

PERCHLORATE PREPARATION COMBINED WITH ZINC DEPOSITION IN A PACKED BED ELECTROLYZER

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The results of a preliminary investigation have been presented on deposition of zinc with electrochemical preparation of perchlorate in a packed bed cell having a cationic exchange membrane.

Key words: Packed bed electrolyser, zinc deposition, perchlorate preparation

INTRODUCTION

The electrochemical production reaction in electrowinning arises as a result of a more efficient use of total electrolysis energy. Essentially the wasteful water electrolysis is replaced by a useful electrochemical reaction. The electrochemical reaction that has been studied as an anodic substitute in electrowinning is oxidation of Mn^{2+} to MnO_2 [1-2]. Simultaneous electrochemical removal of copper and chemical oxygen demand (C.O.D.) in simulated waste water in a rectangular packed bed electrode cell have also been reported [3]. In a recent work, the feasibility of using cationic membrane to combine electrowinning of zinc or copper from a sulphate electrolyte at cathode with electrochemical production of sodium perchlorate at the anode is presented [4]. In this paper investigations have been carried out on deposition of zinc with electrochemical production of perchlorate using cationic exchange membrane packed bed cell.

EXPERIMENTAL

Experiments have been carried out using a single packed bed membrane reactor which is schematically shown elsewhere [5-7]. The cell system comprised of a packed bed anode, made up of 3mm size lead dioxide coated graphite particles and a planar aluminium cathode in a divided compartment system with cation exchange membrane (C-20) supported by a perforated PVC plate. The exposed area of the bed was $2.5 \times 4.5 \text{ cm}^2$, its thickness being 1.85 cm with 41% voidage. The exposed area of the cathode was $9 \times 5 \text{ cm}^2$. The cell had been fabricated out of PVC with appropriate provision for electrical contact and for flow of anolyte as well as catholyte through respective chambers. The volume of anode and cathode compartments was approximately 80 ml and the volume of reservoir for anolyte and catholyte was one litre. The uniform flow of electrolyte in each compartment was achieved through the use of flow distributors situated at the bottom and top of the cell. The electrolyte was allowed to flow from the bottom and the outflow was recirculated. A constant current D C power supply was used. The catholyte was prepared using zinc oxide and sulphuric acid whereas the anolyte was a solution of potassium chlorate. First a trial experiment was carried out at maximum flow rate (180 l.h^{-1}) till there was no depletion in the concentration of electrolyte. Then the experiments were continued by varying the flow rates from 60 l.h^{-1} to 180 l.h^{-1} for 3 hrs at constant current. Periodically the concentration of anolyte and catholyte was

estimated using iodometry for chlorate and EDTA for zinc. Catholyte concentration was maintained by adding fresh zinc oxide corresponding to the assumed current efficiency 90%.

RESULTS AND DISCUSSION

The operating conditions are given in Table I. It has been found that the current efficiency for cathode reaction based on zinc

TABLE - I: Operating conditions*

Cathode	: Al sheet
Anode	: Lead dioxide coated graphite particles of 3mm size
Cathode current density (kA.m^{-2})	: 0.6
Anode current density (kA.m^{-2})	: 1.6
Catholyte concentration (g.l^{-1} of zinc)	: 86
Anolyte concentration (g.l^{-1} of chlorate)	: 41
Current (A)	: 1.82
Temperature (K)	: 305 - 313

deposit is around 92% at all the flow rates. The result of the experiment for depletion of concentration of anolyte and catholyte at maximum flow rate is presented in Fig.1. Both the concentrations decrease exponentially with time. After 3 hours of electrolysis there is not much change in the concentration of electrolyte. From the initial depletion rate and assuming the packed bed behaves as a plug flow reaction and the reservoir as a back mix reactor, the overall rate coefficient is computed to $5.5 \times 10^{-4} \text{ cm.s}^{-1}$ for zinc ion and $0.5 \times 10^{-4} \text{ cm.s}^{-1}$ for chlorates. The equations relating the concentration factors and mass transfer properties have been discussed elsewhere [5]. It has also been observed that there is not much significant difference in the rate of depletion for the range of flow rates studied. Generally in the anodic oxidation of chlorate to perchlorate, the reaction kinetic favours the electrolysis of water below 1.0 kA.m^{-2} . As a result, perchlorate is commercially produced at current densities between 1.5 and 4.5 kA.m^{-2} . In the present study using the anode and cathode current densities as 0.6 and

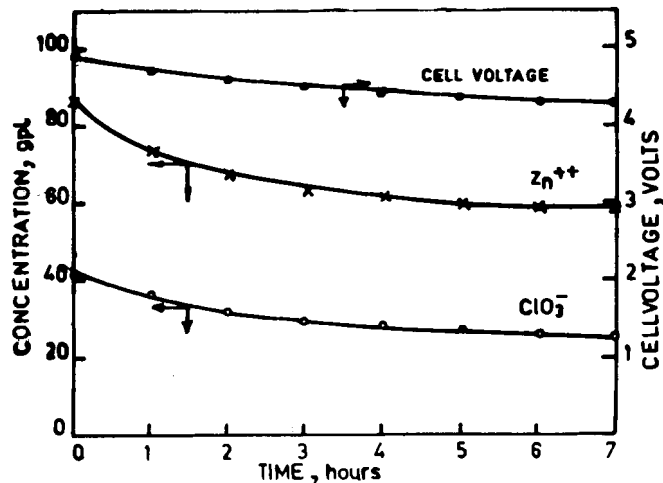


Fig.1: Variation of unreacted Zn^{++} , ClO_3^- and cell voltage with time of electrolysis

1.6 $kA.m^{-2}$ respectively the cell voltage for the combination cell is around 4.55 V compared to 8.4V for the sum of the cell voltages of the independent cells.

From this work, it can be concluded that both the useful products can be produced in packed bed reactor.

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