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Electrochemical treatment of alkali decrement wastewater containing terephthalic acid using iron electrodes

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ABSTRACT

In this study, treatment of simulated alkali decrement wastewater containing terephthalic acid (TPA) has been investigated by electrochemical treatment using stainless steel as electrode material. Five operational parameters, namely current density: 31.25–156.25 A/m^2; electrode gap: 1–2 cm; NaCl concentration: 0–2 g/l; pH: 7–12 and TPA concentration: 400–1000 mg/l were investigated for their effect removal efficiency. For a solution having 400 mg/l TPA and 560 mg/l chemical oxygen demand (COD), 72% COD removal and ≈77% TPA removal efficiency was obtained at optimal conditions of current density ≈ 118 A/m^2, electrode gap = 1 cm and pH ≈ 7. Presence of ethylene glycol in the wastewater was found to decrease the treatment efficiency. Settling characteristics of the slurry obtained at optimum condition have been studied. Thermal decomposition of residues has also been investigated.

1. Introduction

Textile industries generate wastewater comprising of residual dyes and auxiliary chemicals is a major source of pollution [1]. During alkali decrement method, the weight of polyester fabrics gets reduced by 15–30% due to degradation of polyethylene terephthalate (PET) because of the action of sodium hydroxide solution. This PET forms sodium terephthalate which is one of the major pollutants in the wastewater discharged from polyster facbric manufacturing industry. Also this wastewater has very high pH [2]. PET is manufactured by polymerizing terephthalic acid (TPA) and ethylene glycol with an acid catalyst. TPA is the starting material for making polyester with annual production exceeding 50 million [3]. It is very toxic in nature as it has endocrine disrupting ability. It also affects reproductive system and overall development in humans. In addition to that, it is carcinogenic in nature [4]. Chemical treatment using strong acids to decrease the pH and precipitate TPA have shown chemical oxygen demand (COD) removal up to 90% [5,6]. However, this method of pH adjustment consumes a lot of acids. Biological methods reported in the literature [7,8] have low COD removal efficiency and are expensive for large scale treatment.

Electrochemical (EC) treatment has gained reputation as a water treatment technology in recent years. It is now considered to be a simple, reliable and cost-effective tool for treatment of a wide variety of wastewaters with high removal efficiencies [9–12]. Various techniques of electrochemical treatment such as electrofloatation, electrocoagulation and electrooxidation have been reported in the literature [13].

The objective of the present study is to investigate the removal of TPA by EC treatment using stainless steel as electrode material. Several parameters namely current density, inter-electrode distance, NaCl concentration, initial pH and initial TPA concentration were investigated for their effects on the TPA removal efficiency. Effect of presence of ethylene glycol on treatment efficiency has also been studied.

2. Experimental

2.1. Chemicals and wastewater

All the chemicals used in the study were of analytical reagent (AR) grade. TPA was supplied by HIMEDIA Pvt. Ltd., Mumbai, India. Sodium hydroxide used for preparing the initial basic medium was supplied by MERCK. Alkalized TPA wastewater was prepared as per the method described by Wen et al. [14]. The characteristics of the alkalized TPA wastewater prepared and used in the present study are given in Table 1.

2.2. Reactor

All the EC experiments were performed in a 2 L cylindrical reactor (internal diameter = 0.12 m) made of polymethylmethacrylate

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Table 1
Characterization of alkalized TPA wastewater before and after electro-chemical treatment at optimum conditions.

Parameter	Before treatment	After treatment
COD (mg/l)	560	157
BOD (mg/l)	90	28
TOC (mg/l)	230	90
TPA (mg/l)	400	93
Chloride (mg/l)	580	220
Conductivity (mS/cm)	3.24	3.20
Total dissolved solids (mg/l)	2.6	2.9
pH	7	9.2
Iron (mg/l)	0	12

(PMMA). The dimensions of each electrode were 80 mm × 100 mm × 3 mm. The area of electrode dipped inside the solution was 80 mm × 90 mm. Current density was maintained constant using precision digital direct current power supply apparatus (0–20 V, 0–5 A). Agitation of the solution in the reactor was done using magnetic stirrer.

2.3. Analysis of physico-chemical parameters

The physico-chemical parameters pH, conductivity, COD, biological oxygen demand (BOD), total solids (TS) and chloride concentration were analyzed according to standard methods [15]. COD was measured using digestion unit (DRB 200, HACH) and double beam UV–vis spectrophotometer (Lovibond Checkit Direct CODVario). Concentration of TPA was measured using HPLC (Waters 1525 Binary HPLC Pump, Waters 2487 Absorbance Detector, Waters 2414 Refractive Index detector). HPLC was operated in an isocratic mode with a C-18 column at room temperature with the mobile phase at a flow rate of 0.8 mL/min and the detector set at 250 nm. The mobile phase consisted of acetic acid (2 vol%), 2-propyl alcohol (7 vol%), and water (91 vol%) with phosphoric acid (300 μl/l solvent) [16]. The concentration of total iron was measured in the wastewater using atomic absorption spectrometer (AVANTA GBC, Australia). Total organic carbon (TOC) was determining using TOC analyser (TOC-V-CSN 39208967, Shimadzu).

2.4. Experimental procedure

In the beginning of each run, 1.6 L solution of the desired TPA concentration was poured into the reactor and NaCl was used as an electrolyte to enhance the ionic strength of the solution. Initial COD was measured for all different initial concentrations. In all experiments, initial pH was adjusted using 0.1 N NaOH and 0.1 N H_2SO_4. Current density and inter-electrode distance was maintained constant during each experiment. After 120 min of EC treatment, samples were drawn from supernatant liquid and its final COD was immediately measured and percentage COD removal was calculated.

2.5. Settling characteristics

The suspensions after the EC operation were thoroughly mixed and then the resultant slurry was used for determining the settling characteristics of the sludge. The sedimentation was carried out using a 1 L graduated glass cylinder having a total height of around 105 cm. No stirring was done during the test. The position of the upper interface formed between the clear supernatant liquid and solids was noted as a function of time and a graph was plotted. The frequencies of these measurements were chosen in such a manner so as to obtain full settling curve.

2.6. Physico-chemical characterization of scum and sludge

Scanning electron microscopy (SEM) images of the sludge produced after coagulation and flocculation treatment were obtained using the scanning electron microscope (LEO 435VP, England) operating with SE1 detector. Electron dispersive spectroscopy (EDAX) analysis was done to determine elemental composition of the different sludges using the FE-SEM (QUANTA 200 FEG). To identify the various functional groups present in the sludge, FTIR analysis of the sludge was carried out using a FTIR spectrophotometer (Nicolet Avtar 370, USA). Thermogravimetric analysis (TGA) of the sludge was carried out using a thermal analysis instrument (Perkin-Elmer Pyris Diamond). Approximately 10 mg of the sample was heated at 10 °C/min from ambient upto 1000 °C under air atmosphere at a standard air flow rate of 200 ml/min.

3. Results and discussion

3.1. Effect of pH

Fig. 1 (curve a) shows the variation of COD removal efficiency with varying pH. The initial pH different concentrations of TPA in sodium hydroxide solution was above 12. The pH range for investigation is limited by the fact that terephthalte starts precipitating at pH lower than 6. Maximum COD removal was obtained at initial pH ≈ 7. It is well known from speciation chemistry of Fe(III) that they exist as as $Fe(OH)_3$ at pH ≈ 7. As the pH increases above 7, $Fe(OH)_4^-$ become the dominant species. Terephthalate anions show better removal with $Fe(OH)_3$ flocs [14]

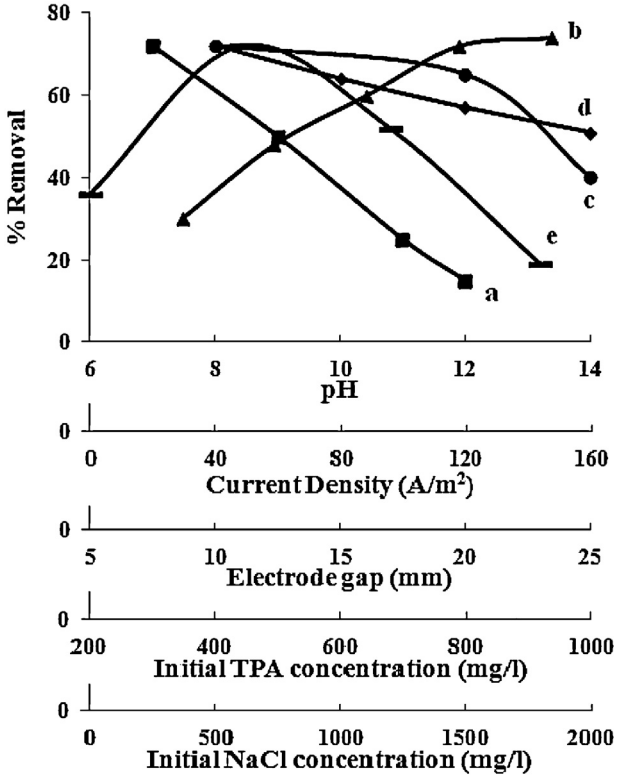

Fig. 1. Effect of various parameters on COD removal [(a): pH (current density = 118 A/m^2, electrode gap = 10 mm, initial TPA concentration = 400 mg/l, initial NaCl concentration = 600 mg/l), (b): current density (pH = 7, electrode gap = 10 mm, initial TPA concentration = 400 mg/l, initial NaCl concentration = 600 mg/l), (c): electrode gap (pH = 7, current density = 118 A/m^2, initial TPA concentration = 400 mg/l, initial NaCl concentration = 600 mg/l), (d): initial TPA concentration (pH = 7, current density = 118 A/m^2, electrode gap = 10 mm, initial NaCl concentration = 600 mg/l), (e): initial NaCl concentration (pH = 7, current density = 118 A/m^2, electrode gap = 10 mm, initial TPA concentration = 400 mg/l)].