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Degradation of *Reactive Black* 5 by Fenton/UV-C and ferrioxalate/H_2O_2/solar light processes

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Abstract

The feasibility of employing different photoxidation systems, like Fenton/UV-C and ferrioxalate/H_2O_2/solar light in the decolorization and mineralization of an azo dye, has been investigated. Batch experiments were carried out to evaluate, on the first stage, the influence of different processes on *Reactive Black* 5 (RB5) decolorization. During the second stage were investigated the optimal operational conditions of Fenton/UV-C and ferrioxalate/H_2O_2/solar light processes, like pH, H_2O_2 dosage, iron dosage, RB5 concentration and source of light. The experiments indicate that RB5 can be effectively decolorized using Fenton/UV-C and ferrioxalate/H_2O_2/solar light processes with a small difference between the two processes, 98.1% and 93.2%, respectively, after 30 min. Although there is lesser difference in dye decolorization, significant increment in TOC removal was found with Fenton/UV-C process (46.4% TOC removal) relative to ferrioxalate/H_2O_2/solar light process (29.6% TOC removal). This fact reveals that UV-C low-pressure mercury lamp although with its small effect on dye decolorization is particularly important in dye mineralization, when compared to solar light. However, ferrioxalate/H_2O_2/solar light system shows large potential on photochemical treatment of textile wastewater with particular interest from the economical point of view.

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1. Introduction

Textile industry is one of the highest water consuming sectors, between 25 and 250 m^3 per ton of product depending on the processes – and the largest consumer of colorants for various dyeing, printing and finishing processes [1,2]. This water consumption, allied to high dosages of dyes, originates effluents that are extremely colorized. The release of these wastewaters to natural environments is described as very problematic to aquatic life [3] and mutagenic to human [4].

The conventional treatment techniques applied in textile wastewaters, such as chemical coagulation/flocculation, membrane separation (ultrafiltration, reverse osmosis) or elimination by activated carbon adsorption, are not only costly, but also result in phase transfer of pollutants. Biological treatment is not an efficient solution to these effluents due to the complex structure of some dyes that provokes resistances to biodegradation. Hence, the resource to Advanced Oxidation Processes (AOPs) could be a good alternative to treat and remove textile dyes from the wastewaters.

The azo dyes, characterized by an azo group (–N=N–), are the largest class of dyes used in textile industry for dyeing several natural and synthetic materials [1,5]. Between the several azo dyes (acid, reactive, disperse, vat, metal complex, mordant, direct, basic and sulphur) the most used are the 'reactive' type. These dyes are the most problematic pollutants of textile wastewaters. This fact occurs because, after the dyeing process, more than 15% of the textile dyes are lost in wastewater stream [6]. Therefore, in this study was selected the azo dye *Reactive Black* 5 (RB5) as a representative dye pollutant of textile wastewaters.

Advanced Oxidation Processes offer a highly reactive, non-specific oxidant, namely hydroxyl radicals ($HO^•$), capable of

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destroying a wide range of organic pollutants in water and wastewater [7,8]. Fenton's reagent oxidation is a homogeneous catalytic oxidation process using a mixture of hydrogen peroxide and ferrous ions. In an acid environment if hydrogen peroxide is added to an aqueous system containing an organic substrate and ferrous ions, a complex redox reaction will occur [9–11]. The overall reaction is

$$H_2O_2 + Fe^{2+} \rightarrow Fe^{3+} + HO^- + HO^\bullet \qquad (1)$$

The ferrous ion initiates and catalyses the decomposition of H_2O_2, resulting in the generation of hydroxyl radicals, $HO^\bullet$ [12,13]. Hydroxyl radicals are capable of rapidly attacking organic substrates and causing chemical decomposition of these compounds by H-abstraction and addition to C═C unsatured bonds [14].

The addition of UV radiation to Fenton's reagent process could be an interesting allied in dye decolorization due to its capacity in influencing direct formation of $HO^\bullet$ radicals [15,16]. This AOP combination has gained progressive attention in recent years due to its higher efficiency when compared to the dark process. In Fenton/UV-C process, in addition to Eq. (1), the formation of hydroxyl radical also occurs by the following reactions (Eqs. (2) and (3)):

$$H_2O_2 + UV \rightarrow HO^\bullet + HO^\bullet \qquad (2)$$

$$Fe^{3+} + H_2O + UV \rightarrow HO^\bullet + Fe^{2+} + H^+ \qquad (3)$$

Recently, other processes have been studied trying to use solar light instead of artificial UV radiation. Ferrioxalate, used for decades as a chemical actinometer, has been applied in the Fenton's reagent process as iron source thus allowing further benefit from solar radiation [17]. The use of ferrioxalate in the degradation of organic pollutants was reported to be very effective, and using solar light was found to be an economic alternative when compared to artificial UV radiation [18–20]. The photolysis of ferrioxalate generates Fe(II) in acid solutions reported as follows [18,21]:

$$[Fe(C_2O_4)_3]^{3-} + h\nu \rightarrow Fe^{2+} + 2C_2O_4^{2-} + C_2O_4^{-\bullet} \qquad (4)$$

$$C_2O_4^{-\bullet} + [Fe(C_2O_4)_3]^{3-} \rightarrow Fe^{2+} + 3C_2O_4^{2-} + 2CO_2 \qquad (5)$$

$$C_2O_4^{-\bullet} + O_2 \rightarrow O_2^{-\bullet} + 2CO_2 \qquad (6)$$

The main objective of this study is to analyse the feasibility of decolorization and mineralization of RB5 by Fenton/UV-C and ferrioxalate/H_2O_2/solar light processes. The influence of different operational parameters (source and intensity of light, pH and H_2O_2, iron and RB5 concentration) that affect the efficiency of Fenton/UV-C and ferrioxalate/H_2O_2/solar light processes during the oxidation of *Reactive Black* 5 are studied.

2. Experimental

2.1. Material

The azo dye, *Reactive Black* 5 (Color Index 20505), was kindly provided by *DyStar Anilinas Têxteis Lda.* (Portugal) and used as received without further purification. UV–vis absorption spectra and molecular structure of RB5 in non-hydrolyzed form are illustrated in Fig. 1. The chemicals used in the experiments, $FeCl_3$ (*Riedel-de-Haën*), $C_2H_2O_4$ (*Fluka*), $FeSO_4 \cdot 7H_2O$ (*Panreac*), H_2O_2 (*Merck*, Perhydrol, 30% w/w) and Na_2SO_3 (*M&B*) were of reagent grade. All the solutions were prepared by dissolving requisite quantity of dye in deionised water from a *Millipore* purification system. When appropriate, the pH of the solution was adjusted using H_2SO_4 and NaOH solutions. Initial pH of solution was monitored using a Basic pH Meter from *Denver Instrument Company*.

2.2. Photoreactor

Batch experiments for Fenton/UV-C oxidation, were performed in a *Heraeus* photoreactor. The cylindrical reactor of 800 mL capacity was made of borosilicate glass with ports, at the top, for sampling. For Fenton/UV-C oxidation was used a low-pressure mercury vapor lamp, *Heraeus TNN 15/32*, placed in an axial position inside the reactor. The reaction temperature was kept at the desired value within ±0.5 °C by using a thermostatically controlled outer water jacket. For every experiment performed, the reactor was initially loaded with 500 mL of RB5 aqueous solution and continuous mixing was maintained by means of a magnetic stirrer. Ferrioxalate/H_2O_2/solar light experiments were carried out, in open air, at the University of Trás-os-Montes and Alto Douro campus, Portugal (41°18′N; 7°45′W) under clear sky conditions. All photocatalytic experiments were carried out under similar conditions on sunny days of May–August, between 11 AM and 3 PM. Solar reactions were conducted in a glass reactor wrapped externally with aluminium foil to avoid lateral penetration of irradiation through the sidewalls. During the experiments solar light intensity was measured at 5-min intervals using a *Macam Q102 PAR* Radiometer (400–1000 nm). The measurements were standardized in the way such that the sensor was always set in the horizontal position. The average light intensity over the duration of each experiment was calculated.

2.3. Analysis

For Fenton/UV-C process a desired quantity of dye/Fe^{2+}/H_2O_2 solution was freshly prepared from $FeSO_4 \cdot 7H_2O$, H_2O_2 and the dye stock solution. Most of the experiments were carried out at pH = 5 of the solution. The required amount of Fe^{2+} was added into the dye solution. In ferrioxalate/H_2O_2/solar light process the required amounts of Fe^{3+} and oxalic acid were added simultaneously into the dye solution and mixed by means of a magnetic stirrer. Finally, in both processes, the desired volume of H_2O_2 was injected into the

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