



Recovery of V(V) from complex vanadium solution using capacitive deionization (CDI) with resin/carbon composite electrode

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HIGHLIGHTS

- A novel resin-carbon composite electrode was fabricated for CDI treatment.
- The composite electrode presents high selectivity for V(V).
- The adsorption mechanism of ions on the composite electrode was illuminated.
- V(V) can be separated and enriched by CDI with the composite electrode.

ARTICLE INFO

Article history:

Received 17 April 2018

Received in revised form

23 May 2018

Accepted 24 May 2018

Available online 25 May 2018

Handling Editor: E. Brillas

Keywords:

Capacitive deionization

Selectivity

Vanadium

Recovery

Composite electrode

ABSTRACT

The resin-activated carbon composite (RAC) electrodes were fabricated and applied in capacitive deionization for recovery of V(V) from complex vanadium solution. The adsorption capacity of the RAC electrode for V(V) is extremely low and the reduction of V(V) is significant in low pH solution, but the adsorbed V(V) on the electrode increases obviously and the reduction of V(V) gradually diminishes with the rise of pH. However, as the pH is increased to 10, the adsorbed V(V) on the RAC electrode declines. The higher applied potential is beneficial to the adsorption of V(V) and 1.0 V is appropriate for the adsorption. The impurities ions (Al, P and Si) are mainly adsorbed in the electric double layers on the RAC electrode and V(V) is dominantly adsorbed by the resins in the electrode. The adsorbed impurity ions can be easily removed by diluted H₂SO₄ and V(V) can be effectively eluted by 10% NaOH solution. The vanadium-bearing eluent can be recycled to recover and enrich vanadium from the complex solution. The performance of the RAC electrode keeps stable during the cyclic operation. This study may provide a promising and novel method for the recovery and separation of metals from aqueous solution.

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1. Introduction

Capacitive deionization (CDI) is an emerging charge-based desalination technology where ions are separated under electrical field (Al Marzooqi et al., 2014). When an electrical potential is applied to the electrodes in CDI, charged ions migrate to the electrodes and are held in the electric double layers (EDLs) on the surface of the electrodes. Once the potential is removed, the

adsorbed ions are quickly released back to the bulk solution (Kim and Choi, 2010). The CDI process commonly is conducted at a relatively low electric potential (typically 0.8–2.0 V), and it also does not produce contaminants during the treatment (Mossad and Zou, 2013; Porada et al., 2016). Hence, CDI technology has received a great deal of attention in the fields of separation and recovery of ions from aqueous solutions as an economical and environment friendly technique.

The separation and removal of ions are achieved by adsorption of the ions in the EDLs on the electrode surface, which is highly dependent on the physicochemical properties of the electrode (Jia and Zhang, 2016). Thus, many studies have been conducted to screen or fabricate the electrode materials with outstanding characteristics, for example, excellent electrochemical properties,

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suitable pore structure and high specific surface area (S_{BET}), to improve the performance of CDI. Carbon materials, such as activated carbon (AC) (Jande and Kim, 2013), carbon nanotube (CNT) (Liu et al., 2015; Wang et al., 2011), graphene (Li et al., 2010a) and carbon aerogel (CA) (Gabelich et al., 2002), due to their superior electrical conductivity and large S_{BET} , are commonly used as electrode materials in CDI.

The CDI process generally presents no or weak adsorption selectivity for ions because the ions are adsorbed by Coulomb force, resulting in the fact that CDI is only widely used to desalinate seawater or brackish water and is rarely applied in the fields needing selective removal of ions (Li et al., 2016), such as chemical engineering and environment. However, some researchers found that CDI can present selective adsorption capability for certain ions by using the composite or surface-modified materials as electrodes. Liu et al. (2013) used CDI technology to selectively adsorb Ca^{2+} from the solution containing Ca^{2+} , Mg^{2+} and Na^{+} by means of CNTs/Ca-selective zeolite composite electrodes. Xu et al. (2008) manufactured the composite carbon aerogel using a resorcinol/formaldehyde polymerization and pyrolyzation process and applied it in CDI to recover iodide from brackish water. Kim et al. (2017) synthesized sodium manganese oxide ($\text{Na}_{0.44}\text{MnO}_2$) via the solid-state reaction of Na_2CO_3 and Mn_2O_3 as the electrode in CDI and the CDI exhibited extremely high selectivity for Na^{+} than for K^{+} , Mg^{2+} and Ca^{2+} in the electrolyte. Lee et al. (2017) fabricated a spinel type of LiMn_2O_4 as the lithium-selective electrode by using Li_2CO_3 and MnCO_3 as reactants. Their studies verified the feasibility of the proposed concept that lithium can be recovered from aqueous solution containing lithium by using the CDI with LiMn_2O_4 electrode. Although these studies indicated the feasibility of selective adsorption of ions by CDI, there are very little reports on the separation and recovery of metals from the complex solution by CDI.

Vanadium is an important versatile rare element, which is dispersedly distributed in the earth's crust. Separation and recovery of vanadium from the complex vanadium leaching solution is a necessary procedure for production of vanadium (He et al., 2007; Li et al., 2010b; Zhang et al., 2011). Moreover, the removal of vanadium from the wastewater in vanadium industry is also a problem which is widely concerned in environment (Kaczala et al., 2009). At present, the widely adopted methods for the recovery of vanadium from aqueous phases are solvent extraction and ion exchange. However, solvent extraction may cause potential threat to environment and ion exchange process usually needs high-pressure pumps and long operation time (Liang et al., 2016; Tang et al., 2017). CDI is hopeful to overcome these shortcomings and provide a novel and potential method for the separation and recovery of metals from complex solution. Our previous study demonstrated that the ion exchange resin-activated carbon composite electrode (RAC electrode) in CDI presents high affinity for vanadium ions (Duan et al., 2018). In this study, a novel RAC electrode was fabricated and applied in CDI for the separation and recovery of pentavalent vanadium (V(V)) from complex vanadium-bearing solution. This study may prove the feasibility of selective adsorption by CDI and give a promising alternative for the separation and removal of metals from complex solutions.

2. Materials and methods

2.1. Materials

D201 anion exchange resin (mean diameter 0.15 mm) with high affinity for V(V) [Zhu et al., 2017], provided by Zhejiang Zhengguang Industrial Co. Ltd, and the AC with S_{BET} of $1027\text{ m}^2/\text{g}$ and mean diameter of 0.038 mm (Huang et al., 2014), purchased from Provedor de Laboratorios Co. Ltd., were used to prepare the RAC

electrode in this study. The high-purity graphite flake with the dimensions of 100 mm (length) \times 50 mm (width) \times 1 mm (thickness) was supplied by Haimen Shuguang Carbon Industry Co. Ltd. *N*-dimethylacetamide (DMAC) with A.R. grade and Polyvinylidene fluoride (PVDF) with A.R. grade came from Sinopharm Chemical Reagent and Sigma Aldrich Co. Ltd., respectively.

The simulated vanadium-bearing solution containing 1000 mg L^{-1} V(V), 3000 mg/L Al, 230 mg L^{-1} P and 35 mg L^{-1} Si, referring to the common leaching solution in vanadium industry (Li et al., 2010a,b; Chen et al., 2010), was prepared by dissolving sodium metavanadate ($\text{NaVO}_3 \cdot x\text{H}_2\text{O}$), aluminum sulfate hexadecahydrate ($\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$), sodium phosphate ($\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$) and sodium silicate ($\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$) in deionized water (Millipore Milli-Q[®]). The pure vanadium solution containing 1000 mg L^{-1} V(V) was prepared by only dissolving sodium metavanadate in deionized water. The pH of solution was adjusted by sulfuric acid with A.R. grade in the experiments. Sodium metavanadate, ordered from Alfa Aesar (Tianjin) Chemical Co. Ltd. Aluminum sulfate hexadecahydrate, sodium phosphate and sodium silicate were obtained from Sinopharm Chemical Reagent Co. Ltd. All of these reagents are of C.P. grade.

2.2. Fabrication of electrodes

The fabrication process of the RAC electrode is same to that described in our previous study (Duan et al., 2018). Briefly, D201 resins were firstly treated by soaking in 5% (v/v) HCl and 5% (m/v) NaOH solutions alternately to remove the remained monomers and other types of impurities which may be produced in the fabrication process, followed by washing with deionized water to neutral, and then were filtered and dried at 60°C in vacuum oven for 12 h before use. Subsequently, 2 g AC powder, 2 g pretreated resin and 0.4 g PVDF were mixed in 12 mL DMAC for 4 h to prepare the resin-AC composite material slurry, and then the slurry was uniformly smeared on the collector electrode, a high-purity graphite flake. Finally, the flake smeared with the slurry was put in vacuum oven at 65°C for at least 4 h to form the RAC electrode. PVDF was used in the fabrication process to bind the composite material with graphite flake and endow the electrode with appropriate mechanical stability. The preparation procedure for the AC electrode used in this study is same to that for the RAC electrode just no resin is added.

2.3. CDI adsorption

The CDI equipment is made up of CDI cell, constant voltage direct-current (DC) power supply (IT6861A, ITECH Co. Ltd, China), peristaltic pump, tubes and beaker (Fig. SM-1). The CDI cell is assembled by three pairs of parallel electrodes which are accommodated in a polymethyl methacrylate tank. The space of each pair of electrodes is 3 mm, which can allow solution to flow freely and maintain suitable electric field intensity. Each pair of parallel electrodes is supplied a same constant voltage in the experiments. The RAC or AC electrodes were used as anode and the high-purity graphite flakes without the resin-AC composite material were used as cathode in CDI treatment. 200 mL feed solution was pumped into the CDI cell from a beaker through the inlet by peristaltic pump, and the effluent flowed into the beaker through the outlet for circular treatment.

In the CDI adsorption experiments, the processing time was 120 min, and the flow rate was kept at 30 mL min^{-1} . The electroadsorption capacity of electrode was calculated by Eq. (1).

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