



Synergistic effects of combining ultrasound with the Fenton process in the degradation of Reactive Blue 19



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ABSTRACT

The decoloration of reactive dye C.I. Reactive Blue 19 (RB 19) using combined ultrasound with the Fenton process has been investigated. The effect of varying the concentrations of hydrogen peroxide and iron sulfate, initial pH, ultrasonic power, initial dye concentration and dissolved gas on the decoloration and degradation efficiencies was measured. Calibration of the ultrasound systems was performed using calorimetric measurements and oxidative species monitoring using the Fricke dosimeter and degradations were carried out with a 20 kHz probe type transducer at 2, 4, 6 and 8 W cm⁻² of acoustic intensity at 15, 25, 50 and 75 mg L⁻¹ initial dye concentrations. First order rate kinetics was observed. It was found that while the degradation rate due to ultrasound alone was slow, sonication significantly accelerated the Fenton reaction. While the results were similar to those reported for other dyes, the effects occurred at lower concentrations. The rate and extent of decoloration of RB 19 increased with rising hydrogen peroxide concentration, ultrasonic powers and iron sulfate concentration but decreased with increasing dye concentration. An optimum pH value of pH = 3.5 was found. The rate of decoloration was higher when dissolved oxygen was present as compared with nitrogen and argon confirming the solution phase mechanism of the degradation.

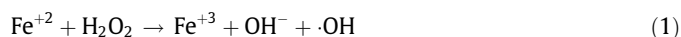
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1. Introduction

A variety of chemically different dyes are used for various industrial applications such as textile dyeing, paper printing, leather, shoe polish, plastics, food coloring *etc.* A significant amount of these dyes enter the environment as wastewater [1]. There are more than 100,000 types of dyes commercially available and over 7×10^5 tons of dyestuff are produced annually [2]. Reactive dyes are resistant to light, water and oxidizing agents and are therefore difficult to degrade once released into aquatic systems. The presence of very low concentrations of dyes in effluent can be highly visible and undesirable [3] on grounds. Their presence disturbs aquatic communities present in ecosystem by obstructing light penetration and oxygen transfer into water bodies [4]. Moreover, they can be toxic and carcinogenic [5–7].

A number of treatment techniques have been developed to remove dyes from the wastewaters. Among these, advanced oxidation processes (AOPs) are effective in degrading many reactive dyes. The Fenton process is a homogeneous advanced oxidation process using an acidic mixture of hydrogen peroxide and ferrous ions [8,9] to produce highly oxidative hydroxyl radicals which

react with dissolved species, removing color and lowering chemical oxygen demand. The ·OH radicals (Eq. (1)), attack the unsaturated dye molecule and the chromophore of the dye molecule is destroyed and decolorized [9].



The use of power ultrasound as an advanced oxidation process has been also employed in the degradation of textile dyes [8,10]. This is also generally based on the formation of short-lived radical species generated in violent cavitation bubble collapses. These radical species can diffuse out of the bubble into the bulk fluid medium where they are able to react with solute molecules. A steady-state concentration of reactive radical species in the liquid phase can be maintained by continuous irradiation with ultrasound. Volatile solutes may evaporate into the bubble and be degraded by the harsh conditions generated during cavitation. Non-volatile organic compounds present in the liquid phase can undergo degradation mainly by reaction with powerful oxidizing agents such as ·OH radicals produced [11]. The sonochemical enhancement of several AOPs has been reported. A number of studies have reported the use of ultrasound for the decoloration and degradation of textile dyes, but it has often been difficult to completely mineralize the dye stuff using ultrasound alone [12].

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The combination of ultrasound with other advanced oxidation process is a convenient approach in degrading reactive dyes. There are a number of reports on the combined use of ultrasound and Fenton process for the degradation of several textile dyes [8,9,13,14]. While Guimaraes *et al.* [15] showed that oxidation of Reactive Blue 19 can be promoted by use of Fenton reagent and accelerated photochemically, the sono-Fenton process has not previously been applied to the degradation of this dye. Given its widespread use, [15,16] the present study has focused on RB 19 dye to undergo treatment with combined Fenton and ultrasonic processes. The effect of the various experimental parameters including hydrogen peroxide and iron sulfate concentrations, pH, initial dye concentrations and dissolved gases on color removal were investigated.

2. Experimental procedure

2.1. Materials

Reactive Blue 19 (RB 19) was purchased from Sigma–Aldrich (UK) and was used as received; its structure is shown in Fig. 1. $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (analytical grade) was obtained from BDH Laboratory Supplies (England). Hydrogen peroxide (analytical grade) 35% w/v, H_2SO_4 and NaOH were obtained from Fisher Scientific (UK). All solutions were prepared with distilled water. Gases were obtained from BOC and were used without further purification.

2.2. Procedure

A stock solution of RB 19, prepared by dissolving 1 g of dye in 1 L of distilled water, was diluted to give initial concentrations for each experiment of 25 mg L^{-1} ($4 \times 10^{-5} \text{ mol L}^{-1}$). Sulphuric acid and sodium hydroxide were used to adjust the pH of the dye solution. 100 mL volumes from the stock solution were placed in the glass reactor, and the appropriate amount of hydrogen peroxide and iron sulfate added to the solution. The glass reactor was equipped with a water circulating jacket for maintaining reaction temperature to $\pm 1^\circ\text{C}$. Sonication was performed with a Sonic processor L500-20 ultrasonic generator (20 kHz, 200 W, Sonic Systems) equipped with titanium probe transducer (23820T). The tip of the horn was 1 cm in diameter and was placed 1.5 cm into the liquid layer (Fig. 2). At 5 min time intervals, samples were taken from the reactor and analyzed by UV/visible spectrophotometry (Agilent 8453) using detection wavelengths of 256 and 594 nm. Each experiment was performed in duplicate. Sonication was conducted in the presence of air with no added gas except when the effect of dissolved gas was being investigated. In these experiments, the initial solution was vigorously purged with Argon, O_2 or N_2 for 20 min prior to ultrasonic irradiation and a gas flow of approx. 1 mL min^{-1} was maintained throughout the experiment.

The percentage (%) decolorization was found from Eq. (2)

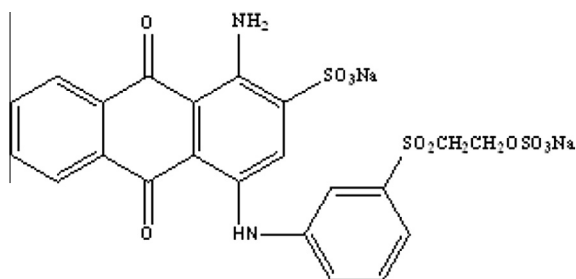


Fig. 1. Structure of RB 19 dye.

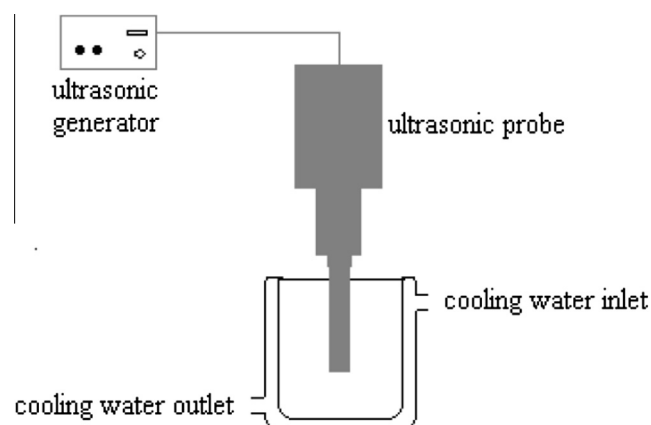


Fig. 2. Experimental setup.

$$\text{Dye \% decolorization} = (1 - C_t/C_0) \times 100 \quad (2)$$

where C_t and C_0 are the concentrations (mg L^{-1}) of dye at reaction time t and prior to sonication, respectively [12]. The change in concentration in the solution was calculated from the Beer–Lambert law [13]

$$A = l \varepsilon C \quad (3)$$

where A is the absorbance, l is the path length (cm), ε is the molar extinction coefficient ($\text{L mol}^{-1} \text{ cm}^{-1}$) and C is the dye concentration (mol L^{-1}).

2.3. Instrumentation

The samples were collected at different time intervals and the degradation products were identified using Liquid chromatography/mass spectrometry (LC/MS). The mass spectrometry was performed using a microTOF electrospray time of flight (ESI-TOF) mass spectrometer (Bruker Daltonik GmbH, Bremen, Germany) coupled to an Agilent 1200 LC system (Agilent Technologies, Waldbronn, Germany). The LC was equipped with a Phenomenex Luna $5\mu\text{C18}$ (2) column with stationary phase mesh size of 100 \AA and dimensions ($50 \times 2 \text{ mm}$), connected directly to the MS. The other experimental conditions were: nitrogen supplied at a pressure of 1 bar as a nebulising gas and also used as the drying gas, with the flow rate of 8 L min^{-1} at a temperature of 200°C , water/acetonitrile was employed as mobile phase with a flow rate of 0.4 mL min^{-1} , sample volume $1 \mu\text{L}$, injection temperature 25°C . For the detection of positive/negative ions a capillary voltage of $-4000 \text{ V}/+4000 \text{ V}$ was used.

2.4. Effects of acoustic intensity and sonolysis

The acoustic power dissipated by the horn into the solution in the reactor was measured using the usual calorimetric method [17] employing a digital thermometer that recorded the solution temperature every 5 min, over a 30 min interval. The overall power input was estimated according to Eq. (4)

$$\text{Energy input, } q = mc \, dT/dt \quad (4)$$

where m is the mass of water; c is the heat capacity of water and dT/dt is the temperature gradient over time. The acoustic intensity (W cm^{-2}) was determined by dividing the power input (q) by the horn surface area (A).

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