

Optimization of solar-driven photo-electro-Fenton process for the treatment of textile industrial wastewater



Edison GilPavas^{a,*}, Izabela Dobrosz-Gómez^b, Miguel Ángel Gómez-García^c

^a GIPAB: Grupo de Investigación en Procesos Ambientales, Departamento de Ingeniería de Procesos, Universidad EAFIT, Cr 49 # 7 Sur 50, Medellín, Colombia

^b Grupo de Investigación en Procesos Reactivos Intensificados con Separación y Materiales Avanzados – PRISMA, Departamento de Física y Química, Facultad de Ciencias Exactas y Naturales, Universidad Nacional de Colombia, Sede Manizales, Campus La Nubia, km 9 vía Aeropuerto la Nubia, Manizales, Colombia

^c Grupo de Investigación en Procesos Reactivos Intensificados con Separación y Materiales Avanzados – PRISMA, Departamento de Ingeniería Química, Facultad de Ingeniería y Arquitectura, Universidad Nacional de Colombia, Sede Manizales, Campus La Nubia, km 9 vía Aeropuerto la Nubia, Manizales, Colombia

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ABSTRACT

This work deals with the evaluation of a solar-driven Photo Electro-Fenton (SPEF) process as an alternative for the effective degradation of an industrial textile wastewater sample. Experiments were carried out in a laboratory scale batch cell reactor, using boron-doped diamond (anode) and titanium (cathode) electrodes in monopolar configuration. The effect of the main operational parameters (pH, current density (j), conductivity (σ), Fe^{2+} concentration and anode area to effluent volume (A/V) ratio) on the COD removal and energy consumption were studied using a Box-Behnken experimental design. The SPEF process was optimized using the Response Surface Methodology. At optimum operational conditions (pH = 4, $j = 40 \text{ mA/cm}^2$, $\sigma = 5768 \mu\text{S/cm}$ and $\text{Fe}^{2+} = 0.3 \text{ mM}$), the solar-driven process achieved total discoloration, COD reduction of 83% and TOC mineralization of 70%, after 15 min of electrolysis. The process yielded a highly oxidized (AOS = 2.24) and biocompatible ($\text{BOD}_5/\text{COD} > 0.4$) effluent. Additionally, the most suitable effective surface area of the electrodes (A/V ratio) was determined (3.75 m^{-1}). The analysis of operational costs was also performed. The SPEF process demonstrated to be an efficient alternative for the treatment of industrial wastewater effluent, allowing to achieve Colombian permissible discharge limits.

1. Introduction

The textile industry uses a great amount of chemical compounds (v.g., detergents, waxes, dyes, surfactants, solvents, salts, etc...) and water during its different processes. Consequently, textile wastewater (TWW) is a complex mixture of chemicals characterized with high values of Chemical Oxygen Demand (COD), Total Organic Carbon (TOC) as well as low biodegradability. Indeed, most of TWW is considered as toxic one [1]. In general, TWW is quite difficult to degradate using conventional and/or biological wastewater treatments [2–4]. Thus, the development of new, environmentally friendly technologies, able to reach complete mineralization of pollutants, become an urgent challenge.

Electrochemical oxidation or electro-oxidation (EO) has been recognized as one of the most promising electrochemical procedures for wastewater treatment [5]. The use of simple equipment, easy operation, versatility and environmental compatibility (it does not require the addition of chemicals) are considered as the most important advantages of this technology [6]. In this process, organic matter can be oxidized in

two ways: (i) by direct oxidation (anodic oxidation), proceeding on anode surface; and/or (ii) by indirect oxidization (v.g., Electro-Fenton, EF), provoked by electrochemically generated strong oxidants (v.g., hydroxyl radicals: $\cdot\text{OH}$ via Fenton's reagent).

In anodic oxidation, $\cdot\text{OH}$ radicals are formed due to water oxidation on a high O_2 -overtaged anode (reaction (1)).



Among the wide variety of available electrodes (anodes) that can be applied in anodic oxidation, the boron-doped diamond (BDD) one stands out due to its following properties: inert surface with low adsorption properties, high resistance to deactivation and to corrosion, high thermal stability, high hardness, good electrical conductivity and an extremely wide potential in aqueous and non-aqueous electrolytes [7].

On the other hand, the EF consists of adding Fenton's reagent to the EO [8]. Thus, the electrogenerated H_2O_2 reacts with Fe^{2+} , present in the medium, leading to the formation of $\cdot\text{OH}$ radicals, as presented in reaction (2).

* Corresponding author.

E-mail address: egil@eafit.edu.co (E. GilPavas).

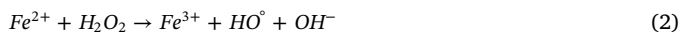
Table 1

Characterization of the industrial TWW together with permissible discharge limits, defined by Colombian legislation, vs. characteristics of the obtained effluent after SPEF process.

Parameter	pH	Absorbance (660 nm)	Color (mg Pt-Co/L)	Conductivity ($\mu\text{S}/\text{cm}$)	Turbidity (NTU)	COD (mg O ₂ /L)	TOC (mg C/L)	BOD ₅ (mg O ₂ /L)	BOD ₅ /COD ratio	Operating costs, (USD/m ³)
Industrial TWW sample	9.96	1.626	1248	4560	184	545	164	118	0.2165	
Permissible limit ^a	6–9	–	–	–	–	400	–	200	> 0.4	
SPEF ^b (global efficiency,%)	3.9	0 (100)	51.7 (96)	6280	0 (100)	59.9 (90)	45.9 (79)	35.0 (70)	0.58	1.56

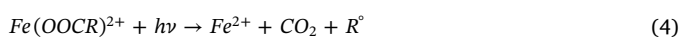
^a Emission limit values for industrial wastewater discharges into the municipal sewer system, according to Res 0631, 17/03/2015, issued by the Ministry of Environment and Sustainable Development, Colombia.

^b Optimal operating conditions: pH = 4, $j = 40 \text{ mA}/\text{cm}^2$, $\sigma = 5768 \text{ }\mu\text{S}/\text{cm}$, A/V ratio = 7.5 m^{-1} , $\text{Fe}^{2+} = 0.3 \text{ mM}$.



The preferred anode for EF is also the BDD one. It presents a remarkable capacity of $\cdot\text{OH}$ radicals production comparing with the other anode materials (v.g., Pt, IrO₂ and PbO₂), allowing the mineralization of organic contaminants in much larger extent [9].

Some drawbacks have to be faced when EO processes are used. Specifically, in the case of EF, its main limitation from the operational point of view, can be the formation of Fe(III)-carboxylate complexes, difficult to destroy by $\cdot\text{OH}$ radicals. It can be solved by subjecting the solution to UV radiation (such process is known as Photo-Electro-Fenton (PEF)). It favors: (i) the enhancement of Fe²⁺ regeneration and $\cdot\text{OH}$ production by photoreduction of Fe(OH)²⁺, Eq. (3); and (ii) the photodecarboxylation of Fe(III)-carboxylate intermediates, Eq. (4) [10–12].



Moreover, from an economical point of view, an important constraint against the application of EO processes is their high-energy costs associated with the use of artificial light/UV irradiating source. A possible solution for this restriction is the application of direct solar UV. This process is known as Solar Photo-Electro-Fenton (SPEF). Solar-based systems have become an alternative to reduce fossil consumption and greenhouse gas emissions [13,14]. Certainly, solar energy is the most abundant, inexhaustible and clean of all existing energy resources [15,16]. One of the most widespread and studied applications of solar energy is for photovoltaic (PV) power generation [17,18]. It can provide energetic autonomy for solar-driven EO processes since it does not require any connection to the grid.

As far as we know, only few papers presented the applicability of EF and SPEF processes for dyes removal from industrial wastewater [6,9–12]. Among them, it has been reported: (i) direct electrolysis using active electrodes (v.g., TiO₂/RuO₂, reaching 80% of COD and 95% of color removals, after 6 h of treatment) or non-active electrodes (v.g., Nb/BDD anode: 99% of COD removal; SnO₂ electrode: 70% of COD removal after 2 h; PbO₂ electrode: 65% of COD removal) and (ii) heterogeneous photo-electrocatalysis technologies (v.g., TiO₂ electrode, 1 mA cm^{-2} , irradiation time of 1 h with a UV lamp of 21 W cm^{-2} ; letting 25% of TOC removal). In these works, synergistic effects due to the combination of the photo irradiation and the electrochemical processes yielded higher removal percentages than these expected basing on the separate contribution of each of oxidation technologies.

This work deals with the evaluation of the SPEF process and its optimization as an alternative for the effective degradation of an industrial TWW sample, not amenable to biodegradation. Experiments were carried out in a laboratory scale batch cell reactor, using BDD (anode) and titanium (cathode) electrodes in monopolar configuration. The effect of the main operational parameters (current density, pH, Fe²⁺ concentration, conductivity, and anode area to effluent volume ratio) on COD removal, TOC mineralization and energy consumption

were studied. The solar treatment systems were optimized using the Response Surface Methodology (RSM) [19–23]. A kinetic analysis allowed determination of the time required to meet Colombian permissible discharge limits. The analysis of operational costs was also performed.

2. Materials and methods

2.1. Industrial wastewater samples

TWW samples were collected directly from an equalization tank of an industrial textile plant located in Medellín (Colombia). Before analysis and treatment in the laboratory, samples were kept refrigerated to avoid compounds degradation during storage and transportation, following standard procedures [24]. Table 1 presents their main characteristics. Notice that the wastewater's COD value was higher than the permissible discharge limits in Colombia, implying the presence of large amount of organic matter. In addition, it was characterized colored effluent with high conductivity value due to high salts concentration. Finally, its initial BOD₅/COD ratio suggested that the analyzed effluent was not amenable to biodegradation [25,26].

2.2. Reactants & analytical methods

All reagents were obtained from Merck and used as received without any further purification: ferrous sulfate hepta-hydrate (FeSO₄·7H₂O, 99.98%, used as SPEF reagent), H₂SO₄ (99.1%, to adjust samples pH), MnO₂ (reagent grade, $\geq 90\%$). All solutions were prepared using ultra-pure water (Milli-Q system; conductivity $< 1 \text{ }\mu\text{S cm}^{-1}$).

Samples, industrial and these resulting from laboratory tests, were analyzed by triplicate using a UV–VIS double-beam spectrophotometer (Spectronic Genesys 2PC), in the range of 200–700 nm, with a 1 cm path length quartz cell. Standard methods (SM) were used for the quantitative analysis of: COD (the closed reflux method with colorimetric determination, SM-5220D), Total Organic Carbon (TOC, SM-5310D), BOD₅ (the respirometric method, SM-5210B) and turbidity (SM-2130B) [24]. H₂O₂ concentration was measured by iodometric titration with KI and Na₂S₂O₃. To avoid its interference during COD measurements, the residual H₂O₂ was quenched using MnO₂. In all cases, the average values of the measurements are reported.

2.3. Experimental set-up

Electrochemical experiments were performed using the energy supplied by a solar panel (SOLAREX, ATERSA Inc, Spain). Table 2 shows its main characteristics. The applied potential difference (ΔE_{Cell}) between the cathode and the anode was adjusted using a voltage regulator. The electrical charge was integrated from tabular data (current vs. time), recorded manually from a voltage regulator display.

The SPEF experiments were conducted in a cylindrical quartz cell with 100-mL working volume. It contained two vertical electrodes,

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