acid (HAc), an ozone inert compound, was selected to degrade by E-O₃^[19, 20]. The experimental parameters of E-O₃, such as current and pH, were optimized. The efficiency of E-O₃ for the degradation of acetophenone in an acid solution was examined, and in an actual acid wastewater as well. The aim of this work is to study the oxidative efficiency of E-O₃ for the treatment or pretreatment of industrial acid wastewaters at pH less than 3.

2 Experimental

2.1 Experimental Apparatus

Figure 1 shows the experimental setup. The apparatus mainly consisted of an ozonation reactor, an ozone generator, an ozone destroyer, an electrolytic tank, and a regulated DC power supply. The anode material was graphite (15 cm × 12 cm, 180 cm²). The cathode was stainless steel mesh (19 cm × 12 cm, 228 cm²) with the active area of 152 cm². The pipeline was Teflon tube, and the ozonation reactor was a glass tube with the diameter of 100 mm. Models of ozone generator and destroyer were CFS-1A and GDF-003 (Ozonias Switzerland), respectively. The DC

regulated power supply was DH1716-3D. Ozonized gas produced from pure oxygen went into solution as bubbles through a sand distributor at the bottom of the reactor.

2.2 Experimental Materials

All reagents used were of analytical grade, and all solutions in the study were prepared with double distilled water. The electrolyte was 5 g·L⁻¹ of Na₂SO₄ solution. The pH of the solution was adjusted with 98% H₂SO₄ or 40.0 g·L⁻¹ NaOH solution. The initial concentration of HAc or acetophenone was 100 mg·L⁻¹. The gas flow rate was 0.4 L·min⁻¹ and all experiments were performed at room temperature. Each degradation test was repeated three times, and the data in figures was the average of the triplicate results, and the relative errors were all less than 5%.

2.3 Analytical Methods

The concentrations of HAc and acetophenone were determined by a high performance liquid chromatography (HPLC, Waters 1525-2996, USA), equipped with a C18 column (5 μm, 4.6 mm × 250 mm) and a UV detector. The solution pH was measured using a pH meter. The chromatographic conditions for determination of acetic acid: the mobile phase was a mixed aqueous solution of 1.3131 g·L⁻¹ phosphate buffer and methanol (95:5, v/v), and the flow rate of mobile phase was 1.2 mL·min⁻¹. The sample injection volume was 20 μL, the wavelength was set at 210 nm, and the column temperature was 25 °C. The chromatographic conditions for determination of acetophenone: the mobile phase was a mixture of methanol and double distilled water (50:50, v/v). The flow rate was 1.2 mL·min⁻¹, and the wavelength was 210 nm. Chemical

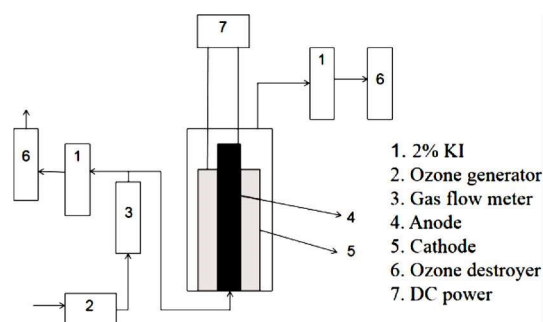


Figure 1 Schematic view of the experimental setup

oxygen demand (COD) was measured by the potassium dichromate method^[21]. The concentration of O₃ in gas was determined by an ozone detector (IDEAL-2000, IDEAL, USA). The concentration of dissolved ozone was measured by the indigo method^[22]. The concentration of H₂O₂ was determined by an ultraviolet spectrophotometer using the potassium titanium oxalate method^[23].

3 Results and Discussion

3.1 The Synergistic Effect of E-O₃ Under Acid Conditions

The removal rates of HAc by electrolysis, ozonation, and E-O₃ at pH 1 are compared, and the results are shown in Figure 2. Electrolysis and ozonation removed 2.2% and 2.4% of HAc after the 120-min treatment, respectively. The process of E-O₃ obtained 52.2% of HAc removal under the same conditions, much higher than the sum of those by electrolysis and ozonation. The results indicate that the combination of electrolysis and ozonation can lead to a good synergistic effect at pH 1.

3.2 Effect of Methanol on the Efficiency of E-O₃

Methanol was chosen as a radical scavenger to investigate the role of hydroxyl radicals in E-O₃ because HAc would be generated when tert-butanol reacts with hydroxyl radicals. As shown in Figure 3,

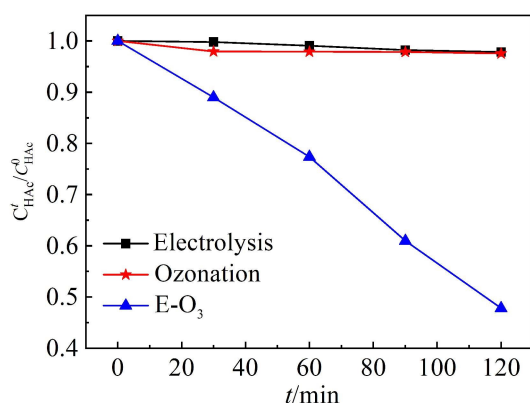


Figure 2 Degradation plots of HAc by electrolysis, ozonation, and E-O₃. Reaction conditions: initial pH of the solution: 1; electrolysis current: 2.0 A; amount of ozone application: 23.8 mg · min⁻¹; initial concentration of HAc: 100 mg · L⁻¹. (color on line)

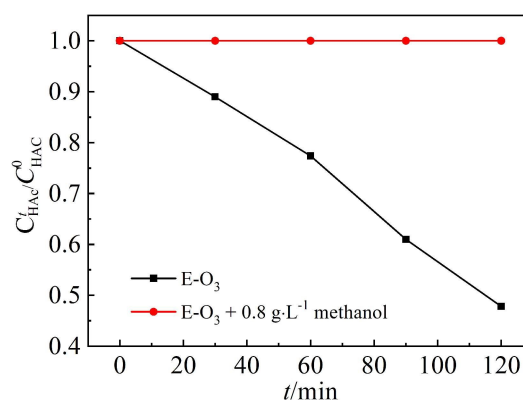


Figure 3 Effect of methanol on the degradation of HAc by E-O₃. Reaction conditions: initial pH of solution: 1; electrolysis current: 2.0 A; amount of ozone application: 23.8 mg · min⁻¹; initial concentration of HAc: 100 mg · L⁻¹. (color on line)

the degradation of HAc in E-O₃ system was almost completely inhibited after the addition of methanol, indicating that hydroxyl radicals should play an important role in this process because HAc is an ozone inert chemical.

3.3 Effect of Acidity on the Efficiency of E-O₃

The pH value of solution is an important factor for degradation of pollutant in E-O₃, which affects not only the existent state of a pollutant in the solution, but also the stability of dissolved O₃. In our previous work, the efficiencies of ozonation in the anode and cathode chambers for the degradation of nitrobenzene at neutral pH were investigated^[15]. The results verified that the synergistic effect of E-O₃ in the anode chamber was negligible compared with that in the cathode chamber, which was also proved by other literatures^[11,24]. It is well-accepted by most researchers that three pathways can explain the synergistic effect of E-O₃ at a cathode chamber: (1) generation of OH⁻ at the cathode; (2) production of H₂O₂ by reduction of O₂; (3) generation of ·O₃⁻ and ·O₂⁻ from reduction of O₃ and O₂^[11,15,24]. Our work has verified the order of importance in the three pathways at neutral pH: ·O₃⁻ and ·O₂⁻ > OH⁻ > H₂O₂^[15]. This work was carried out at very low pH (< 3), so both the pH increase at the cathode and the role of H₂O₂ should be negligible, which was well in accordance with other similar reports^[25-27].

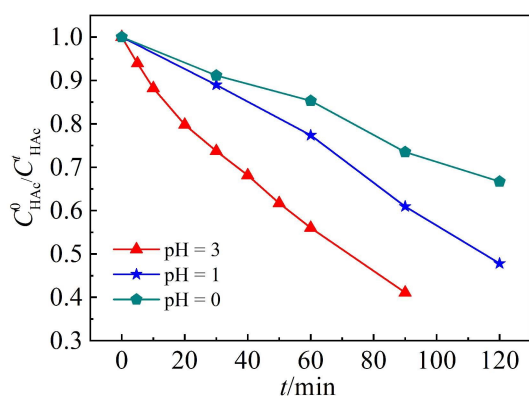


Figure 4 Effect of aqueous pH on the removal rate of HAC by E-O₃. Reaction conditions: electrolysis current: 2.0 A; amount of ozone application: 23.8 mg·min⁻¹; initial concentration of HAC: 100 mg·L⁻¹. (color on line)

However, as is shown in Figure 4, E-O₃ still had high efficiency at pH value less than 3. Therefore, it can be inferred that the role of $\cdot\text{O}_3^-$ and $\cdot\text{O}_2^-$ should play an important role for degradation of HAC at low pH.

The effect of pH on the efficiency of E-O₃ for the degradation of HAC is shown in Figure 4. The pK_a of HAC is 4.757 at 25 °C, thus, HAC is not ionized at the studied pH value. The removal rates of HAC by E-O₃ at pH values of 3, 1, and 0 were 58.9%, 39.1%, and 26.5% in 90 min, respectively. The decrease of pH led to the reduced efficiency of E-O₃, which might be attributed to two factors: (1) hydrogen evolution decreases the production rates of $\cdot\text{O}_3^-$ and $\cdot\text{O}_2^-$, (2) the competition consumption for $\cdot\text{OH}$ between HAC and

H⁺[28]. Nevertheless, the removal of HAC was still effective even at pH value of 0, indicating that $\cdot\text{OH}$ could be produced at an acceptable rate in E-O₃ system.

3.4 Effect of Current

Current is an important parameter in E-O₃[16,18,27]. Figure 5(A) and Figure 5(B) display the effect of current on the removal of HAC and the concentration of dissolved ozone in E-O₃ system, respectively. As the current increased in the range of 0 to 2.0 A, the efficiency of E-O₃ increased, but the concentration of dissolved ozone accordingly decreased. These results indicate that the oxidation efficiency of ozone is improved with the increase of electrolysis current, especially in the current range of 0 to 1.0 A. Therefore, the electrolysis current should be carefully controlled to achieve a cost-effective operation of E-O₃. This enhancement can be explained by the fact that more $\cdot\text{OH}$ can be produced from the faster reduction of O₃ and O₂ at the cathode as the current increases.

3.5 Degradation of Acetophenone by E-O₃ at pH 1

In order to further test the oxidative power of E-O₃ process for degradation of an aromatic compound, acetophenone was selected to be degraded by E-O₃ system at pH 1. As shown in Figure 6(A), the removals of acetophenone by electrolysis, ozonation and E-O₃ were 35.33%, 73.09%, and 100%, respectively. The COD removal of the acetophenone solution was measured to study the accumulation of by-products in the

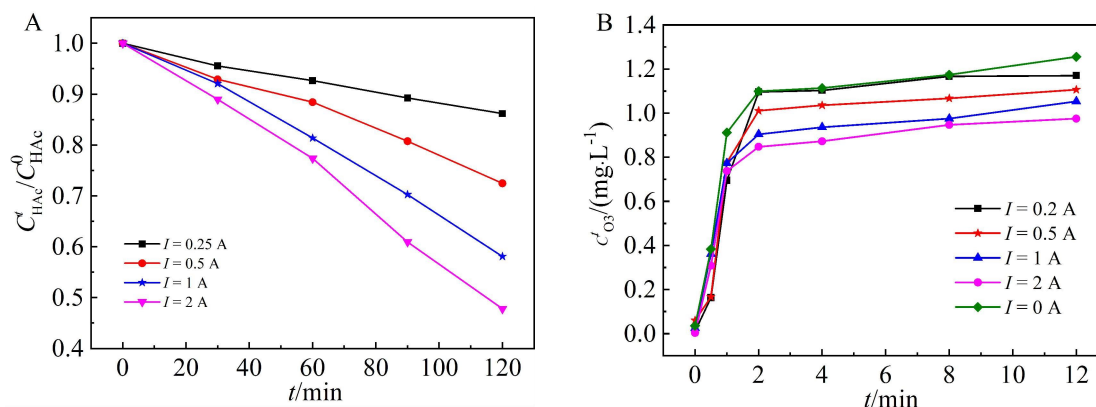


Figure 5 (A) Effect of current on the removal rate of HAC by E-O₃, (B) Change in concentration of dissolved O₃ at different electric currents in E-O₃. Reaction conditions: initial pH of solution: 1; amount of ozone application: 23.8 mg·min⁻¹; initial concentration of HAC: 100 mg·L⁻¹. (color on line)

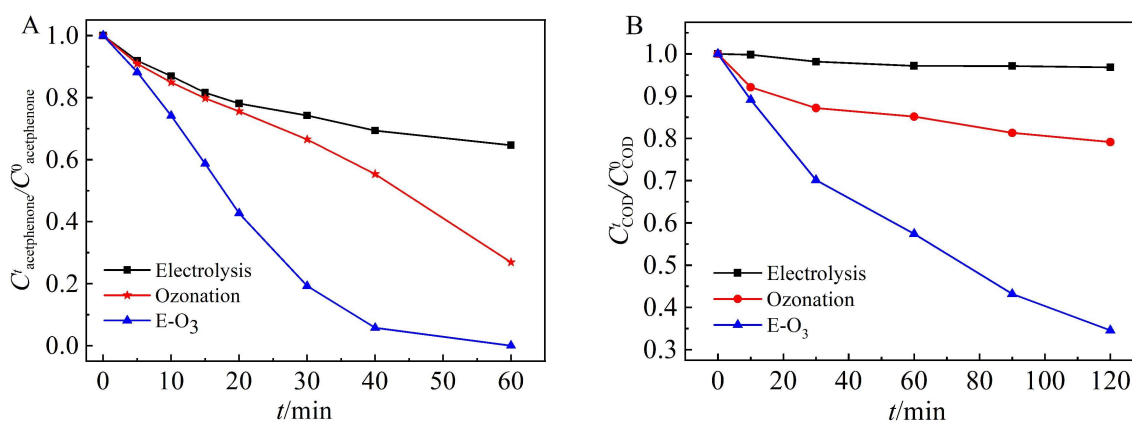


Figure 6 (A) Removal curves of acetophenone and (B) COD removal curves of acetophenone solution by electrolysis, ozonation, and E-O₃. Reaction conditions: initial pH of solution: 1; electrolysis current: 2.0 A; amount of ozone application: 23.8 mg·min⁻¹; initial concentration of acetophenone: 100 mg·L⁻¹. (color on line)

degradation of acetophenone. It can be seen from Figure 6(B) that the amounts of 3.17%, 20.87%, and 65.43% of COD were removed by electrolysis, ozonation and E-O₃, respectively. Acetophenone can be removed by single ozonation, but the COD removal is limited owing to the selective oxidative power of ozone. The percentage of COD removal by the process of E-O₃ was high, indicating the by-products of acetophenone degradation could also be effectively removed in E-O₃.

3.6 Treatment of an Acidic Wastewater

Acetic acid is widely used in medicine, fuel, spices, pesticides and other industries, thus, some acid wastewater sometimes contains high concentration of acetic acid. An acidic wastewater from Hangzhou Flente Fine Chemical Co. Ltd. was selected to test the effectiveness of E-O₃ for practical application. Besides the main component of acetic acid, the acidic wastewater also included some indole derivatives such as 1,2-diphenyl indole, 1-isopropyl-2-phenyl indole, 2-(2-naphthalene) indoles, 2-(3-thiophene) indoles et al. On the basis of the optimized conditions of E-O₃, the acidic wastewater was diluted first before experiments, and the initial COD of the diluted acidic wastewater was 1560 mg·L⁻¹, and the pH was 1.15. The COD removal rate curves of the wastewater by electrolysis, ozonation and E-O₃ are shown in Figure 7. After the 120-min of treatment,

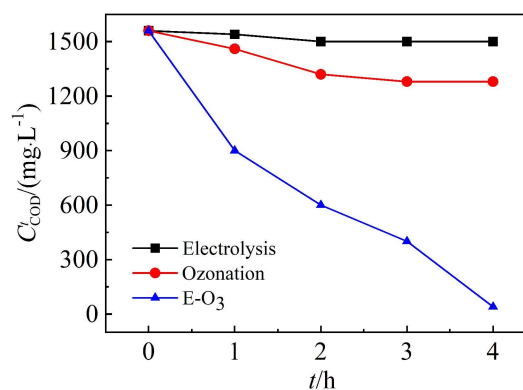


Figure 7 COD removal curves of an actual acidic wastewater by electrolysis, ozonation and E-O₃. Reaction conditions: initial pH of solution: 1.15; electrolysis current: 2 A; amount of ozone application: 23.8 mg·min⁻¹. (color on line)

60 mg·L⁻¹, 280 mg·L⁻¹, and 1520 mg·L⁻¹ of COD were removed by electrolysis, ozonation, and E-O₃, respectively. Obviously, E-O₃ has high efficiency in treatment of a real acidic wastewater.

4 Conclusions

A combined process of ozonation coupled with electrolysis was used to degrade acetic acid and acetophenone in acid solutions, and an actual acidic wastewater as well. The effects of different parameters on the efficiency of E-O₃ were also studied. The results are summarized as follows: (1) The combination of ozonation and electrolysis resulted in a significant synergistic effect in an acidic solution. (2) Hy-

droxyl radicals ($\cdot\text{OH}$) could be effectively produced in the pH range of 0 ~ 3 in the process of E- O_3 . (3) Electrolysis current should be carefully optimized to achieve an economical operation.

Acknowledgements

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电解耦合臭氧氧化体系处理酸性废水的氧化效能

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摘要: 建立能有效处理酸性废水的臭氧类高级氧化技术(AOPs-O₃)是一个有待解决的难点。已有报道表明, 臭氧氧化与电解结合(电解臭氧化, E-O₃)可以有效降解中性溶液中的污染物。本文研究了 E-O₃ 在酸性溶液中降解乙酸(HAc, 臭氧惰性物)的效率, 发现 E-O₃ 在 pH 小于 3 时仍具有较高的氧化效率, 如在 pH 为 1.0 时处理 100 mg·L⁻¹ 乙酸溶液 2 小时后 E-O₃ 的效率达到 52.2%, 而相同条件下电解和臭氧氧化的效率分别只有 2.2% 和 3.5%。尽管酸度增加会降低 E-O₃ 的氧化效率, 但在 pH 等于 0 时其仍有相对较高的氧化效率。芳族化合物苯乙酮在 pH 等于 1.0 条件下也能被 E-O₃ 有效地降解并矿化。机理解析表明, 溶解臭氧或氧气可以从阴极获得电子, 从而产生高活性的氧化物种, 如羟基自由基。在预处理了一种实际酸性废水中 E-O₃ 也具有较好的效率。本研究为酸性废水的有效(预)处理提供了一种新方法。

关键词: 电解; 臭氧; 乙酸; 效率; 酸性废水