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A Novel, Pilot Scale Electrolysis System for Production of P-Aminophenol using Parallel Screen Electrode

The present research investigates the nitrobenzene reduction to p-aminophenol by using a novel pilot scale electrolysis system consisting of parallel amalgamated copper screens electrode followed by direct acetylating to paracetamol. The preparative electrolysis of nitrobenzene was carried out using supporting electrolytes consisting of (2M) H₂SO₄ in a solution of 50% 2-propanol/50%water (v/v) and two modes of operation galvanostatic (controlled current) and potentiostatic (controlled potential). A Factorial experimental design was adopted for studying the effect of electrode potential, nitrobenzene concentration, flow rate, and number of screens in the electrode on the yield of p-aminophenol.

The results indicated that yield of p-aminophenol increases with cathode potential more positive than (-400mV vs. SCE), increasing flow rate, decreasing nitrobenzene concentration, and number of screens. The best results were obtained at cathode potential (-375mVvs.SCE), catholyte flow rate (111cm³s⁻¹), nitrobenzene concentration (0.1gcm⁻³) and number of screens not grater than five in which p-aminophenol yield was (80%) ,with maximum conversion (83%) of p-aminophenol to paracetamol.

Key words: Nitrobenzene, P-aminophenol, electrochemical reduction, Preparative electrolysis, Screens electrode.

1. INTRODUCTION

P-Aminophenol is a commercially important chemical intermediate used in the manufacture of several analgesic and antipyretic such as paracetamol, acetanilide, and phenacetin etc. Apart from pharmaceuticals, p-aminophenol is also used in the manufacture of various industrial dyes such as sulfur and azo dyes, which are especially useful in dyeing hair, furs and feathers[1]. P-Aminophenol is a strong reducing agent and is also marketed as a photographic developer under the trade names of Activol, Azol, Certinal, Citol, Paranol, Rodinol, Unal, and Ursol-P either alone or in combination with hydroquinone. The oxalate salt of p-aminophenol marketed under a trade name of Kodelon is used as a corrosion inhibitor in paints and anticorrosion – lubricating agent in two – cycle engine fuels. Another use of p-aminophenol is in the production of rubber antioxidants [2].

Because of its industrial importance, synthesis of p-aminophenol has attracted considerable interest. It is manufactured chemically by several methods: (i) one method consists of the reduction of p-nitrophenol

with iron and HCl, which causes waste disposal problems; (ii) phenol may be treated with HNO₃, which on reduction with poly – sulfide, gives p-aminophenol with very poor yield; (iii) nitrobenzene may be reduced to p-aminophenol in the presence of catalysts such as Pt or Pd and this method is highly expensive. The recovery of the catalysts used is a significant economic factor in this process [3]. An alternative method for production of p-aminophenol is by the electrolytic method, which is clean and without pollution problems. The yield is higher in comparison with the chemical method, leading to purer product. The electro reduction of p-nitrophenol to p-aminophenol involves 6 Fmol⁻¹ whereas the electro reduction of p-nitrosophenol or nitrobenzene involves only 4 Fmol⁻¹. The maximum yield of p-aminophenol from p-nitrosophenol was only (78%) while a higher yield was obtained from nitro benzene [4].

The electro reduction of nitrobenzene in acidic medium has been well studied, and known for nearly a century. A simplified version of the reduction sequence is shown in figure 1[5]. The reduction of nitrobenzene involves the transfer of two electrons to form nitrosobenzene. The nitrosobenzene is then further reduced to phenyl hydroxyl amine with a further transfer of two electrons. Finally, with the transfer of two more electrons the phenyl hydroxyl amine formed is reduced to aniline.

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