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EFFECT OF RADICAL SCAVENGERS AND PROPOSED PATHWAYS FOR AZO DYE DEGRADATION IN A PERSULPHATE-BISULFITE SYSTEM

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ВЛИЯНИЕ АКЦЕПТОРОВ РАДИКАЛОВ И СХЕМА ДЕСТРУКЦИИ АЗОКРАСИТЕЛЯ В СИСТЕМЕ ПЕРСУЛЬФАТ-БИСУЛЬФИТ

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Abstract

Introduction. The textile industry is one of the most significant manufacturing sectors that produce large volumes of highly polluted and toxic wastewater. Along with the light industry, pigments and dyes industry, domestic service, dry-cleaning, etc., it contributes significantly to water pollution, where dyes are among the top pollutants. **Methods.** In this study, the degradation of Acid Orange 7 (AO7) dye in a persulfate-bisulfite system under visible (Vis) light (wavelength ≥ 420 nm) was performed. Based on the electron spin resonance spin-trapping technologies and radical scavenger measurements, the produced hydroxyl radical (·OH) is regarded as the predominant reactive oxidant for AO7 decolorization also involving the sulfate radical ($SO_4{}^{\cdot-}$). The formation of short-lived radicals during AO7 decolorization was detected by the ESR spin-trapping technique at room temperature using a Bruker ESR A-300 spectrometer with the following parameters: center field 3516 G, sweep width 100 G, microwave frequency 9.86 G, modulation frequency 100 kHz, microwave power 1 mW. The intermediate products of the AO7 degradation reaction were analyzed by mass spectrometry. The experiments were performed using an Esquire LC ion trap mass spectrometer (Bruker Daltonics, Bremen, Germany) equipped with an orthogonal geometry ESI source. Nitrogen was used as a drying (3 L/min) and nebulizing (6 psi) gas at 300 °C. The spray shield voltage was 4.0 kV and the capillary cap voltage was 4.5 kV. Scanning was performed from m/z 90 to 400 in the standard resolution mode at a scan rate of 13 kDa/s. Before the analysis, each sample was diluted ten-fold. The intermediates were determined by electrospray ionization-mass spectrometry (ESI-MS) analysis and, as a result, a plausible degradation pathway was proposed. **Results.** The results of the study can be useful in designing a simple, effective, and economically sound system for the treatment of non-biodegradable azo dyes.

Keywords: dye degradation, Acid Orange 7, radical scavengers, reaction intermediates, degradation pathway, mineralization.

Аннотация

Введение. Текстильная промышленность принадлежит к одной из наиболее важных отраслей производства, которые являются источником больших объемов сильно загрязненных и токсичных сточных вод. Наряду с легкой промышленностью, производством пигментов и красителей, сферой бытового обслуживания, химчисткой и т.д., она вносит значительный вклад в загрязнение водной среды, в которой красители рассматриваются как одни из главных загрязняющих веществ. **Методы.** В данном исследовании изучен процесс деструкции красителя кислотного оранжевого 7 (КО7) в системе персульфат-бисульфит при облучении видимым светом (длина волны ≥ 420 нм). С помощью использованного нами метода электронного спинового резонанса и детектирования акцепторов радикалов установлено, что образующийся гидроксильный радикал (·OH) можно рассматривать в качестве основного реакционно-способного окислителя для обесцвечивания КО7 с участием в процессе сульфатного радикала ($SO4^{\cdot-}$). Образование короткоживущих радикалов в процессе обесцвечивания КО7 детектировали методом ESR спинового улавливания при комнатной температуре с использованием спектрометра Bruker ESR A-300 со следующими характеристиками: центральное поле 3516 G, ширина развертки 100 G, микроволновая частота 9,86 G, частота модуляции 100 кГц, микроволновая мощность 1 мВт. Определение промежуточных продуктов процесса деструкции КО7 проводили масс-спектрометрически. В ходе эксперимента использовали масс-спектрометр с ионной ловушкой Esquire LC (Bruker Daltonics, Бремен, Германия), оснащенный источником ESI с ортогональной геометрией. Азот использовали в качестве осушающего (3 л/мин) и распыляющего (6 фунтов/кв. дюйм) газа при температуре 300 °C. Напряжение защитного экрана составляло 4,0 кВ, а напряжение капиллярного колпачка было равно 4,5 кВ. Сканирование проводили при значениях отношения m/z (масса/заряд) в интервале от 90 до 400 в режиме стандартного разрешения со скоростью сканирования 13 кДа/с. Перед проведением анализа каждый образец разбавляли в десять раз. **Результаты.** Промежуточные продукты реакции определяли с помощью метода масс-спектрометрии с ионизацией электрораспылением (ESI-MS), и в итоге предложена вероятная схема процесса деструкции. Результаты исследования могут быть полезны при разработке не сложной, эффективной и экономически обоснованной системы обработки стоков от не биоразлагаемых азокрасителей.

Ключевые слова: деструкция красителя, краситель кислотный оранжевый 7, акцепторы радикалов, промежуточные продукты реакции, путь деструкции, минерализация.

Introduction

Earlier, the authors of the paper performed degradation of Acid Orange 7 (AO7) dye in a persulfate-bisulfite system under visible (Vis) light (wavelength ≥ 420 nm). Activities of different systems including HSO_3^-/Vis, HSO_3^-/PS, PS/Vis and HSO_3^-/PS/Vis were evaluated. It was found that the efficiency of PS/Vis system was negligible, and almost 29 and 40 % of AO7 were oxidized by HSO_3^-/Vis and HSO_3^-/PS, respectively, within 30 min; that was further improved to almost 94 % within 30 min in the HSO_3^-/PS/Vis system at room temperature. Various operational parameters, such as PS concentration, HSO_3^- concentration and initial pH, were investigated to optimize the process.

In previous reports, free radicals, such as the generated hydroxyl radical (·OH) and sulfate radical ($SO_4^{\cdot -}$), were thought to be produced during the reaction between HSO_3^- and PS [5, 12, 14]. In this study, primary reactive oxidants and final degradation products of AO7 in the HSO_3^-/PS/Vis process were investigated to propose AO7 degradation pathways.

Methods and materials

Acid Orange 7 (AO7, $C_{16}H_{11}N_2NaO_4S$) dye was purchased from Aladdin Industrial Corporation. Sodium hydrogen sulfite ($NaHSO_3$), sodium persulfate ($Na_2S_2O_8$), tert-Butyl alcohol (TBA, AR), ethanol (EtOH, AR) and other chemicals were analytically pure, if not noted otherwise, and were obtained from Sinopharm Chemical Reagent Co., Ltd. Acetonitrile was supplied by Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China) but it was of the HPLC grade. Spin trap reagent 5,5-dimethyl-1-pyrroline-N-oxide (DMPO) was purchased from Sigma Chemical Co. All chemicals were used as received without further purification. Sample solutions were prepared using deionized water (Aquapro, USA) throughout the experiments.

Degradation procedures

All batch experiments were conducted in a 100 mL undivided glass beaker containing 50 mL solution at about 25 °C with or without exposure to Vis light irradiation (CHF-XM-500W, Beijing Trusttech Co., China). After the desired amounts of AO7 and PS in the 50 mL aqueous solution were added into the reactor, the reaction was initialized by adding HSO_3^-. Each reaction solution was constantly agitated by a magnetic stirrer (model 78-1, Hangzhou Instrument Motors Factory, China).

A stock solution of AO7 with the initial concentration (C_0) of 20 mg/L was prepared freshly with deionized water before each run. Since PS is an acidic oxidant, the addition of PS led to a significant decrease of pH, and the experiment was conducted in the acidic medium (pH 4.75, no adjustment). Before the beginning of the reaction, the PS solution and HSO_3^- solution were added to the reactor.

Analysis

The AO7 degradation process was monitored by a METASH UV-5500PC spectrophotometer at the maximum absorption wavelength (484 nm). Solution samples were taken at predetermined time intervals and measured immediately to record temporal UV–vis spectral variations of the dye. The related reaction progress was monitored using the UV-vis absorption spectra of AO7 in terms of the percentage of the degraded azo dye, which was calculated based on the following equation (1):

$$\text{Decolorization efficiency (\%)} = (C_0 - C_t) / C_0 \times 100 \quad (1)$$

where C_t is the absorbance at 484 nm for AO7 at a given reaction time t, and C_0 is the related initial absorbance.

The formation of short-lived radicals during AO7 decolorization was detected by the ESR spin-trapping technique at room temperature using a Bruker ESR A-300 spectrometer with the following parameters: center field 3516 G, sweep width 100 G, microwave frequency 9.86 G, modulation frequency 100 kHz, microwave power 1 mW.

The intermediate products of the AO7 degradation reaction were analyzed by mass spectrometry. The experiments were performed on an Esquire LC–ion trap mass spectrometer (Bruker Daltonics, Bremen, Germany) equipped with an orthogonal geometry ESI source. Nitrogen was used as a drying (3 L/min) and nebulizing (6 psi) gas at 300 °C. The spray shield voltage was 4.0 kV and the capillary cap voltage was 4.5 kV. Scanning was performed from m/z 90 to 400 in the standard resolution mode at a scan rate of 13 kDa/s. Before the analysis, each sample was diluted ten-fold.

Results and discussion

Radical detection

To understand the reaction mechanism of the persulfatebisulfite system, the formation of short-lived radicals during AO7 decolorization was detected by the ESR spin-trapping technique. ESR

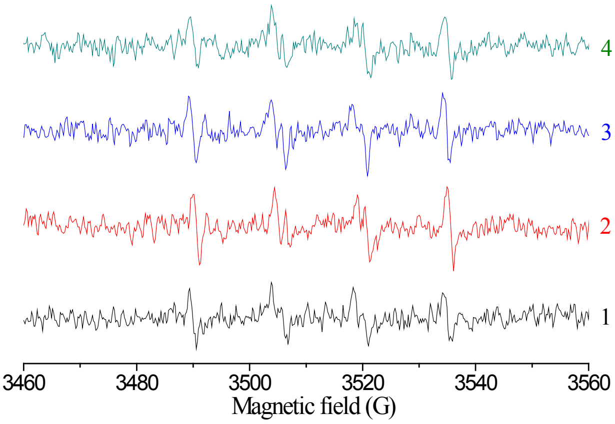

Fig. 1. DMPO spin-trapping ESR spectra at different time intervals (min): (1) 0, (2) 5, (3) 10, (4) 20.

spectra measured during the reaction are shown in Figure 1. An ESR spectrum of a sulfur trioxide anion radical adduct with the hyperfine coupling constants N = 14.7 G and H β = 16.0 G was observed in the experiments with persulfate, bisulfite, and DMPO under Vis light, similar to the reported rectorite-sulfite system [13]. The intensity of $SO_3^{\cdot-}$ adduct first increases rapidly (5 min) and then slowly decreases within 20 min, implying the $SO_3^{\cdot-}$ adduct transformation into other species.

Effect of radical scavengers

To evaluate the contribution of the oxidizing species $SO_4^{\cdot-}$ and ·OH, two kinds of radical scavengers were used: ethanol (EtOH) and tert-butyl alcohol (TBA). As indicated in Figure 2, the addition of 1 M EtOH almost suppressed the oxidation process. EtOH contains α-hydrogen, which can rapidly quench both $SO_4^{\cdot-}$ and ·OH. The reaction constants for EtOH with $SO_4^{\cdot-}$ and ·OH are $1.6–7.7\times10^7$ M^{-1} s^{-1} and $1.2–2.8\times10^9$ M^{-1} s^{-1}, respectively [6]. Thus, the presence of EtOH led to the consumption of reactive radicals and removal efficiency decrease.

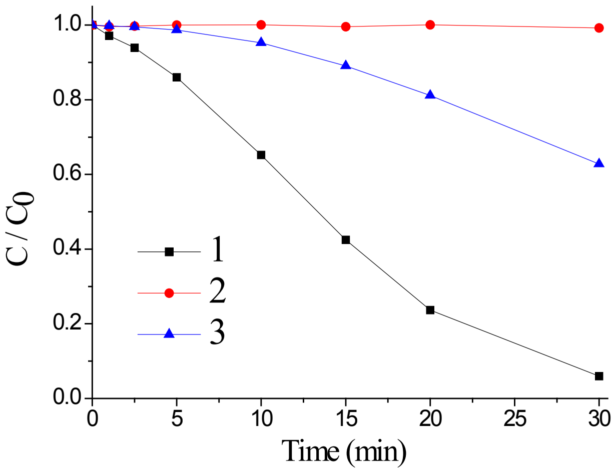

Fig. 2. Effect of radical scavengers: (1) none, (2) EtOH, (3) TBA

According to literature data, ·OH can be generated via the reaction between $SO_4^{\cdot-}$ and water as follows [9]:

$$SO_4^{\cdot-} + H_2O \rightarrow HSO_4^- + \cdot OH \qquad (2)$$

To investigate the contribution of ·OH to the AO7 removal in the HSO_3^-/PS/Vis process, TBA was introduced. TBA can quickly quench ·OH with the reaction constant $3.8–7.6\times10^8$ M^{-1} s^{-1}, which is much more than the rate constant of TBA with $SO_4^{\cdot-}$ ($4.0–9.1\times10^5$ M^{-1} s^{-1}). Hence, TBA is generally used as an ·OH scavenger [5]. Figure 2 shows that the addition of 1 M TBA inhibited the oxidation process significantly and the AO7 removal efficiency decreased from 94 to 37 % within 30 min.

Compared with the control experiment, the addition of 1 M TBA resulted in 57 % AO7 decay drop, while the addition of 1 M EtOH almost inhibited the degradation process. These results provide evidence that ·OH radicals are the major radical species in the persulfate-bisulfite system in the acidic medium; $SO_4^{\cdot-}$ radicals are also involved but their contribution is minor compared with that of ·OH radicals.

Photodegradation products and proposed process pathways

To analyze the process of AO7 degradation in a persulfate-bisulfite system under Vis light, we attempted to identify main reaction intermediates. Direct-infusion electrospray ionization mass spectrometry (ESI-MS), which has been the technology of the choice for detection and identification of reactants, products, and intermediates in a solution [7], was applied to identify intermediate products during AO7 degradation in the HSO_3^-/PS/Vis system.

Figure 3 shows representative spectra in the negative ion mode at time intervals from 0 to 60 min. The molecular weight of AO7 was 350. Its corresponding structure is shown in Figure 5. In an aqueous solution, RSO_3Na groups in the dye dissociate into RSO_3^- and Na^+ ions. At the beginning of the reaction, an intense ion of m/z 327

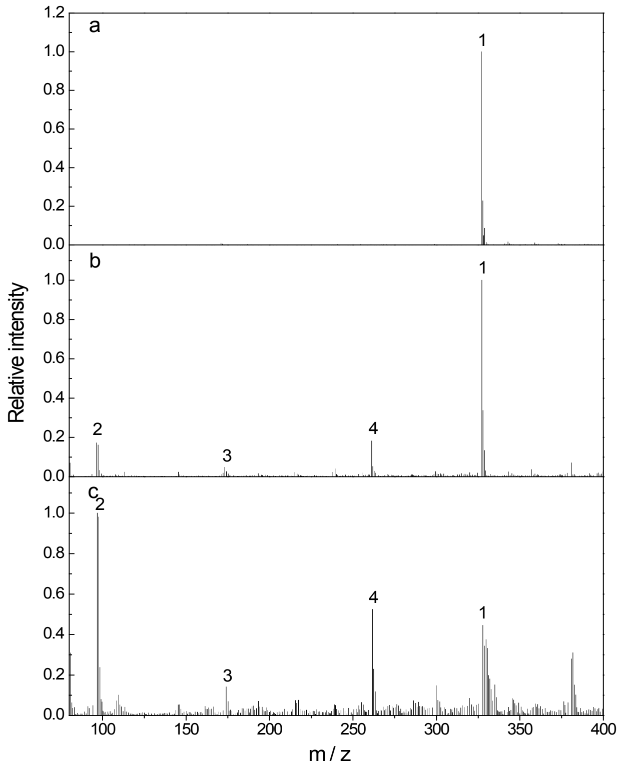

Fig. 3. ESI (−) mass spectra of AO7 solution during degradation in the Vis/HSO_3^-/PS system at different time intervals (min): (a) 0, (b) 30, (c) 60. Peak values: (1) 327, (2) 97, (3) 173, (4) 261

corresponding to AO7 $[M–Na]^-$ was observed as expected. After 60 min treatment, the intensity of AO7 at m/z 327 decreased significantly and signal peaks at m/z 97, 173 and 261 occurred, indicating that AO7 was degraded into some intermediate products. According to the values of m/z (173) and the suggested structures, this product can be regarded as p-phenolsulfonic acid (p-PSA), which is a common product of AO7 destruction, as was reported previously [1–4, 8, 11, 15–19]. The intermediate product can then be continually oxidized to ions of m/z 97 and 261, which may be a result of ring-opening reactions. With the reaction proceeding, AO7 was further degraded to the mentioned products as the relative intensity of these ions increased after 60 min.

The positive ionization mode was also tested to detect positively charged species. The corresponding results are shown in Figure 4. At the beginning of the reaction, an intense ion of m/z 373 corresponded to AO7 $[M+Na]^+$, which was identical to the result of $[M–Na]^-$ at m/z 327. After 30 min of incubation, new peaks at m/z 165, 215 and 257 were observed. The ions of m/z 215 and 165 can be attributed to the oxidized compounds of 1,2-naphthoquinone. Besides, m/z 257 can be a product of radical attack and further oxidation of the ion m/z 261, 165. It should be noted that the observation of ions after a long reaction time does not mean their later appearing as the detection of ions by ESI-MS is related not only to the concentration but the degree of ionization as well.

Therefore, a reaction sequence for AO7 degradation in the HSO_3^-/PS/Vis system was proposed based on the analyses of the ESI-MS data. It is shown in Figure 5. Firstly, an initial successive radical attacked the N atom of the dye, which decomposed to p-PSA and oxidized products of 1,2-naphthoquinone. Then, subsequent oxidation of these products and loss of the $-SO_3^-$ group occurred. Finally, acyclic carboxylic acids formed from the central ring-opening, and then they were further oxidized to simple carboxylic acids. Obviously, this proposed partial pathway only depended on ESI-

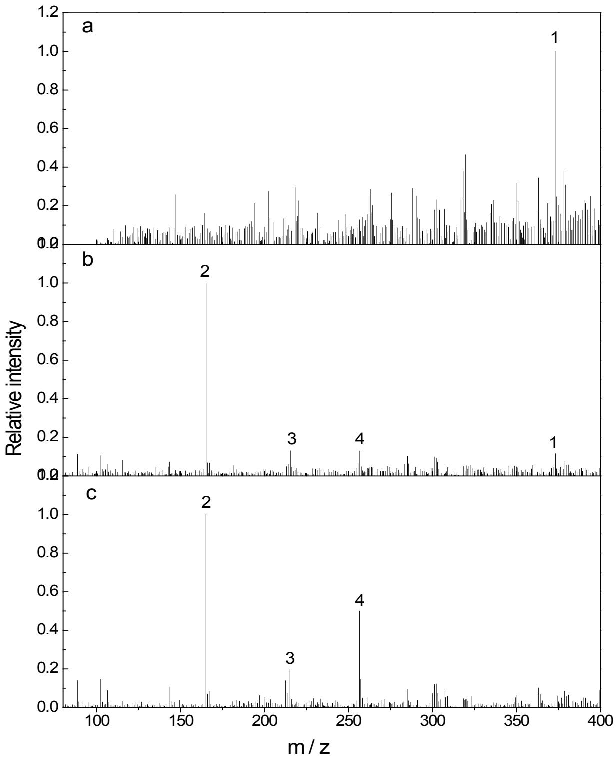

Fig. 4. ESI (+) mass spectra of AO7 solution during degradation in the Vis/HSO_3^-/PS system at different time intervals (min): (a) 0, (b) 30, (c) 60

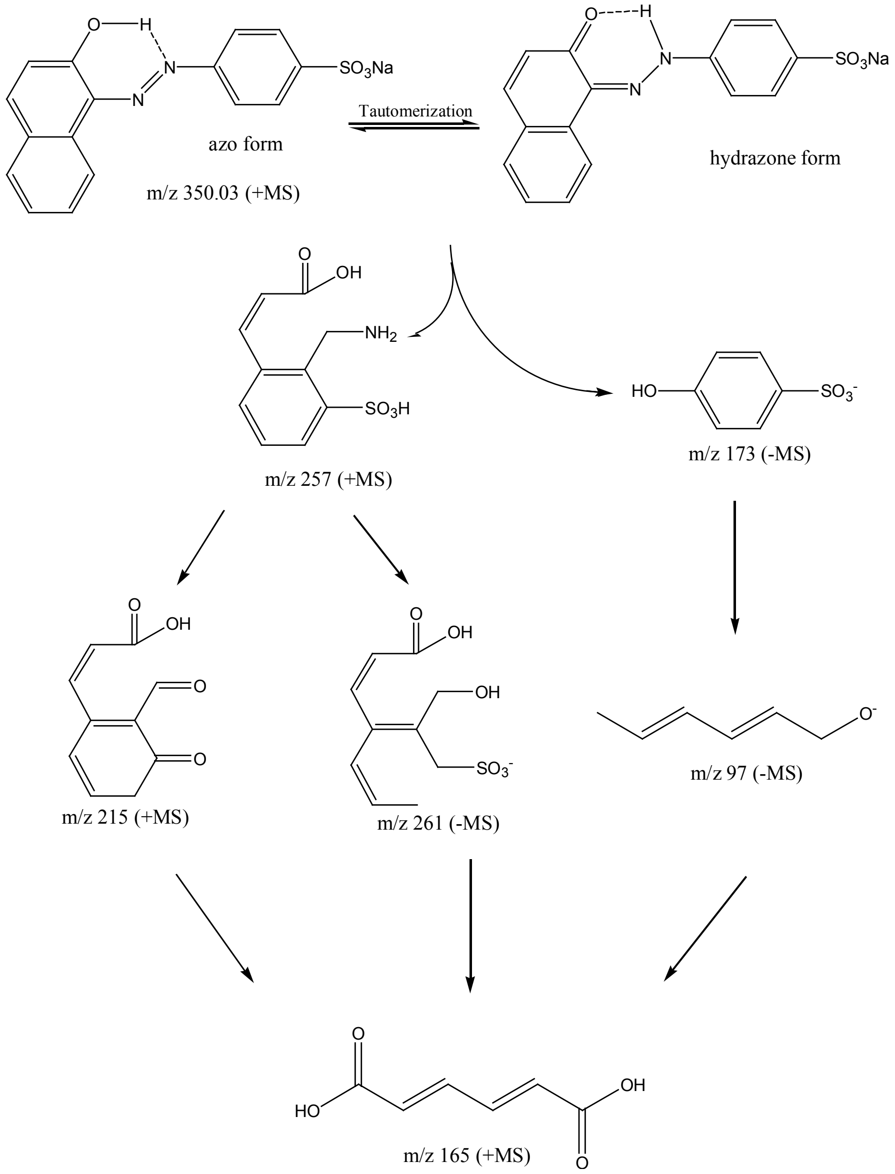

Fig. 5. Scheme of a proposed pathway for AO7 degradation in the Vis/HSO_3^-/PS system

MS and relevant literature, and it still needs to be investigated systematically.

Arrangement of effective quality control over the discharge of process water and products of its treatment at local wastewater treatment facilities of industrial plants prevents hazardous biological substances from entering the hydrosphere. Such quality control is especially important if treated wastewater and wastewater processing products are re-used [10].

Peak values: (1) 373, (2) 165, (3) 215, (4) 257.

Conclusion

The mechanism of AO7 degradation in the HSO_3^-/PS/Vis treatment process was investigated, with a particular focus on AO7 degradation pathways. The presence of EtOH and TBA hindered AO7degradation, indicating that ·OH radicals are the major radical species participating in the AO7 decolorization process, where $SO_4^{\cdot -}$ is also involved. Based on the ESI-MS analysis of the degradation intermediates, the pathway of AO7 degradation in the HSO_3^-/PS/Vis system was proposed. The C-N bonds of AO7 were attacked by the radicals, and smaller compounds, such as p-PSA and simple carboxylic acids, +formed.

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