



Removal of phenolic compounds from oil refinery wastewater by electrocoagulation and Fenton/photo-Fenton processes



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ABSTRACT

A coupling between electrocoagulation (EC) and the Fenton and photo-Fenton processes was used to treat oil refining industry wastewaters that contained phenolic compounds. The effect of several experimental parameters of the EC treatment, such as current density (j), treatment time (t), and orientation and configuration of the electrodes was studied. In addition, treatment time and concentration of Fe^{2+} and H_2O_2 in the Fenton and photo-Fenton processes were varied to detect possible changes on the coupling capabilities. The EC treatment removed 51% of total phenols content and 42% of total organic carbon (TOC) content by applying 40 mA cm^{-2} during 20 min with electrodes horizontally positioned. The Fenton process application to the previously electrocoagulated water provided total phenols removal beyond the detection limit (0.01 mg L^{-1}) and 57.6% of TOC removal by using a 1:66 $\text{Fe}^{2+}/\text{H}_2\text{O}_2$ molar ratio. The application of the photo-Fenton process to the electrocoagulated water provided a TOC removal of 88%; the remaining TOC is associated to the presence of biodegradable short chain carboxylic acids.

1. Introduction

The release of large quantities of polluted industrial wastewaters to the environment creates the need for developing and implementing suitable treatment technologies, capable of removing hazardous contaminants present in many of these industrial streams [1].

In the petroleum industry, oily water appears in the stages of production, transportation and refining, as well as when using derivatives. The production phase is by far the largest source of this pollution. During the production process, oil is commonly extracted along with water and gas. When present in water, oil can represent 50% of the liquid volume and even approach 100% at the end of the productive life of wells [2]. In addition, the refining process involves the use of large amounts of water, especially for distillation, hydrotreating, desalination, and cooling processes, leading to important water pollution problems [3,4]. The expected growth of the global oil demand in the next two decades indicates that production and discharge of polluted effluents from the oil refining industry will continue to grow [5].

The composition of this polluted water is highly variable and complex. Depending on its origin, it can contain a wide variety of chemicals, such as organic salts, aliphatic and aromatic hydrocarbons, oils and greases, metals, and occasionally radioactive materials. In

particular, the concentration of phenolic compounds can be in the range of 20–200 ppm [2,3], while, e.g., EPA sets the maximum permissible limit of phenol discharge in wastewater in less than 1 ppm [1,6].

These streams are difficult to treat due to large concentrations of oil. Several conventional techniques have been used to remove the oil from water: flotation, chemical coagulation, biological treatment (including wetland disposal), packed bed adsorption, evaporation, membrane separation, reverse osmosis, etc., but the success of those techniques has never been complete for reasons that range from economic considerations to poor removal yields, especially when considering diluted toxic compounds [7]. For example, ultrafiltration (UF) is a very promising for the removal of oil from water, but it has been limited by economic obstacles due to severe membrane fouling [8]. Also, the conventional biological treatment of the organic compounds contained in oil industry wastewaters has been widely demonstrated; however, it is now known that recalcitrant compounds may contribute to chronic toxicity in reclaimed environments [9].

Electrochemical techniques, such as electroflotation, electrodecantation, EC or electrokinetic remediation (removal of organic, inorganic and heavy metal particles from the soil by application of an electric potential), are easy to implement, and only require robust and

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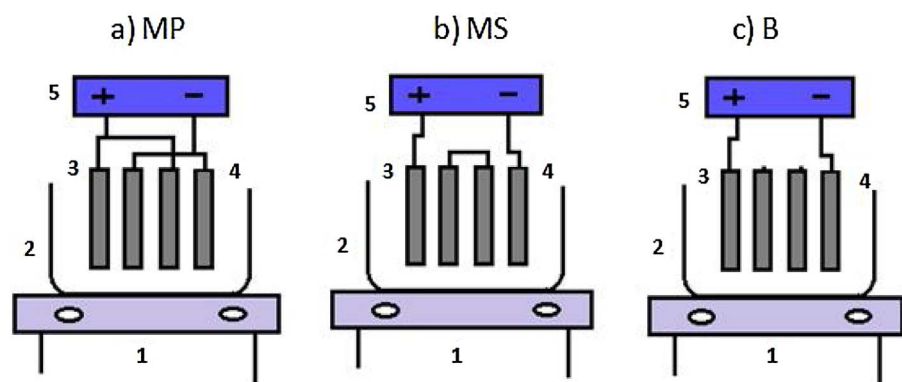


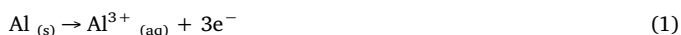
Fig. 1. EC reactor: (a) Monopolar electrodes with parallel connection (MP); (b) monopolar electrodes with series connection (MS); (c) bipolar electrodes with parallel connection (B). Legend: (1) Stirrer and temperature controller; (2) EC cell; (3) anode; (4) cathode; (5) power supply.

compact instrumentation, and a minimum amount of chemicals. These techniques might have economic and environmental advantages compared with other more traditional methods that make an extensive use of chemicals. Therefore, electrochemical techniques offer an efficient alternative for the removal of contaminants from industrial wastewaters [10–14].

EC is an electrochemical process where a sacrificial electrode is used to *in situ* generate a coagulant agent (usually iron and aluminum hydroxides) that breaks emulsions and creates flocs that settle and help separating water contaminants [10,15,16]. The reactions that occur when using aluminum and iron electrodes in EC are described by Mechanism 1 and 2, respectively:

Mechanism 1

At the anode:

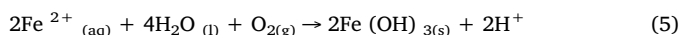


At the cathode:



Mechanism 2

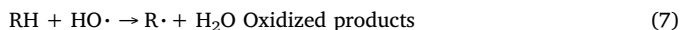
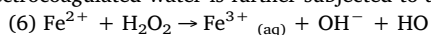
At the anode:



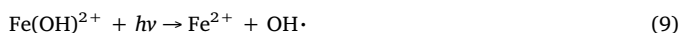
At the cathode:



The generation of gases (O_2 and H_2) also produces the appearance of flotation that can provide extra physical removal of the organic material. It is also possible to apply combined treatments. The combination of EC and the Fenton reaction is highly effective because of the generation of strong oxidizing hydroxyl radicals. In that coupling, the electrocoagulated water is further subjected to a Fenton process:



Another possible coupling is to subject electrocoagulated water to a photo-Fenton process. It has been established that the photo-Fenton reaction further improves the degradation of organic pollutants, either by direct photolysis, or by increasing the production of hydroxyl radicals according to the following reactions [17,18]:



The objective of the present study was to investigate the removal of phenolic compounds present in an oil refinery wastewater by a coupling between an EC and a Fenton or a photo-Fenton process. The effect of

several EC variables, such as current density and the nature, configuration and orientation of the electrodes, among others, was studied. The electrocoagulated water was then treated with a Fenton or a photo-Fenton system, and the effect of concentration of Fe^{2+} and H_2O_2 and treatment time on reaction efficiency was studied. The removal of pollutants was determined by quantification of total phenols and TOC content.

2. Experimental part

The wastewater samples were taken from an effluent of a petroleum refining industry located in Mexico. They were collected in polypropylene bottles and taken to the laboratory for preservation at 4 °C before use. All the chemical reagents used in this study were, at least, of reagent grade. Water used to prepare aqueous solutions was Milli-Q grade.

2.1. EC treatment

The EC treatment was carried out with aluminum and/or iron plates of 36 cm² of contact area, and with a gap between plates of 1 cm. These plates were previously cleaned up with an emery cloth to remove surface oxides and then degreased with acetone. Plates were placed vertically in a rectangular polyethylene reactor (9 cm × 6 cm × 4 cm) that contained a wastewater volume of 100 mL and experiments were conducted at constant current for 20 min. A 200 Power Pac power supply was used to feed the electrodes as shown in Fig. 1. The necessary amounts of NaCl (between 2 and 10 g L⁻¹) were added to the solutions to maintain an applied voltage between 4 and 5 V.

2.2. Fenton and photo-Fenton processes

To perform the Fenton treatment to the electrocoagulated water, a 30% H_2O_2 aqueous solution was prepared and added in suitable volumes to the electrocoagulated water sample while keeping a constant stirring. The pH of the electrocoagulated solution was previously adjusted to 3 with NaOH or H_2SO_4 . A 0.01 mol L⁻¹ $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ solution was added to the reaction solutions in the volumes needed to generate the desired Fe^{2+} initial concentrations. The same procedure was used to apply the photo-Fenton process along with irradiation of the reactor with four 25 W black-light compact fluorescent UVA lamps.

2.3. Analytical procedure

The total phenols content was determined by the 4-aminoantipyrine method according to the NMX-AA-050-SCFI-2001 procedure at a wavelength of 510 nm [11], using a Cary UV-vis 100 spectrophotometer (detection limit 0.01 mg L⁻¹). TOC content was determined with a Shimadzu VCSH TOC analyzer (detection limit 1 ppm). The pH was measured with an Oakton 1100 pH meter. Physicochemical parameters,

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