



Full Length Article

Combined electrosorption and chemisorption of low concentration Pb(II) from aqueous solutions with molybdenum disulfide as electrode

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ABSTRACT

In order to develop a more effective method for removal of heavy metals with low concentration and explore the feasibility of using MoS₂ as a super electrode material, removal of Pb(II) at low concentration was studied with combined electrosorption and chemisorption (CEC) by using molybdenum disulfide electrode. The experimental results indicated that Pb(II) could be highly removed by using CEC method when the initial concentration of Pb(II) was less than 6 mg/L. MoS₂ electrode achieved 86.26 mg/m² uptake capacity of Pb(II) with CEC, which was almost five times of that with pure chemisorption. The great improvement was attributed to the cooperation of electrosorption and chemisorption. The electric field helped to attract positively charged Pb(II) to the surface of the cathode and thus concentrated Pb(II) in the electric double layer (EDL) of MoS₂ electrode, while the intrinsic S atoms on the surface of MoS₂ immobilized Pb(II) on the electrode through the formation of Pb-S complex. It is suggested that CEC might be a promising process to immobilize heavy metals with low concentration from aqueous solutions.

1. Introduction

Lead exists as one of the toxic and carcinogenic heavy metal pollutants which arouses great attention owing to its harmful influences on environment and human health [1]. Lead originates from various industrial activities including metal plating, mining, fertilizers and pesticides utilization [2]. Exposure to lead related pollutants or long-term drinking water containing lead would cause nervous system, immune system, liver, kidney disorders as well as heart disease [3]. Several remediation technologies, such as chemical precipitation, ion exchange, adsorption and membrane process have been developed to remove lead from wastewater [4–7]. Among these methods, adsorption is considered to be a promising method in terms of its simple operation, low cost and high efficiency. However, the removal capacity of conventional adsorption process for wastewater with low concentration of lead ions is relatively low. As a consequence, there is a great interest in the development of economically attractive technologies for removing Pb(II) with low content. In order to overcome this problem, it is necessary to search some efficient methods for the enhancement of low concentrated Pb(II) water treatment. It is known that the Pb(II) uptake capacity in adsorption process is positively related to the concentration of Pb(II)

around the adsorbent, therefore accumulation of Pb(II) near the adsorbent might be an effective way to improve its adsorption capacity. Electrosorption is a simple and feasible method to adsorb counter-ions approaching the electrode through external electric field, which results in a great increase of the ions concentration in the EDL formed at the electrode surface [8–10]. Thus, the pre-concentrated ions by electrosorption would highly promote the further adsorption if the adsorbent is attached on the electrode.

Conventional carbon-based electrode materials, such as graphitized-carbon, carbon nanotubes (CNTs), graphene oxide and graphene aerogels, have been prepared as electrodes for the removal of Pb(II) from aqueous solutions due to their large surface area, good conductivity, rich adsorption sites and chemical stability [11–14]. Compared with those carbon materials, MoS₂, which is naturally occurred as molybdenite, features intrinsic sulfur-rich surface. Besides, its high specific surface area, excellent electronic conductivity, good chemical and electrochemical stability also make it an ideal candidate as an electrode material for electrosorption [15]. Recently, several studies have researched the desalination by using MoS₂ composites as electrodes. For example, the ce-MoS₂ nanosheets achieved high removal capacity of 8.81 mg/g at 1.2 V voltage in 400 mmol/L NaCl solution and exhibited

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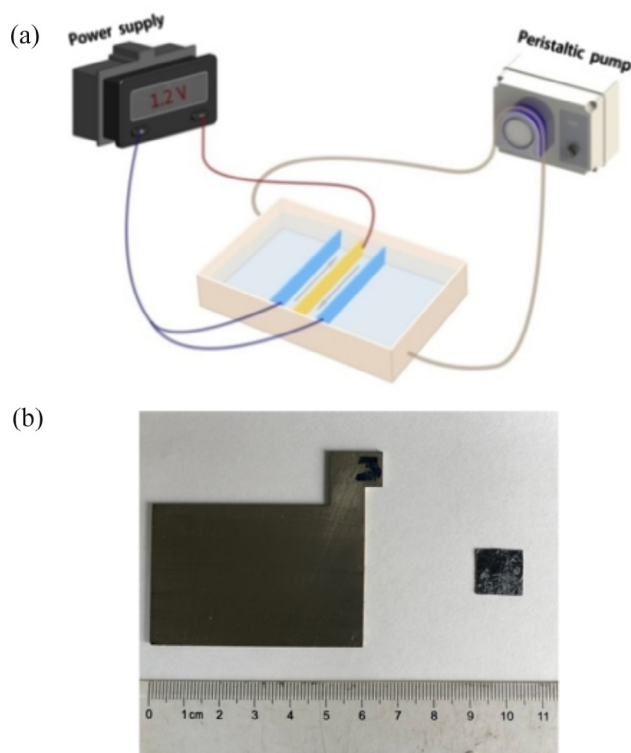


Fig. 1. (a) Schematic representation of the self-made electrosorption device and (b) image of titanium plate and square molybdenite flake.

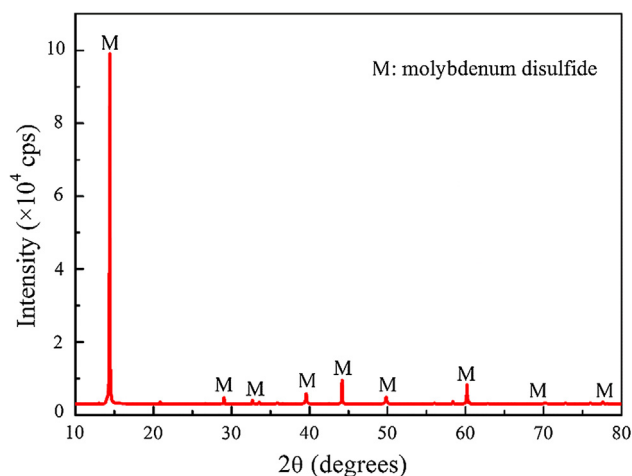


Fig. 2. XRD pattern of natural molybdenite.

better removal capacity than carbon materials even at a low concentration of 50 mmol/L salt [16]. The electrosorption capacities on MoS₂/CNT were 10, 13, 18, and 25 mg/g in 5, 25, 100, and 500 mmol/L NaCl aqueous solutions, respectively [17]. Besides, many studies have verified that the abundant sulfur groups exposed on the surface of MoS₂ led to a chemical complexation of heavy metal atoms and S atoms [18–21]. For example, flower-like MoS₂ nanosheets had a super Hg²⁺ removal capacity of 2563 mg/g [22]. The two-dimension MoS₂ had a remarkable efficiency for Pb(II) removal from water with a 1479 mg/g adsorption capacity [23]. However, their excellent adsorption performance achieved in heavy metal solutions with high initial concentrations ranged from 30 mg/L to 2000 mg/L. If decreasing the initial concentration of heavy metals to less than 10 mg/L, the adsorption capacity of MoS₂ must have a dramatic descent. Considering the good electronic conductivity of MoS₂, preparation of MoS₂ as electrode for

removing low concentration heavy metals from aqueous solutions might be a good idea. In this case, heavy metals could be firstly concentrated in the EDL of the electrode and then be adsorbed by MoS₂. To the best of our knowledge, no researches have concerned on the electrosorption or CEC of heavy metals with low concentration from aqueous solutions using MoS₂ electrode.

In this study, Pb(II) was selected as a representative heavy metal to explore the adsorption behavior of heavy metals on MoS₂ electrode. The characterization of MoS₂ electrode was investigated by X-ray diffractometer (XRD), inductivity coupled plasma optical emission spectroscopy (ICP-OES), field-emission scanning electron microscope (FESEM) and electrochemical work station. Furthermore, the adsorption mechanism was revealed through Raman spectroscopy and X-ray photoelectron spectroscopy (XPS) measurements. The objective was to explore the feasibility of CEC by using MoS₂ as an electrode material for the removal of heavy metals with low concentration from aqueous solutions.

2. Materials and methods

2.1. Materials

Natural molybdenite collected from Wuzhou Mine, Guangxi Province, China, was used in this study for the preparation of molybdenum disulfide electrode. Titanium plates with length, width and thickness of 60, 45 and 1 mm were purchased from Yongsheng Company, China. Conductive silver adhesive was obtained from Shanghai Bao Yin Electronic Materials Ltd, China. All chemical reagents used in the experiments were of analytical grade without further purification and purchased from Sinopharm Chemical Reagent Company. Pb(II) stock solution was prepared by dissolving Pb(NO₃)₂ in deionized water. All the water used in this work was produced from the Millipore Milli-Q Direct 8/16 water purification system.

2.2. Fabrication of molybdenite electrode

The square MoS₂ flakes were cut from natural molybdenite, followed by cleaving the flakes with a scotch tape to expose fresh surfaces. Each flake had side length of 12 mm and thickness of around 0.5 mm. Molybdenite electrode was then prepared by pasting 10 pieces of MoS₂ flakes on both side of the titanium plate with the help of conductive silver adhesive. Finally the electrode was dried at 60 °C for 2 h in a drying oven.

2.3. Adsorption of Pb(II) and recycle of electrode

The adsorption experiments of Pb(II) were performed in the self-made electrosorption device, which was schematically represented in Fig. 1. The system consisted of an electric adsorption cell which was made of polymethyl methacrylate, an external power supply and a peristaltic pump (BT600-2J). Three electrodes were assembled in the cell with a gap distance of 2 mm. The cathode in the middle was coated with MoS₂ flakes on both sides, being placed by two anodes facing to it. 200 mL of Pb(NO₃)₂ solution with the initial concentration of 0.3 mg/L to 6.0 mg/L was continuously recycled in the cell at the flow rate of 46 mL/min under room temperature. The pH value was adjusted to 5.0 ± 0.05 with 0.1 mol/L NaOH and 0.1 mol/L HNO₃ in order to avoid the precipitation of Pb(OH)₂. The applied voltage was 1.2 V. And CEC was conducted for 180 min which was the same as that in an open circuit. When the adsorption equilibrium was reached, the electrode was then put into 200 mL 1 mol/L HNO₃ solution for 3 h to perform the desorption tests. The above adsorption and desorption tests was repeated three times to investigate the recycling and reusing performance of the electrode. The adsorption capacity (*q*), defined as the quantity of Pb(II) adsorbed by per unit surface area of molybdenite electrode on the cathode, was calculated as follows.

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