



Research paper

A miniaturized injection-moulded flow-cell with integrated conducting polymer electrodes for on-line electrochemical degradation of azo dye solutions



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ABSTRACT

This study investigates the effectiveness of a novel micro electrochemical flow-cell containing carbon fiber electrodes for the degradation of Azo dye solutions. The flow-cell was fabricated by an injection moulding technique, integrating three electrodes system design using an overmoulding procedure. All electrodes were fabricated from carbon fiber polymer, however, the reference electrode was then coated with Ag/AgCl paste. The effect of the process variables such as flow-rate, NaCl concentration, applied bias current, and solution pH on the degradation process were comprehensively studied. At optimum conditions, complete degradation of the dye was achieved on the bare carbon fiber electrode, without the need for surface modification, in 150 min upon the application of 15 mA bias current. The degradation process was suggested to follow the indirect oxidation mechanism where the presence of chlorine ions was very essential. Moreover, the micro-flow-cell had proven its stability over the entire operation time, which indicates its practicality for such on-line applications.

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1. Introduction

Synthetic dyes had found extensive applications not only in textile industries but in many state-of-the-art technologies [1]. However, the release of their untreated or partially treated dye-containing effluents, into soil and water bodies had caused serious environmental problems, due to the toxicity nature of most of these dyes and their recalcitrance to microbial degradation.

In view of this, the removal of dye stuff from wastewater was one of the well investigated problems that attracted many researchers. Various chemical, physical [2,3] and biological [4] techniques were employed among which the chemical oxidation methods were the most commonly used [5]. One classification of chemical oxidation methods that had had proved their effectiveness as an alternative is the electrochemical technology. The rapid developments in this technology arise from its simplicity, versatility, and easiness to being automated and control. Moreover, electrochemical methods are compatible to the environment since they depend on electrons as the main reagent “clean reagent” to degrade all the organics without the

production of any pollutant. They are also considered highly efficient technologies in terms of degradation rate, energy consumption, and cost, if well optimized and used [6,7]. However, it is worthwhile noting that the performance of the electrochemical oxidation methods and hence rate of degradation is highly governed by the electrode material upon which the reaction take place. Thus, the two major factors to be noted when selecting the electrode material are their surface structure and conductivity. Usually electrochemical degradation process takes place in bulk batch reactors with different electrode materials such as metals [8], metal oxides [9] boron-doped diamond (BDD) [10], etc. This process takes long time and requires considerable power for mixing. Moreover, such electrodes are either expensive or require advanced fabrication procedure. Carbonaceous materials [11], on the other hand, have been reported as low cost alternative, however, for these electrodes to experience good degradation abilities, their surface structure usually need to be modified [12,13].

Since with electrochemical oxidation the degradation reaction occurs at the surface of the electrode; miniaturization can be highly effective for such applications. Miniaturization can provide the advantages of increasing the reaction rate, and reducing the degradation time as well as the power consumption due to the increase in the effective surface area. There are some limited reports on the use of miniaturization for

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such application. For example, Sun et al. reported 90% Methyl orange degradation in 60 min using activated carbon fibers modified with carbon nanotubes [14], while 95.6% degradation was achieved in 180 min on platinum–bismuth bimetallic carbon paper (Pt–Bi/C) nanostructured electrode [15].

Accordingly, this study aims at investigating the applicability of a miniaturized, low cost, disposable, electrochemical micro-flow-cell for the degradation of dye solutions. This novel design micro-flow-cell has been used for the first time for such applications. Degradation was measured on a bare carbon fiber polymer electrode fabricated by injection moulding technique. The three electrodes were incorporated into a flow cell by an over moulding procedure. A typical azo dye, methyl orange (MO), was selected as the model pollutant in this study, based on the fact that the azo dyes are the one most widely used and account 65–70% of the total dyes produced [2].

2. Materials and methods

2.1. Chemicals

Methyl orange dye (MO) ($C_{14}H_{14}N_3O_3SNa$; molar mass 327.3 g/mol, Aldrich, UK) as a model organic pollutant, used as a powder without further purification. Different concentrations of MO dye (20, 30, 60, 70 mg/L) were prepared using deionized water in 0.1 M sodium chloride solution (NaCl) (Scharlau Chemie, Spain) 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. The electrochemical degradation of the dye was carried out in a miniaturized flow-cell integrated with a three electrode system, as described in the fabrication section. The solution was continuously pump into the micro flow-cell using a variable-flow pump purchased from Cole-Parmer. Chronoamperometric/Galvanostatic measurement, at different bias currents, was conducted on different concentrations of MO solutions, all containing 0.1 M NaCl. The degree of decolorization was determined at its maximum absorption ($\lambda = 464$ nm) using UV–Vis spectroscopy (path length 1 cm, apparatus: Shimadzu UV-1800). IKA®-Werke Stirrer and Heater (Germany) was used for temperature control experiments.

2.3. Fabrication of the micro flow-cell

The micro-flow-cell was fabricated in-house at the University of Manchester [16]. The fabrication procedures were as follows:

Blocks of aluminium (75 mm \times 75 mm \times 10 mm) [Righton Metals, Manchester, UK] were milled using micrograin tungsten-carbide 2 flute routers [Toolex Ltd., Wincarton, Somerset, UK] to generate the milled aluminium mould for the injection moulding machine [Babyplast 6/6 or 6/10, Cronoplast SL, Barcelona, Spain]. Two pairs of moulds comprising of the fixed and moving parts were produced for the injection moulding of the electrodes, the overmould and the fluidic channel. The moving part was mounted onto the mould bolster mount, where the ejector pins were inserted. The bolster comprise of 4 \times 5 array of 2 mm holes for the insertion of the ejectors pins and four 5 mm holes for the bigger ejector pins. The shortest ejector pin was placed into the hole corresponding to the sprue. The two mould halves were aligned using the dowel pin holes on both sides of the moulds.

The moulding process is divided in to two steps: (1) moulding of the electrodes and (2) moulding of the flow cell. The polymer electrodes are injection moulded from a conducting polymer (40% carbon fiber filled-high impact polystyrene (HIPS)) [RTP 487, RTP Company (UK) Plastics Ltd., Bury, UK] and the moulding conditions are shown in Table 1. The

Table 1
Injection moulding operating conditions.

Moulding parameters	Carbon fiber	HIPS
Plasticizing temp./°C	230	230
Injection chamber temp./°C	220	220
Injection nozzle temp./°C	200	200
Injection pressure/bar	65	50
Injection time/s	3.5	2.5–3.5
Cooling time/s	4	3–8
Load material/mm	25	40

electrode to be used as a reference electrode is coated with Ag/AgCl paste (60% Ag and 40% AgCl) [C6003P7, Gwent Electronic Materials, Pontypool, UK] and cured at 60 °C in an oven for 30 min. A schematic of the electrodes with their dimensions are shown in Fig. 1.

The flow cell is moulded from crystal polystyrene [Northern Industrial Plastics Ltd., Chadderton, UK] and the moulding conditions are shown in Table 1. It comprises two plates; a base plate for the insertion of the electrodes and a top plate containing the flow channel and the integrated fluidic connectors. The fabrication process of the flow-cell involves two stages: overmoulding and flow channel production. In the first stage, the previously fabricated electrodes are incorporated into the base plate of the flow cell by an over-moulding procedure, where clear polystyrene flows to fill the cavity around the electrode thus creating the base plate. In the second stage, the top plate is moulded with channel dimensions of 1 mm wide, 200 μ m deep and 30 mm long. A schematic of the side view of the complete flow cell with the corresponding dimensions is shown in Fig. 2.

Finally, the two plates (top and base) were sealed together using a 500 W ultrasonic welder [Mini delta 9500, FFR Ultrasonic Ltd., Queniborough, Leicestershire, UK]. This requires an energy-directing ridge of 0.25 mm deep to be moulded around the channel during the milling and moulding stage. Welding was then achieved by applying a pressure/force on the two plates. Consequently a vibrating ultrasonic energy is generated and travels through the polymer, generating frictional heat at the ridge between the two parts. As a result, the polymer melts at the joint, and then solidifies when the ultrasonic energy stops creating a molecular bond between the two parts, thus, forming a complete device. A picture of the complete and ready to use electrochemical flow cell with the integrated electrodes is illustrated in Fig. 3.

The stability of the Ag/AgCl reference electrode was examined by continuously monitoring its potential with respect to a conventional double junction Ag/AgCl reference electrode using a high input impedance meter. The results revealed potentials in the range of approximately –68.9 to –86.1 mV with an average of approximately –77.1 mV with an observed drift at approximately 0.125 mV/h.

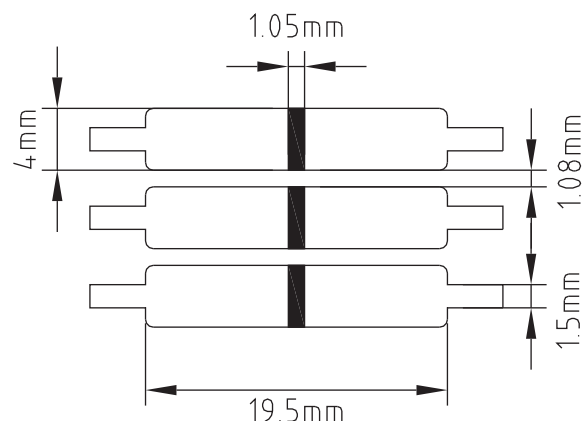


Fig. 1. Schematic of the designed polymer electrodes.

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