



Current–voltage characteristics in a hybrid electro dialysis–ion exchange system for the recovery of cesium ions from ammonium molybdophosphate–polyacrylonitrile



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HIGHLIGHTS

- I–V curves of EDIX for Cs recovery showed three regions as in electro dialysis.
- Limiting currents increased with electrolyte flow rates and anolyte concentrations.
- Operation at 80% LCD gave highest Cs recovery.
- Catholyte pH changed to alkaline at overlimiting currents, serving as indicator.

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ABSTRACT

A hybrid electro dialysis–ion exchange (EDIX) method was investigated for the recovery of cesium from cesium-sorbed ammonium molybdophosphate–polyacrylonitrile (AMP–PAN) ion exchange resin. Concentration polarization was studied by observing current–voltage (I–V) characteristics of a three compartment electro dialysis cell containing AMP–PAN in the middle compartment. The effect of process parameters such as flow rates of electrolytes and anolyte concentration on the current–voltage behavior was examined. Limiting currents, determined using Cowan plots, increased with electrolyte flow rates and anolyte concentration. In the present study, limiting current density (LCD) was measured for the electro dialysis system as a function of linear velocity and anolyte concentration using the equation $i_{lim} = aCu^b$. Coefficients a and b for relating limiting current density and flow velocity were evaluated for different anolyte concentrations and electrolyte flow rates. Application of overlimiting currents resulted in lower recovery of cesium from AMP–PAN. During continuous operation of the cell, the onset of concentration polarization was detected by observing the pH of the acidic catholyte which turned to alkaline.

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1. Introduction

Electro dialysis ion exchange is a hybrid electrochemical separation process, which attracted considerable attention in recent times. In electro dialysis, high current densities are desirable to achieve fast deionization with the lowest possible effective membrane area. But high operating currents are restricted by the occurrence of concentration polarization phenomena. Thus, electro dialysis is not economical for treating dilute solutions because of the high electrical resistance.

Introduction of ion exchange resin into the ion depleting compartment of an electro dialysis stack reduces the occurrence of concentration polarization, due to the ability of the resins to exchange counter ions. Thus, ion exchange resins reduce the power consumption considerably and increase the ion transport through the membranes. Studies were reported on the application of EDIX for the separation of monovalent and divalent species like Ni^{2+} [1–4], Co^{2+} [5], Cr^{6+} [6], Cs^+ [7], and Cu^{2+} [8–10] from dilute solutions.

Concentration polarization is a well-known phenomenon occurring at the interface between an ion exchange membrane and an electrolyte solution, when current passes through it. Due to the effect of counter ion present in the membrane the permeate ion moves through the ion exchange membrane at a faster rate than through the solution. Consequently, a depletion of electrolyte is created in the aqueous layer adjoining the membrane face and a concentration gradient develops in the

Abbreviations: EDIX, electro dialysis–ion exchange; AMP–PAN, ammonium molybdophosphate–polyacrylonitrile; CEM, cation exchange membrane; LCD, limiting current density; I–V, current–voltage.

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film that adjoins the membrane. This phenomenon is called concentration polarization. Because of this depletion of the salt at the membrane surface, the voltage drop dissipated in transporting ions across the boundary layer is higher than through the membrane. As a result, the energy consumption per unit of salt transported increases significantly. The point at which the ion concentration at the membrane surface becomes zero corresponds to the maximum transport rate of ions through the boundary layer. The current per unit area of membrane at this point is called the limiting current density. Once the limiting current density is reached, any further increase in voltage applied across the membrane will not increase ion transport or current through the membrane. Normally the extra power is dissipated by side reactions, such as dissociation of the water in the cell into ions.

Concentration polarization on the desalting membrane surface in electro dialysis was investigated by various researches by analyzing the current–voltage (I – V) relationship [11–13]. The presence of ion exchange resin in the electro dialysis setup enhances the transport of cations or anions under the application of electric potential and minimizes the concentration polarization [14–17]. A typical current–voltage relationship of an electro dialysis system consists of three distinct regions: ohmic region, limiting current region and overlimiting region [18–20]. The ionic transport both in the ohmic and limiting current regions can be explained by concentration polarization near ion exchange membranes. Overlimiting currents are attributed mainly to water splitting, current induced convection and co-ion leakage in the electro dialysis cell [21]. Concentration polarization can be partially controlled by circulating the salt solutions at high flow rates through the cell. The magnitude of concentration polarization is a function of various parameters including the applied current density, the feed flow velocity parallel to the membrane surface, the cell design and the membrane properties [22–24].

The limiting current density (i_{lim}) is obtained when the ion electrodiffusion transfer through a diffusion boundary layer near an ion exchange membrane is saturated and the boundary electrolyte concentration becomes small in comparison with its bulk value. The limiting current density in electro dialysis based on Nernst–diffusion model [25,26] is represented as Eq. (1).

$$i_{lim} = \frac{FCzD}{\delta(t_m - t_s)} \quad (1)$$

Korngold [14] suggested an equation for limiting current density with ion exchange resin in electro dialysis chamber, taking into account the lower surface contact between the solution and membrane

$$i_{lim} = \frac{FCzD}{\delta(t_m - t_s)} \frac{A_s}{A_t} \quad (2)$$

where, C is concentration of the diluate, D the diffusion coefficient, F the Faraday constant, z the valence, δ the boundary layer thickness, t_s and t_m are the ion transport numbers in the solution and the membrane respectively, A_s the membrane surface in direct contact with solution, and A_t the total surface of membrane.

It is difficult to determine the limiting currents directly by using the above equations either in electro dialysis or in electrodeionization. A basic empirical equation which is a function of the flow velocity and concentration is widely accepted and gives reasonable results within practical limits as shown below.

$$i_{lim,emp} = aCu^b \quad (3)$$

where, u is the linear velocity, and $i_{lim,emp}$ is the empirical limiting current density. Coefficients a and b in the equation depend on hydrodynamics and design parameters of the cell [27].

In our previous study [28], the EDIX performance was investigated for recovery of cesium from ammonium molybdophosphate-

polyacrylonitrile (AMP-PAN) ion exchange resin by keeping the resin in the middle compartment of an electro dialysis cell. It was observed that the recovery of cesium from AMP-PAN increased with increasing current density and regenerative solution concentration. The objective of present study is to investigate the current–voltage characteristics and factors affecting the limiting current density on cesium recovery from AMP-PAN using EDIX. The objective of the present study also includes determination of the empirical constants a and b for the system under investigation.

2. Materials and methods

Cesium solution of 750 ppm concentration was prepared using cesium chloride salt (analytical grade, SRL Chemicals, 99.5% pure) and demineralized water. Ammonium salt solution was prepared using ammonium sulfate (analytical grade, Fisher Scientific, 99.5% pure) and demineralized water.

2.1. Ion exchange resins and ion exchange membranes

The AMP-PAN ion exchange resin (M/s. Thermax Ltd, Pune, India) and cation exchange membranes (Membranes International, USA) are the same as those used in our earlier study [28].

2.2. Experimental procedure to determine current–voltage curves

A schematic of the experimental set up is shown in Fig. 1. The EDIX cell consists of three compartments. The middle compartment of dimensions 10 cm × 10 cm × 0.5 cm, is separated from the anode and the cathode compartments by two cation exchange membranes (CEM). Half the volume of the middle compartment is filled with AMP-PAN loaded with Cs^+ , thus resulting in effective membrane area of 50 cm². Twenty grams of resin containing 640 mg of Cs was placed in this compartment. Platinum coated titanium electrodes of 10 cm × 10 cm are placed in the outer compartments. One liter of ammonium sulfate solution of 10,000 ppm was circulated in the anode compartment. In the other middle compartment and cathode compartment, 0.1 N HNO₃ solution of 1 L each was circulated by using peristaltic pumps. When electric potential is applied, NH₄⁺ ions migrate towards cathode through the middle compartment and get exchanged with Cs^+ in the resin present in the middle compartment. Cs^+ thus released is attracted towards cathode and gets concentrated in the cathode compartment.

The electrical current was supplied by a DC power supply connected to the electrode plates. Current–voltage curves were determined by a

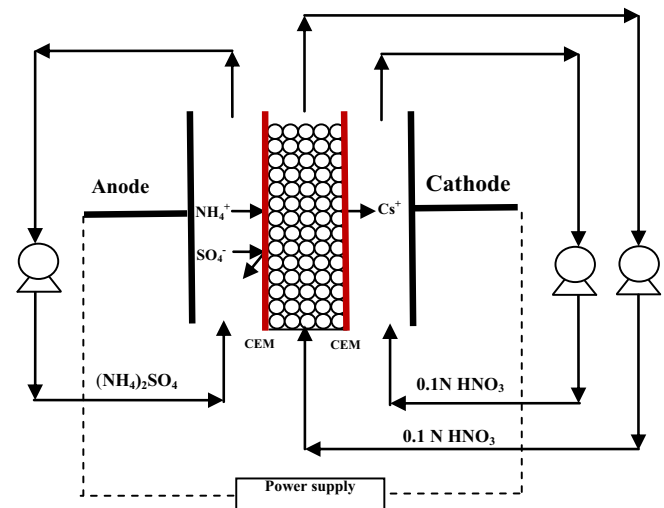


Fig. 1. Schematic diagram of the experimental setup.

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