



Technical Note

Photodegradation of Acid Orange 7 in a UV/acetylacetone process



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HIGHLIGHTS

- AcAc was used as a photo-activator for the photodecoloration of AO7.
- The UV/AcAc process was more effective for AO7 decoloration than the UV/H₂O₂ method.
- The UV/AcAc process significantly improved the biodegradability of the AO7 solution.
- A self-acceleration phenomenon was observed for the UV/AcAc process.
- The possible pathways of AO7 degradation in the UV/AcAc process were proposed.

GRAPHICAL ABSTRACT

The UV/acetylacetone process is highly efficient for the decoloration of azo dyes.



ARTICLE INFO

Article history:

Received 10 April 2013

Received in revised form 28 August 2013

Accepted 29 August 2013

Available online 27 September 2013

Keywords:

Acetylacetone

Acid Orange 7

Photodecoloration

Mechanism

ABSTRACT

Acetylacetone (AcAc) was employed as a photo-activator for the degradation of Acid Orange 7 (AO7) under UV irradiation. The feasibility of this process (named as UV/AcAc) was evaluated through comparison with the well-established UV/H₂O₂ process in terms of absorption spectrum and the biodegradability of the solutions. A complete decoloration of the AO7 solution could be fulfilled with AcAc at mM level. A self-acceleration phenomenon was observed for the UV/AcAc process. The pseudo first-order decoloration rate constant of AO7 in the UV/AcAc process was several times higher than that in the UV/H₂O₂ process, depending on the irradiation conditions. The BOD to COD ratio of the solutions increased from below 0.1 to above 0.3, along with a slight mineralization. Based on degradation product analysis, the possible pathways for AO7 degradation in the UV/AcAc process were proposed.

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1. Introduction

The pollution of textile wastewater is becoming increasingly alarming with the extensive use of synthetic dyes. Within the overall category of dyestuffs, azo dyes constitute a significant portion and have caused much concern in terms of the surrounding ecosystem (Richardson, 1983). Due to their recalcitrant and inhibitory nature, azo dyes are normally not amenable for conventional biological wastewater treatment techniques. Many biodegradability studies have shown that they are largely non-biodegradable in aerobic conditions and are reduced to more hazardous intermediates in anaerobic conditions (Pagga and Brown, 1986; Weber and

Adams, 1995; Knackmuss, 1996). Hence, an attractive potential alternative has been proposed to combine a chemical oxidation with the conventional biological treatment (Oller et al., 2011). Chemical oxidation can be applied as a pre-treatment to convert the initially persistent organic pollutants to more biodegradable intermediates, which can then be treated in a biological treatment process at a considerably low cost.

Advanced oxidation processes (AOPs) based on the generation of highly reactive hydroxyl radicals are widely recognized as efficient treatments for recalcitrant wastewaters. Fenton-like reactions, UV/O₃, UV/H₂O₂, and UV/TiO₂ are well known examples of AOPs with ·OH as the main oxidizing species (Bandara et al., 1996; Sarasa et al., 1998; Fernandez et al., 1999; Styliidi et al., 2004). These AOPs are effective for destroying chemically stable or bio-refractory contaminants, such as aromatics, pesticides, and dyes. A comparative

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evaluation of these methods has shown that the UV/H₂O₂ process has a number of advantages over the other photochemical methods: minimal capital investment, simple to operation, no mass transfer limitations associated with gases or catalysts, requires very little human manipulation and maintenance. Nevertheless, the small absorption cross-section of H₂O₂ at the UV band is a real disadvantage and dyes can act as inner filters to H₂O₂, thus a high dosage of H₂O₂ is usually needed (Neamtu et al., 2002).

Neevel et al. (1990) demonstrated photobleaching of azo dyes in UV irradiated diacetyl (2,3-butanedione) solutions. We found that acetylacetone (2,4-pentanedione, denoted as AcAc) was also effective for the photodegradation of dyes. To the best of our knowledge, use of AcAc as a photo-activator for water treatment has not been reported in the literature. The main focus of the present work is to evaluate whether we can use AcAc as a photo-activator for the degradation of dyes. Acid Orange 7 (AO7) was chosen as a model contaminant, since it is nonbiodegradable and is widely used in the textile, food, and cosmetics industries (Mendez-Paz et al., 2005; Mu et al., 2009). The effectiveness of the UV/AcAc process was validated through the comparison with the UV/H₂O₂ process. The structures and the conversion of the intermediates in the investigated process were identified. In addition, a possible degradation pathway was proposed.

2. Materials and methods

2.1. Materials

AO7, AcAc, H₂O₂, isopropanol, and tert-butanol of analytical grade were purchased from Shanghai Reagent Station. Methanol and formic acid of chromatographically pure grade, and 5,5'-dimethyl-1-pyrroline-N-oxide (DMPO) of analytical grade were purchased from Sigma–Aldrich and were used as received. Ultrapure water was used for the preparation of all aqueous solutions.

2.2. Irradiation experiments

Degradation experiments were carried out in a rotating disk photoreactor (Nanjing StoneTech Electric Equipment, China). A medium pressure mercury lamp (200 or 300 W) with a maximum light emission at 365 nm was vertically placed in a cooling water jacket. Sample solutions containing 0.16 mM AO7 and 0.5–100 mM AcAc or H₂O₂ were parallelly arranged in a sample holder around the lamp. The distance between the sample tube and the lamp was 5 cm. The initial solution pH was found to be 6.7 ± 0.2 and was adjusted with HCl or NaOH as needed. During irradiation, the sample holder revolves around the lamp and the tubes rotate.

For dissolved oxygen effect experiments, air or N₂ (99.9%) were purged during the irradiation process.

2.3. Analytical methods

The color fading of the dye solution was analyzed with a double beam spectrophotometer (UV-2550, Shimadzu, Japan). The total organic carbon (TOC) was determined on a Multi N/C TOC apparatus (TOC-L, Shimadzu, Japan). The chemical oxygen demand (COD) was determined with the closed reflux, titrimetric method.

Liquid chromatography–mass spectrometry (LC–MS) was performed using a ThermoFinnigan LCQ Advantage MAX mass spectrometer equipped with an electrospray ionization (ESI) interface source operated in a negative mode. The LC system was equipped with an Agilent 4.6 × 150 mm, 5 μm ZORBAX Eclipse Plus C18 column, and the flow rate was set as 0.2 mL min⁻¹. An aliquot of 10 μL of sample was injected by using an auto sampler. The UV

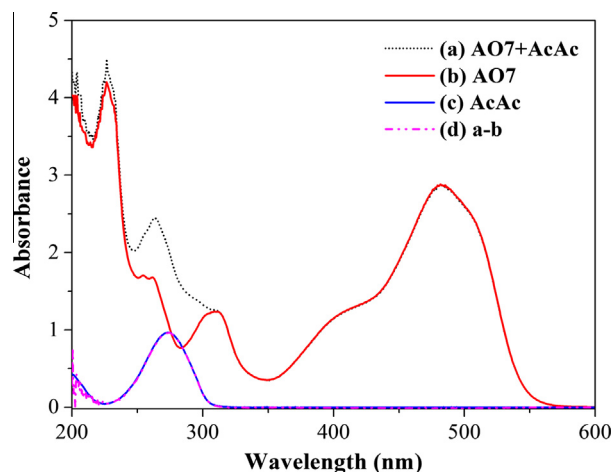


Fig. 1. The UV/Vis spectra of AO7 (0.16 mM), AcAc (0.5 mM), and their mixture.

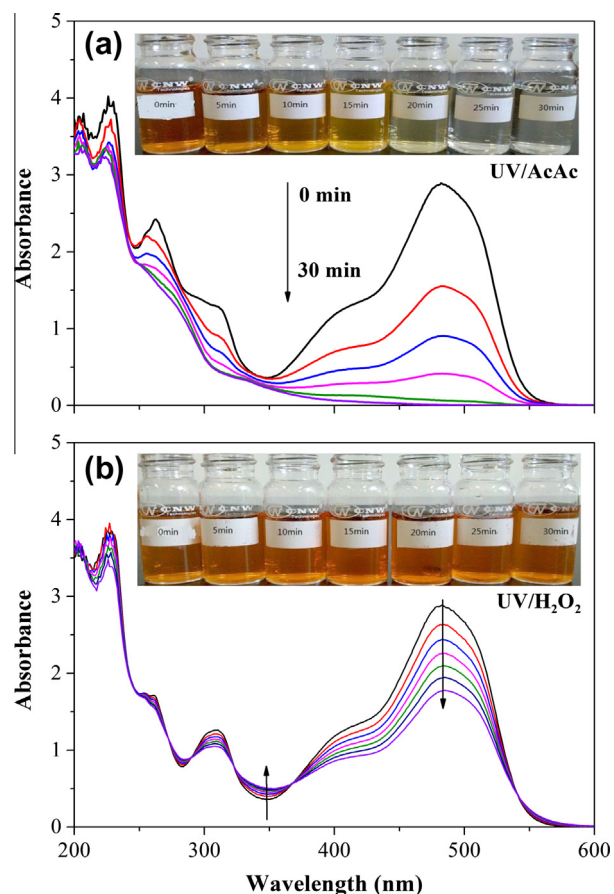


Fig. 2. Evolution of the UV-Vis spectra of AO7 solutions (0.16 mM) under UV irradiation (light intensity: 7.1 mW cm⁻²) in the presence of 0.5 mM AcAc (a) or H₂O₂ (b).

detector was operated at 228, 254, 310, 484 nm. The eluent from the chromatographic column successively entered the photo diode array detector, the ESI interface and the mass analyzer.

MS settings were as follows: capillary temperature 300 °C, spray voltage 4.5 kV, sheath gas flow 35 and auxiliary gas 5 (arbitrary units), capillary voltage 25 V, and tube lens offset 100 V. Data were recorded in the mass to charge (*m/z*) range of 50–550.

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