



An analytical study of the electrochemical degradation of methyl orange using a novel polymer disk electrode



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ABSTRACT

This study presents the degradation of methyl orange in a synthetic solution using an electro-oxidation technique. A novel polymer disk electrode was fabricated in this work using injection molding. Such an electrode is low cost, mass produced, and efficient to achieve a complete decolorization of methyl orange in the synthetic solution. The performance of the electrode was studied comprehensively. The degradation of methyl orange was accomplished in only 800 s. at optimum operating conditions of 0.1 M KCl electrolyte solution and constant applied current of 5 mA. The results revealed that the degradation of dye follows an indirect electro oxidation mechanism. It was also found that stirring is a crucial requirement to improve the mass transfer and to enhance the decolorization rate. The effect of changing the applied current, supporting electrolyte, electrolyte concentration, and initial dye concentration were also investigated.

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1. Introduction

Degradation of dyes in textile wastewater is a major concern in environmental science. It has been estimated that at least 10% of dyes used in industries are discharged to the environment [1]. This represents more than 8000 tons of dyes per year that is wasted nowadays [2]. Textile industries, in particular, release as much as 50% of their processed dyes to the environment [3]. Thus, it is not surprising that removal of dyes from textile effluents has drawn great attention from many researchers in this field.

Removal of colors from wastewaters can be achieved using biological, physical, and/or chemical treatment techniques [4]. Both physical and biological treatment methods are usually environmental friendly [5]. However, satisfactory results are not always promising, especially with biological methods where treatment is inefficient for industrial wastewater containing non-biodegradable dyes [6]. Chemical treatment techniques, on the other hand, can be efficient, but complete degradation is normally costly and sometimes complicated especially when many chemicals are added [7]. Recently, it was found that combining more than one treatment technique could lead to reasonable improvement in the treatment efficiency and reduction in the process cost [8]. Hybridized treatment methods in the textile industry have been discussed comprehensively by many authors [9–11].

Electrochemical treatment, on the other hand, is a single, yet efficient treatment technique. Degradation of dyes electrochemically is not only safe for the environment, but it also does not involve chemical reagents in the process [12]. In electrochemical degradation, electrons act as the reagents that create chemical changes to the dyes. Moreover, the broken down compounds in the electrochemical degradation process are usually nonhazardous [13] making the process clean and hence attractive for the treatment of pollutants [14]. Electrochemical treatment has received great attention due to its efficient applicability to a wide range of textile water compositions and flow rates, thus, making it a suitable candidate for both micro and macro scale industrial processes [15].

Martínez-Huitle and Brillas [16] reviewed several electrochemical treatment methods for dye removal in wastewater. Among those, the electrochemical oxidation technique [17] was one of the most distinctive methods. Removal of azo dyes in particular was achieved using several techniques, some of which were summarized by Singh and Arora [13]. For example, the UV irradiation technique, in the presence of hydrogen peroxide (H₂O₂) was investigated for the degradation of methyl orange [18]. Electrochemical treatment techniques were also reported extensively for the degradation of methyl orange [7,19]. A wide range of electrode materials could be used for the electrochemical degradation of azo dyes in general and methyl orange in particular. Platinum electrodes combined with either microwave activation [20], or a redox mediator [21] showed successful degradation of methyl orange. Dual iron and graphite anodes in the presence of catalysts [22] have also revealed promising results where complete decolorization of methyl orange was

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achieved after 60 min. Kong et al. [12] reported a successful attempt for the removal of methyl orange in 20 min using electro-oxidation on an exfoliated graphite electrode. However, to the authors' knowledge, complete degradation of methyl orange using both a rapid decolorization technique and an inexpensive electrode is limited in the literature.

In this paper, the authors fabricated a novel polymer disk electrode using an injection molding technique. These electrodes were evaluated for the treatment of a synthetic solution containing methyl orange as model. In comparison with previously published studies, these electrodes are low cost, mass produced, and found to be efficient for such application. The treatment technique involves the electro-oxidation of KCl solution on the polymer electrode. The utilized technique in this study is featured by its simplicity and rapidity for dye degradation. Moreover, the performance of the disk polymer electrode was compared to the commercially available electrodes for the degradation of methyl orange dye. The effects of changing the operating conditions, stirring of solution, type and concentration of supporting electrolytes, applied current, and initial dye concentration were also investigated.

2. Material and methods

2.1. Chemicals and reagents

Methyl orange dye (MO) ($C_{14}H_{14}N_3O_3SNa$; molar mass 327.3 g/mol) was used as the target organic pollutant. It was purchased from Aldrich as a powder and used without further purification. Different concentrations of MO dye (13, 20, 30 and 40 ppm) were prepared using deionized water in 0.1 M KCl (Scharlau Chemie, Spain) solution as an electrolyte unless otherwise stated.

2.2. Apparatus

Voltammetric measurements were performed using an Autolab electrochemical system (μ PSTAT III, Eco Chemie B.V., Utrecht, The Netherlands) controlled with GPES software (v 4.9) running on a PC. A conventional three-electrode system was used in a 50 mL standard glass voltammetric cell (Metrohm, Switzerland). The cell was equipped with a double junction Ag/AgCl reference electrode and a Pt wire counter electrode both from Metrohm. A disk polymer electrode was used as the working electrode. A PTFE magnetic stirring bar, agitated by a magnetic stirrer (Corning PC 351), was placed in the bottom of the cell for stirring the solution during the degradation process. The performance of the disk electrode was compared to that of conventional 2 mm glassy carbon, gold, and platinum disk electrodes (Metrohm, Switzerland).

Chronoamperometric/galvanostatic measurement, at different bias currents, was conducted on 10 ml of different concentrations of MO solutions, all containing 0.1 M KCl. The extent of color removal was determined using UV-Vis spectroscopy (path length 1 cm, apparatus: Shimadzu UV-1800) at its maximum absorption ($\lambda = 465$ nm). Samples were taken every 200 s, unless otherwise stated. All measurements were performed at room temperature (normally 21 °C).

2.3. Disk electrode fabrication

The working electrode was fabricated in house (School of Chemical Engineering and Analytical Science, University of Manchester). These electrodes were injection-molded from a conducting polymer (40% carbon fiber filled-high impact polystyrene (HIPS), RTP 487, RTP Company (UK) Plastics Ltd., Bury, UK). Injection-molded parts were fabricated using a Babyplast 6/6 injection-molding machine (Cronoplast SA, Barcelona, Spain). Mold tools were fabricated from brass bar stock using a CAT3D M6 CNC milling machine (Datron Technology Ltd, Milton Keynes, UK) and conventional tungsten carbide end mills of diameters ranging from 0.15 to 1.0 mm. The molding conditions were as follows: plasticizing temperature 230 °C; injection chamber temperature 220 °C; injection nozzle temperature 200 °C; injection pressure

50 bar; injection time 6 s, and cooling time 12 s. The disk electrode surface was created by an over molding procedure, where clear polystyrene (Northern Industrial Plastics Ltd., Chadderton, UK) flows around the previously fabricated carbon fiber parts. The working electrode was molded in plates measuring 20 cm \times 15 cm \times 0.4 cm; individual plate electrodes measuring 1 cm \times 1 cm \times 0.4 cm were directly cut from these sheets (Fig. 1a). The diameter of the disk electrode was 2.5 mm.

The surface of the electrode was examined using a scanning electron microscope (SEM) (EVO® LS10, Carl Zeiss NTS GmbH, Germany) with an EDS energy dispersion X-ray spectrometer (Bruker AXS X Flash Detector 4010). The SEM image of the disk working electrode surface is shown in Fig. 1b; close examination clearly shows the carbon fibers (darker areas) and their random dispersion in the polystyrene matrix (lighter areas).

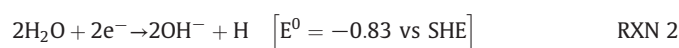
3. Experimental results and discussion

3.1. Cyclic voltammetric studies

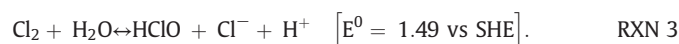
A peak at 0.7 V (oxidation potential) was observed in the presence of dye solution, but not observed in the absence of the dye solution (see Fig. 2). This peak is probably attributed to the adsorption of the dye molecules on the electrode surface and not the direct electro-oxidation of the dye. This is because further increase in the dye concentration did not cause any increase in the peak height. The reversible peaks at approximately +0.280 V are probably amine peaks as described by Kalyuzhnyi et al. [23] and Zille et al. [24].

In addition, scanning between zero and 1.5 V showed an increase in the anodic current at higher potential values. This current increased with increasing electrolyte concentration and scan rate.

It is known that the electrolysis of chlorine solution in an undivided cell leads to the direct oxidation of chloride ions at the anode to chlorine (RXN 1) and reduction of water at the cathode to hydroxide ions and hydrogen gas (RXN 2) [16]. The former explains the rise in the anodic current at potentials greater than 1 V.



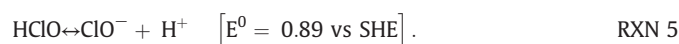
Moreover, at the pH range studied (pH = 3–8) the generated chlorine can be hydrolyzed rapidly to generate hypochlorous acid and chloride ions as follows (RXN 3) [16]:



Trichloride ion Cl_3^- can also be formed but in very low concentrations up to pH ca. 4.0 [16] according to the following reaction:



The hydrochlorous acid in the solution is in equilibrium with the hypochlorite ion according to the following reaction:



Thus, from these CV results, it is suggested that the electrochemical degradation of the dye in this experiment (high applied potentials) follows the indirect electro-oxidation mechanism [25], which involves the reaction of the dye molecules with the strong oxidants generated from the direct anodic oxidation of the chlorine ions present in the electrolyte [16].

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