



# Using central composite experimental design to optimize the degradation of real dye wastewater by Fenton and photo-Fenton reactions



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## ABSTRACT

This work focuses on the use of Fenton reagent and UV-irradiation, in a lab-scale experiment, for the treatment of real dye wastewater coming from a Spanish textile manufacturer.

Response surface methodology and a 2<sup>3</sup> factorial design were used to evaluate the effects of the three independent variables considered for the optimization of the oxidative process: temperature, Fe (II) and H<sub>2</sub>O<sub>2</sub> concentrations, for a textile wastewater generated during a dyeing process with chemical oxygen demand of 1705 mg L<sup>-1</sup> O<sub>2</sub> at pH = 3. Wastewater degradation was followed in terms of chemical oxygen demand reduction.

In the optimization, the correlation coefficients for the model ( $R^2$ ) were 0.985 and 0.990 for Fenton and photo-Fenton treatments respectively. Optimum reaction conditions at pH = 3 and temperature = 298 K were [H<sub>2</sub>O<sub>2</sub>] = 73.5 mM and [Fe(II)] = 1.79 mM.

The combination of Fenton, Fenton-like and photon-Fenton reactions has been proved to be highly effective for the treatment of such a type of wastewaters, and several advantages for this technique arise from the study. Under optimum conditions, 120 min of treatment resulted in a 62.9% and 76.3% reduction in chemical oxygen demand after Fenton and photo-Fenton treatments respectively.

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## 1. Introduction

Textile mills are major consumers of water with high average water consumption [1] and consequently one of largest groups of industries causing intense water pollution. Generated wastewaters collect different effluents from different manufacturing unitary operations: from raw material preparation processes (i.e., desizing, scouring and bleaching), as well as from dyeing, soaping and softening. These complex operations, subjected to frequent changes as a result of shifting consumers preferences, are the cause of the variable volume and the wide diversity of chemical products found in these wastewaters. Though their characteristics depend on the specific operations performed, they commonly present suspended solids, high temperature, unstable pH, high chemical oxygen demand (COD), low biological demand (BOD) and high colourization.

These effluents, produced in great quantities, contain appreciable levels of organic compounds which are not easily amenable to chemical or biological treatment [2,3] and can be very dangerous

to environmental life [4]. Moreover, adsorbable organic halogens (AOX) are formed as a result of the use of bleaching chemicals [5].

Such wastewater composition would be highly deleterious to natural areas. High organic matter content results in depletion of dissolved oxygen, which has an adverse effect on the marine ecological system and the whole ecosystem [6].

As said before, a particularly common aspect of textile effluents is the high COD. In order to overcome this problem, a typical practice in the textile and paper industries is the previous use of hydrolysis in basic media, in order to decrease initial COD levels [7,8].

In this frame, textile industry is confronted with the challenge of effective wastewater remediation.

A varied range of methods have been developed for textile wastewater treatment at laboratory, pilot or full scale. The most widely used are coagulation–floculation, foam flotation, membrane filtration, biological treatment and chemical processes [9–11]. However, most of these methods are quite inefficient or are not suitable when working with toxic and/or non biodegradable textile wastewaters [12]. Therefore, destructive treatment methods for the remediation of recalcitrant or hazardous pollutants are currently under investigation. In this direction and taking

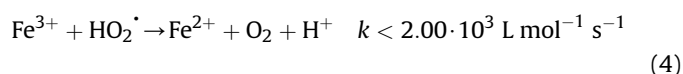
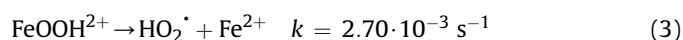
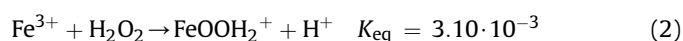
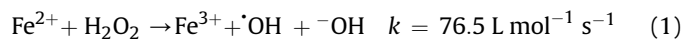
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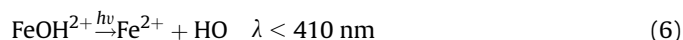
advantage of the high oxidative power of the  $\cdot\text{OH}$  radical (2.8 V vs. NHE), several advanced oxidation processes (AOPs) such photocatalysis, Fenton and photo-Fenton has been used in the remediation of textile wastewaters [13–15].

The effectiveness of these processes has a special interest and constant development [16]. Moreover, it makes possible the achievement of high reactions yields with a low cost treatment.

In the Fenton's processes, the reactions related to generation of hydroxyl radicals and  $\text{Fe}^{2+}$  ion regeneration, are the following:



The rate of contaminants degradation can be considerably increased when ultraviolet light is simultaneously irradiated in the photo-Fenton's process [11]. Under irradiation, ferric ion complexes produce extra  $\text{HO}\cdot$  radicals and the regeneration of  $\text{Fe}(\text{II})$  which will further react with more  $\text{H}_2\text{O}_2$  molecules in Fenton reaction (equation (1)):



The Fenton processes depend on various variables, such as pH, temperature, source of light and the  $\text{H}_2\text{O}_2$  and  $\text{Fe}^{2+}$  concentrations. The level of these variables is the key in the COD decrease and elimination of different organic compounds.

However, most of the literature studies on Fenton reagent treatment of textile wastewaters report decolourization efficiencies, but not COD removal or mineralization. Moreover, in these studies each variable was treated individually, while the other variables remain constant.

This high number of influential variables makes the use of suitable experimental design attractive. These techniques provide a systematic way of working that allows conclusions to be drawn about the variables or its combination, that are most influential in the response factors while carrying out the minimum possible number of experiments. Among these experimental designs, we can find the central composite design (CCD), Doehlert matrices, Box-Behnken designs and three-level full-factorial designs [17].

We can find in the literature some papers that use statistical design of experiments to develop optimal AOPs for wastewaters treatment. Several authors have studied the degradation of pulp and paper wastewaters [18,19] and textile wastewaters [20–22]. However, most of these papers are related to synthetic effluents. In consequence, the goal of the present work has been to identify, by using response surface methodology (RSM), optimum reaction conditions to degrade a real dye wastewater. As could be seen from obtained results, this wastewater could be successfully treated using Fenton and photo-Fenton reactions.

## 2. Experimental

### 2.1. Textile wastewater

The textile wastewater used in this study was collected from a Spanish textile manufacturer, located in Terrassa (Catalonia, North

East of Spain). It was a mixed effluent from two different dyeing processes with polyamide (PA) and polyester (PES) fibres according to the companies specifications by using different LANASET acid dyes and DIANIX disperse dyes for PA and PES dyeing respectively. The liquor ratio used was 1/10 (1 g of fibre for 10 mL water).

This textile wastewater had a COD = 1705 mg L<sup>-1</sup> O<sub>2</sub>, Total organic carbon (TOC) = 621 mg L<sup>-1</sup> C, suspended solids (SS) = 33.18 mg L<sup>-1</sup>, pH = 7.4 and colour index = 1.41 cm<sup>-1</sup>. Colour index relies on the measurement of the spectral absorption coefficient in the visible range at wavelengths of 436, 525 and 620 nm.

The effluent had no excessive colouration because of the high exhaustion degree of dyes in the considered textile process (residual water contained less than 5% of the concentration of dyes).

It was stored at 4 °C before use.

### 2.2. Chemicals

Iron sulphate ( $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ , Merck 99,5%) and hydrogen peroxide ( $\text{H}_2\text{O}_2$  Panreac 33% (w/v)) were used to obtain hydroxyl radical,  $\text{HO}\cdot$ .

Concentrated sulphuric acid and sodium hydroxide solutions were used to achieve desired pH values in working solutions.

Deionized water, from a Millipore Milli-Q system, was used to prepare all solutions.

### 2.3. Reactors and light sources

All Fenton and photo-Fenton experiments were carried out using a cylindrical Pyrex thermostatic cell of 150 mL of capacity. The reaction mixture consist in 100 mL of textile wastewater and necessary amount of  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  and  $\text{H}_2\text{O}_2$ . During all the treatment, solutions were stirred and temperature maintained at the required level [23].

As artificial source of light was used a 6 W Philips black light fluorescent lamp, which basically emits at 350–400 nm. The intensity of the incident UVA light, measured with a uranyl actinometer, was  $1.38 \times 10^{-9}$  Einstein s<sup>-1</sup>.

### 2.4. Analytical methods

Chemical Oxygen demand (COD, (mg L<sup>-1</sup> O<sub>2</sub>)) was measured using the closed-reflux colorimetric method [24] with a HACH DR/2000 spectrophotometer.

Total Organic carbon (TOC, (mg L<sup>-1</sup> C)) was determined with a Shimadzu TOC-VCSH analyser with a solution of potassium phthalate as standard of calibration.

$\text{H}_2\text{O}_2$  consumption was measured using the KI titration method [25]. Remaining  $\text{H}_2\text{O}_2$  was removed with sulphite [26]. Any remaining sulphite was removed by bubbling O<sub>2</sub>.

## 3. Results and discussion

### 3.1. Operational conditions

The performance of the Fenton system depends of different variables like pH [11], initial  $\text{Fe}(\text{II})$  and  $\text{H}_2\text{O}_2$  dosage [27] and temperature [28]. Obviously a great number of experiments would be needed if all these variables were considered in the experimental design. If the role of these variables is previously known it will be possible to simplify the experimental analysis.

In this direction, it is known that the performance of such a complex reactive system is clearly pH dependent, particularly in Fenton-like and photo-Fenton reactions, with the maximum catalytic activity around pH = 2.8 [29]. For higher pH values, low activity is detected because of decrease of free iron species due to

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