



Research article

Photo- and chemocatalytic oxidation of dyes in water



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ABSTRACT

Three commonly used dyes, Acid Red-114 (AR-114), Reactive Black-5 (RB-5), and Disperse Black EX-SF (DB-EX-SF), were treated in a pH-neutral liquid with ultraviolet (UV) light by two reactive methods: photocatalysis with titanium dioxide (TiO₂), and/or chemocatalysis with hydrogen peroxide (H₂O₂) as the oxidant and various ferrous-based electron mediators as catalysts. Important factors for dye oxidation were determined through bifactorial experiments. The optimum combinations and doses of the three key reagents, namely TiO₂, H₂O₂, and EDTA-Fe, were also determined. The degradation kinetics of the studied dyes at their optimum doses reveal that the oxidation reactions are pseudo-first-order in nature, and that certain dyes are selectively degraded more by one method than the other. The overall results suggest that co-treatment using more than one oxidative method is beneficial for the treatment of wastewater from dyeing processes.

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1. Introduction

The modern uses of dyes are rather diverse (Metwally et al., 2012; Sayed et al., 2012). Dye selection usually takes into account factors such as color preference, type of fiber, and operating processes, among others. Dyes vary considerably in physical and chemical characteristics, water solubilities, and biodegradabilities. As dye types continue to expand, the treatment of dyes in wastewater has also become more complicated. Various oxidative methods have been demonstrated to be effective for the treatment of a variety of dyes (Aleboyyeh et al., 2012; Colindres et al., 2010; Gulkaya et al., 2006; Wu and Wang, 2012); however, a single dye is rarely treated by more than one oxidative method (i.e., co-treatment), which is of interest to this study.

Chemical oxidation is considered to be the most efficient means of treating bioresistant substances (An et al., 2010). This process uses an oxidant such as a peroxide, and a catalyst such as iron(II) and/or metal chelates (Park et al., 2006). During the chemical oxidation process, the carbon atom(s) of the target organic pollutants are eventually transformed into carbon dioxide. This treatment method has been used to remove numerous xenobiotic substances and dyes from wastewater (Chen et al., 1999, 2009;

Christoforidis et al., 2010). Photocatalytic oxidation with (titanium dioxide) TiO₂ (Cojocariu et al., 2010; Friedmann et al., 2010; Kim and Choi, 2010; Mansilla et al., 2006; Seshadri et al., 2008; Vohra and Davis, 2000; Yamazaki-Nishida et al., 1996; Zhou et al., 2009) is another oxidative method for treating xenobiotic substances in wastewater (Liang et al., 2012; Pupo Nogueira and Guimarães, 2002). In this process, a light source is used to drive electrons from the target substance to specific electron acceptors, including oxygen. New electrospun-graphene-oxide nanocomposites (Hou et al., 2017), graphene-oxide-based hydrogels (Guo et al., 2015), and polydopamine/graphene-oxide-based composites (Xing et al., 2017), as well as hierarchical porous silver-based (Hou et al., 2016) and diamond-based (Zhao et al., 2017) nanocomposites are under development for the degradation of dyes.

In field-based applications, the treatment of dyes in wastewater typically uses only one type of treatment method (i.e., either photo- or chemo-oxidation) for the removal of dyes; combinations of different oxidation processes are rarely considered. In this study, combinations of chemo- and photooxidation reactions were examined for their efficiencies in removing dyes from wastewater. In order to determine their effect on dye removal efficiency, interaction factors between the photo- and chemo-oxidation reactions were evaluated using both fractional- and full-factorial experiments. Combinations of treatment processes were further evaluated for optimal reagent dose using a grid design. Finally, a pseudo-first-order model was devised to describe the oxidative removal

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efficiencies over time, with the model parameters provided. Potential applications of the results of this study are also discussed in relation to the future treatment of wastewater containing multiple dyes.

2. Material and methods

Three distinctive dyes were tested in this study: Acid Red-114 (AR-114) and Reactive Black-5 (RB-5), which were purchased from Sigma-Aldrich, Inc., USA, and Disperse Black EX-SF (DB-EX-SF), which was purchased from T&T Industrial Corporation, Taiwan, as listed in Table 1. These three dyes are commonly used to dye wool, cotton, and polyester fibers, respectively, and differ considerably in their water solubilities and chemical structures. Five oxidizing agents were used: TiO_2 , hydrogen peroxide (H_2O_2), EDTA-Fe, ferrous oxalate (FA), and Fe^{2+} . EDTA and FeSO_4 (Mallinckrodt Baker, Inc., USA) were used to prepare the EDTA-Fe solution; H_2O_2 was obtained from Merck KGaA (USA); and P25 TiO_2 was purchased from Hombikat (USA).

Oxidation reactions were conducted in a 30 mL KG-33 borosilicate glass bottle with 90–92% light transmittance (Kimble & Chase, USA). In each experiment, a 30-mL sample solution was prepared with one dye, and one oxidant and/or one catalyst, as required, at given concentrations. The sample solution was adjusted to neutral pH and added to the bottle, which was then placed in a reactor with an ultraviolet (UV) light source (Gel Media System, Taiwan) and irradiated at 300 nm with intensity of 149.5 lux at the bottle surface. The treatment was performed for 2 h, after which the residual dye concentration was measured. Concentrations of AR-114 and RB-5 in the treated solutions were determined using a UV–vis spectrophotometer (UV-2900, Hitachi). The residual concentration of DB-EX-SF was determined by high-performance liquid chromatography (HPLC, LC-10AT vp, Shimadzu, USA) with a C_{18} column, with Disperse Red (T&T Industrial Co., Taiwan) as the internal standard for the HPLC analyses. The initial dye concentration was set to 100 mg/L. Dye-only samples were also prepared and tested

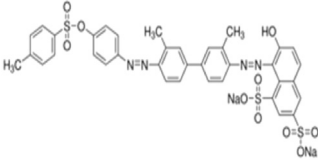
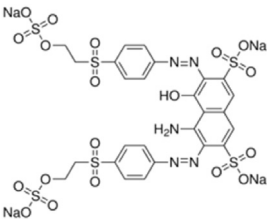
three times as controls for determining the dye removal efficiency of each oxidant.

The combined effects of the different oxidants on dye removal efficiency were evaluated using both fractional and full-factorial designs. For reactions with H_2O_2 , the high (+) concentration was set to 29.4 mM. For TiO_2 , the initial concentration was set to 1.67 g/L, and the initial concentration for all ferrous-based catalysts was set to 0.18 mM (see Table 2). Samples were prepared with oxidants at high (+) or low (–, not used) doses. A full-factorial experiment was performed to further assess the effects of different oxidant levels on dye removal efficiencies. The low (–) dose for each oxidant was set to zero (not used), and a new high (+) dose was set at half that used in the fractional-factorial experiment. The dye removal efficiency was evaluated as described earlier in this section.

Finally, combinations of oxidants for dye removal were optimized using a grid design with up to three factors. The dye removal efficiencies of each oxidant were assessed individually across a wide range of concentrations. Subsequently, the removal efficiencies were assessed with combinations of oxidants within the concentration ranges where higher efficiencies were achieved with an individual oxidant. In each grid run, the dominant oxidant was maintained at a fixed level, and the dye removal efficiency was assessed with different levels of the two other oxidants. After the most effective dose was determined, that oxidant combination was further assessed for dye removal efficiency over time. The observed outcomes were fitted to a pseudo-first-order model using the least-squares method.

For quality assurance, all measurements were repeated in triplicate. Percentage error (%error) was calculated by dividing the standard deviation by the mean of the measurement outcomes, and a 5% limit was adopted to ensure data consistency. Each of the experimental conditions was assessed at least twice, with some assessed in quadruplicate, and the results were summarized as average values.

Table 1
Properties of the dyes used in this study.

Target compound and its structure	Molecular weight (g)	Textiles dyed	Properties
Acid Red 114 (AR-114) 	830.81	Wool, Silk, Nylon, Polyamide fibers	<ul style="list-style-type: none"> • Water soluble (80 g/L) • Used in low pH • Easy to use • Forms an ionic bond
Reactive Black 5 (RB-5) 	991.82	Cotton and Blended fibers, Wool, Silk	<ul style="list-style-type: none"> • Water soluble (60 g/L) • Easy to use • Potential to be hydrolyzed • Forms a covalent bond
Disperse Black EX-SF (DB-EX-SF) Structure not available	Not available	Polyester fibers, Polyamide fibers, Cellulose acetate, Nylon, and Orlon	<ul style="list-style-type: none"> • Minimal water solubility • Added with a dispersive chemical

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