

Contents lists available at [ScienceDirect](http://www.sciencedirect.com)

Process Safety and Environmental Protection

journal homepage: www.elsevier.com/locate/psep

Nickel foam a suitable alternative to increase the generation of Fenton's reagents

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ARTICLE INFO

Article history:

Received 1 March 2015

Received in revised form 17 April 2015

Accepted 29 April 2015

Available online 14 May 2015

Keywords:

Heterogeneous electro-Fenton

Nickel foam

Iron loaded polyacrylamide

hydrogels

Poly R-478

Boron Doped Diamond (BDD)

Decolorization

ABSTRACT

One of the bottlenecks often faced in environmental processes is focused on the design of efficient technologies. Thus, the hunt of suitable alternatives is a must to be tackled in order to improve the conventional methods. In this study the applicability of nickel foam as cathode in electro-Fenton's treatment of Poly R-478, a recalcitrant dye, was demonstrated. Linear sweep voltammetry and initial studies show the ability of nickel foam to increase the H_2O_2 generation during the electro-Fenton treatment, enhancing the removal yields achieved in comparison with other carbonaceous materials. Furthermore, complete mineralization of the pollutant was reached when using Boron Doped Diamond (BDD) as anode. A heterogeneous electro-Fenton process using iron loaded polyacrylamide hydrogels enhance the efficiency of the process with respect to homogeneous electro-Fenton and anodic oxidation technologies. Furthermore, the evaluation of the generated hydroxyl radicals confirms the obtained results. Finally, the reusability of the designed process was evaluated by decolorization of Poly R-478 in successive batches. SEM study of the nickel foam along the successive experiments confirms that no structure changes were detected.

Summing up, nickel foam seems to be a suitable cathode material for the electro-Fenton treatment of recalcitrant compounds in continuous mode.

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1. Introduction

Several physical, biological, and chemical processes have been used in the treatment of industrial effluents (Alvares et al., 2001; Chidambara Raj et al., 1997; Vaez et al., 2012). However, some contaminants found in wastewater could be recalcitrant to commonly applied treatments and for this reason it is necessary to develop processes that can operate in presence of any kind of pollutants (Oturán and Aaron, 2014).

Textile, dyestuffs and tannery industries are a clear example. They produce a substantial quantity of effluents containing different kinds of dyes used on their industrial activities that cause important environmental problems if discharged without a proper treatment (Sadik and Shama, 2002). Effluents from the textile industry are extremely variable in

composition (Correia et al., 1994), which underlines the need for largely unspecific processes for their treatment. Therefore, novel strategies for technological developments are necessary to reach the requirements established on the legislation (Ribeiro et al., 2015; Rivera et al., 2011).

Over the past years, different technologies such as advanced oxidation processes (AOPs) have demonstrated their great effectiveness on the decontamination of wastewater, offering an interesting and fast alternative treatment for these kind of effluents (Isarain-Chávez et al., 2014; Sirés et al., 2014; Stapleton et al., 2006). AOPs are chemical oxidation processes based on the in situ generation of hydroxyl radicals, which are very reactive and short-lived oxidants able to destroy target pollutants up to their mineralization without the generation of harmful byproducts in short treatment times

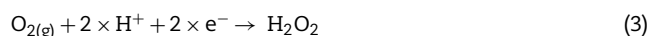
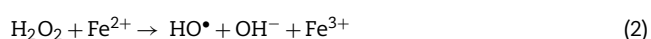
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<http://dx.doi.org/10.1016/j.psep.2015.04.011>

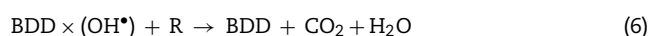
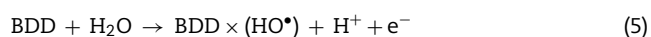
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(Eq. (1)) (Garcia-Segura and Brillas, 2011; Hammami et al., 2008). Among the most promising AOPs, Fenton's reaction with H_2O_2 and transition metals, especially the ferrous ion as shows Eq. (2), results particularly attractive and thus, it has been already largely investigated (Bautista et al., 2008; Mantzavinos, 2003; Rodriguez et al., 2011; Vicente et al., 2012). However, this technology presents several disadvantages such as the low stability of H_2O_2 , the rapid consumption of ferrous ions and the generation of sludge-containing iron at the end of the process that makes it expensive, requiring large amounts of chemicals and manpower. In order to overcome its limitations, numerous modifications of this process have been evolved during the last decades. A clear example are the electrochemical advanced oxidation processes (EAOP) and among them, the so-called electro-Fenton process (Nidheesh and Gandhimathi, 2012; Plakas et al., 2013; Rosales et al., 2012b), who was the first EAOP proposed and that is actually one of the most employed and developed AOP. This treatment has several improvements such as continuous electrogeneration of H_2O_2 as shows Eq. (3), short reaction time and Fe^{2+} regeneration by direct reduction of Fe^{3+} on the cathode as can be seen on Eq. (4). To increase the efficiency of this technology, several key variables, including the electrode material, initial pH and Fenton's reagents dosage have been studied (Almeida et al., 2012; Brillas et al., 2007; Murati et al., 2012; Oturan et al., 2012; Rosales et al., 2009, 2012a). In the electro-Fenton process, the electrode characteristics are one of the main affecting factors. Recently, different materials have been evaluated as anode such as RuO_2 and its mixture with IrO_2 or titanium net coated with $\text{RuO}_2/\text{IrO}_2$ (Chang et al., 2004; Anotai et al., 2010), obtaining in all cases a high efficiency on the electro-Fenton process degrading contaminants of emerging concern at trace levels.



The increase in the electrode surface area is also important in the production of Fenton's reagent (Lei et al., 2010; Wang et al., 2008). The generation of hydroxyl radicals can be incremented by increasing the conductivity and specific surface of the cathode, which finally causes the improvement in the removal of pollutants (Rosales et al., 2012a).

So far, numerous studies have already pointed out the efficiency of BDD anodes compared with others, such as PbO_2 or graphite (Manisankar et al., 2004; Oturan and Aaron, 2014; Panizza and Cerisola, 2004), on the mineralization of different compounds, showing much great performance than other materials for a large variety of organic and persistent pollutants (Panizza and Cerisola, 2009). Eqs. (5) and (6) describe the reactions occurred at the anode surface:



where BDD, anode material; $\text{BDD}(\bullet\text{OH})$, heterogeneous $\bullet\text{OH}$ radicals adsorbed on the anode material and R, organic matter.

Different carbonaceous materials, such as graphite, activated carbon fiber, three-dimensional electrodes are the most often used cathodes in electro-Fenton processes (Brillas et al., 2009; Lei et al., 2010; Nidheesh and Gandhimathi, 2014; Oturan and Aaron, 2014; Sirés and Brillas, 2012; Wang et al., 2010). Since these porous electrodes exhibit high mechanical and chemical stability usually researchers omit the performance of new cathodes, who may improve enormously the process.

On this way, Méndez-Martínez et al. (2012), evaluated the electrochemical reduction and oxidation of the azo dye Reactive Black 5 using nickel-polyvinylchloride as cathode material. Additionally, other authors (Liu et al., 2012) designed a three dimensional electro-Fenton system using nickel foam as particle electrodes, demonstrating its high efficiency on the degradation of a xantene dye such as Rhodamine B. These authors demonstrated the generation of superoxide radicals when aerating directly the nickel foam particles present at the bulk.

Therefore, the main objectives of the present study were to determine (a) the enhancement on the decolorization of a polymeric dye in an electro-Fenton treatment using nickel foam as cathode compared with other materials: carbon fibre and graphite sheet; (b) the mineralization of the studied pollutant comparing the effectiveness of two anodes (graphite sheet and BDD) and (c) the evaluation of hydroxyl radicals production from the selected electrodes.

2. Materials and methods

2.1. Dyes solutions

Poly R-478 provided by Sigma-Aldrich (Barcelona, Spain) was selected as model dye to evaluate the efficiency of the different treatments studied on this work. Solutions were prepared in distilled water and used without further purification. Dye characteristics and concentration used are described in Table 1.

2.2. H_2O_2 generation

Linear sweeping voltammetry (LSV) was performed to study the cathode materials in order to identify the most suitable electrode for the generation of H_2O_2 . The performance was carried out using an Autolab PGSTAT 320 N workstation at a scan rate of 50 mV/s in a three electrode system formed by graphite sheet, used as counter electrode, calomel electrode as reference and the different studied materials as working electrodes. All the assays were performed at room temperature. Experiments were carried out using a 0.25 L cylindrical glass reactor with a working volume of 0.15 L prepared in distilled water with 0.01 M of Na_2SO_4 and pH = 2.

After the selection of the most suitable material for H_2O_2 generation, its production was quantified. Experiments were carried out using a 0.25 L cylindrical glass reactor with a working volume of 0.15 L. Deionized water was mixed with Na_2SO_4 (0.01M), purchased from Fluka Analytical and the solution pH was adjusted to 2 (Bocos et al., 2014; Fernández de Dios et al., 2014; Iglesias et al., 2014) by adding H_2SO_4 . Besides, the pH was measured at the end of the experiments in order to corroborate that it kept constant along the treatment. A 1.6 mm thick nickel foam sheet with 95% of porosity and 95% of purity (Goodfellow Cambridge Ltd, United Kingdom) was immersed in the solution (14 cm × 7 cm). Thus, the sheet was

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