



Electrical double layer ion transport with cell voltage-pulse potential coupling circuit for separating dilute lead ions from wastewater



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ABSTRACT

Continuous separation of dilute Pb²⁺ from wastewater was demonstrated by using a membrane containing highly conductive multiwalled carbon nanotubes (MWCNTs) in an electrochemically switched ion permselective (ESIP) process, with a cell voltage-pulse potential coupling circuit. The membrane with high selectivity for Pb²⁺ separation was fabricated by pressure filtering MWCNT solution through a polytetrafluoroethylene (PTFE) film. In the separation system, the directional uptake and release of Pb²⁺ were realized by modulating the positive and negative charge densities on the electrical double layer (EDL) of the membrane, together with the application of an external electric field. The effects of the operating parameters on the flux of Pb²⁺ and membrane permselectivity were investigated. The highest Pb²⁺ flux across the membrane occurred with considerable permselectivity at a cell voltage of 0.8 V, a pulse width of 40 s, and a membrane MWCNT content of 30 mg. The system reduced the Pb²⁺ concentration in the solution from 30 to 0.36 ppm with a current efficiency of 46.1%. Such a novel EDL membrane-based ESIP system could be used for wastewater treatment.

1. Introduction

Toxic heavy metal ions have created serious pollution problems worldwide that endanger the environment and human health [1,2]. Heavy metal ions could accumulate in the human body via water or food intake, and damage the central nervous system, kidney, liver, and reproductive system [3]. Lead ions (Pb²⁺) are known to cause serious health problems such as anemia, kidney disease, and mental retardation [4]. Therefore, the removal of Pb²⁺ from wastewater is of great importance. Techniques that have been developed for this purpose include: chemical precipitation [5], ion exchange [6], adsorption [7], membrane filtration [8], and electrochemical treatments [9]. Ion exchange and adsorption are considered the two best physicochemical methods. However, they also create large amounts of secondary waste during resin regeneration.

The electrochemically switched ion exchange (ESIX) method [10–13] is an environmentally benign technique for separating target ions. It has attracted great attention in recent years since it can eliminate the secondary wastes. Using electricity as the main driving force, the ESIX process is capable of rapid uptake and release of target ions even at very low concentrations. Weidlich and co-workers devel-

oped an ESIX system by reversing the polarity of the electrodes periodically and simultaneously exchanging the fluids in the two compartments, in order to realize a “semicontinuous operation process” [14–16]. Wallace and Bobacka developed a series of metal ion transport systems based on conducting polymer membranes for ion separation and recovery [17–20]. In order to realize continuous ESIX separation of Ca²⁺ and Mg²⁺ for water softening, our group had designed an in situ potential-enhanced, electrochemically switched ion permselective (ESIP) system with an iron hexacyanoferrate (FeHCF)–polypyrrole/polystyrenesulfonate (PPy/PSS) membrane [21]. However, the membrane was easily oxidized at high potential, resulting in high transfer resistance and low current efficiency. Additionally, this system had no selectivity towards heavy metal ions, for which novel ESIP membranes are required.

For industrial applications, the ESIP membranes should have high electrical conductivity and cyclic stability. Among traditional ESIX films [22,23], the inorganic ones [22] generally possess excellent thermal and mechanical stability but poor conductivity. In contrast, organic conducting polymers are usually limited by their poor cyclic stability due to (i) volume change from swelling, shrinkage, cracks, or breakage of the membrane, (ii) active material loss, and (iii) over-

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oxidative degradation in the doping/de-doping process [23]. In this work, three-dimensional (3D) porous membrane of multiwalled carbon nanotubes (MWCNTs) with superior chemical and mechanical stability, high corrosion resistance, and high anti-oxidative capacity [24–26] are applied for the first time to remove heavy metal ions in an ESIP system. Since the charge-discharge rate of the electrical double layer (EDL) in this material is very high and highly reversible, fast and high-throughput ion enrichment and depletion can be achieved. These membranes are expected to be a great breakthrough in membrane-based permselective ion separation technology. Moreover, as a pseudo one-dimensional material, the MWCNTs [27–29] can be easily processed (e.g. by the pressure filtering technique [30]) on a large scale into membranes that have large surface area, high electrical conductivity, enhanced ion transfer channels, high cycle efficiency, and long-term stability. Wang and co-workers [31] found that acidified MWCNTs had high selectivity for adsorbing Pb^{2+} , which can easily complex with the oxygenous functional groups (-COOH, -OH) on the MWCNTs. However, to the best of our knowledge, no MWCNT membrane has been reported for Pb^{2+} separation using ESIP systems.

In this study, the MWCNT membrane was fabricated on polytetrafluoroethylene (PTFE) by pressure filtering [30], and used for removing Pb^{2+} from wastewater with or without interfering ions. As shown in Fig. 1, the ESIP system consisted of two two-electrode subsystems, in which a pulse potential provided by electrochemical workstation was applied on the ESIP membrane, and a cell voltage provided by DC power supply was applied on the stainless steel plate electrodes to form a constant electric field. Here, the MWCNT/PTFE membrane was mounted in the middle of the cell vertically, where MWCNT and PTFE membranes contacted the receiver and source solutions, respectively. Two pumps were used to pump the source and receiver solutions from two big tanks to compartments of the cell and circulate the solutions, respectively. In this case, when a negative potential was applied to the membrane electrode (Fig. 1A), cations in the source solution (left compartment) should migrate to the EDL region at the electrode

surface. Once the potential was switched to the positive (Fig. 1B), the incorporated ions could quickly release into the receiver solution (right compartment) in the presence of a constant driving electric field. Here, the selectivity of MWCNT/PTFE membrane should be enhanced owing to the oxygenous functional groups on the surface of MWCNTs, which not only provided active sites for target ion due to electrostatic attraction and complexation, but also formed ion channels for the fast transport of target ion. The system was tested for the selective separation of Pb^{2+} from a mixed nitrate solution containing Pb^{2+} , Ni^{2+} , Cd^{2+} , Co^{2+} , and Zn^{2+} , with the help of a novel cell voltage-pulse potential (CV-PP) coupling circuit that facilitated the transport of target ions across the membrane and improves the current efficiency. The synergistic effects of cell voltage and pulse potential were investigated in details, and the optimal operating parameters (i.e., pulse width, MWCNT content, cell voltage, and volume flow rate) were determined.

2. Experimental

2.1. Chemical reagents

All reagents used were of analytical grade, and deionized (DI) water (18.25 M Ω cm) was used to prepare the solutions. MWCNTs (diameter: 10–20 nm, length: 10–50 μm) were purchased from Beijing Science and Technology Co., Ltd., China. Polyvinyl alcohol (PVA, $M_w = 100\,000$) was obtained from Fischer Scientific, China. Hydrochloric acid (36–38%) and succinic acid (99.5%) were bought from Tianjin Guangfu Fine Chemical Research Institute, China. PTFE membranes (pore size: 2 μm) were purchased from Sartorius Inc, China. Lead, nickel, cadmium, zinc, and cobalt nitrates ($\geq 99\%$) were employed for the ion transport experiments.

2.2. Characterization and electrochemical test

The surface and cross section of the composite membrane were

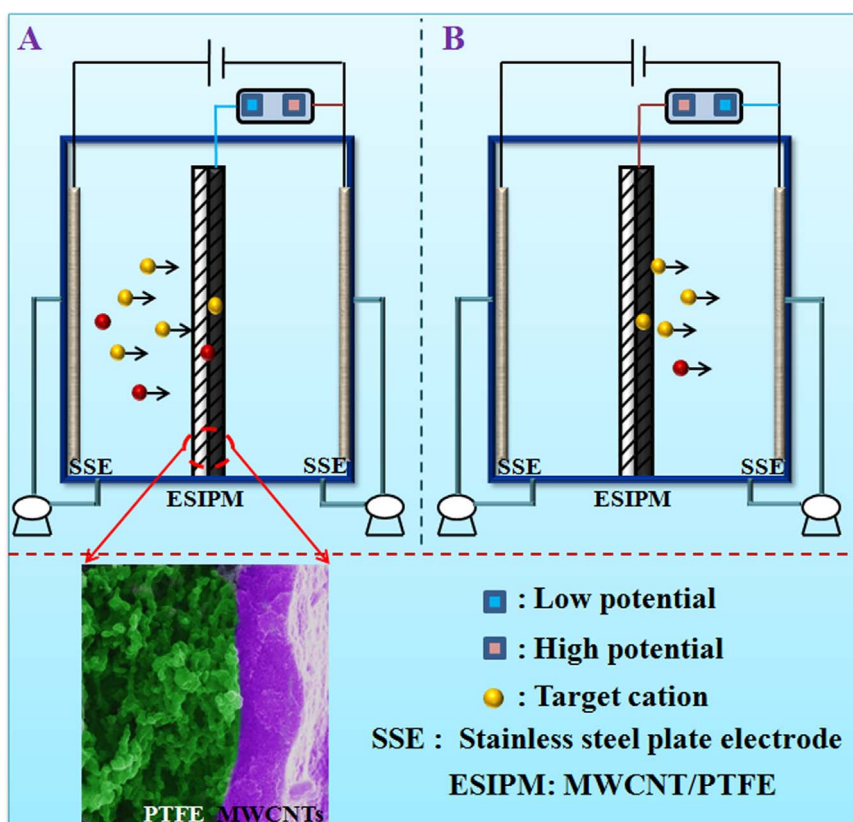


Fig. 1. Schematic illustration of the continuous separation of Pb^{2+} across the MWCNT/PTFE system, ESIPM: ESIP membrane.

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