

Removal of Pb^{2+} from aqueous solutions by a high-efficiency resinHao Guo^a, Yongzheng Ren^{a,*}, Xueliang Sun^a, Yadi Xu^a, Xuemei Li^a, Tiancheng Zhang^b, Jianxiong Kang^a, Dongqi Liu^a^a School of Environmental Science and Engineering, Huazhong University of Science and Technology, Wuhan 430074, PR China^b Civil Engineering Department, University of Nebraska-Lincoln at Omaha Campus, Omaha, NE 68182-0178, USA

ARTICLE INFO

Article history:

Received 7 December 2012

Received in revised form 11 April 2013

Accepted 28 June 2013

Available online 8 July 2013

Keywords:

Ion-exchange

 Pb^{2+}

Kinetic

Equilibrium

Wastewater

ABSTRACT

The removal of Pb^{2+} from aqueous solution by 732 cation-exchange resin in sodium type (732-CR) has been studied in batch experiments at varying pH (2.0–8.0), Pb^{2+} concentration (50–200 mg/L), contact time (5–300 min), temperature (288–308 K) and resin dose (0.125–0.75 g/L). The experimental data show that the ion-exchange process was dependent on pH and temperature, the optimal exchange capacity was found at pH 4.0, and higher temperature was beneficial to lead sorption. Kinetic data indicate that the ion-exchange process followed a pseudo-first order model. The equilibrium exchange capacity could be reached at approximately 4 h, and the maximum sorption capacity of Pb^{2+} at pH 4.0 was 396.8 mg/g resin. The equilibrium data were evaluated with Langmuir and Freundlich model, and were best fitted with Langmuir model. The thermodynamic parameters for removal of Pb^{2+} indicate that the reaction was spontaneous and endothermic. Additionally, column tests were conducted by using both synthetic solution and effluents from lead battery industry. The regeneration of resin was performed for two sorption-regeneration cycles by 1 M NaOH, and the results show that effective regeneration was achieved by this method.

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1. Introduction

The presence of heavy metals in the aquatic system is harmful to humans, plants and animals [1–3]. As one of the most commonly-used heavy metals, lead roots in many industries, such as metal plating, mining and acid battery [4]. Unlike organic pollutants, which are prone to biological degradation, lead is non-biodegradable and accumulates along the food chain in living organisms, causing various damage to the kidney, nervous system, muscle, cardiovascular and other vital organs [5]. Due to its high toxicity [6] and the wide applications, innovative and efficient methods for lead removal from the aqueous solution are continuously being developed, such as chemical precipitation, adsorption, ion-exchange, membrane filtration, reverse osmosis, and electrochemical treatment [7]. Among these techniques, ion-exchange is considered to be a potential method with several advantages associated with high removal efficiency, low cost and better renewability.

Apparently, ion exchanger is crucial for ion-exchange technique. Zeolite [8], sulfonated coal and resin have been used as ion exchangers in theoretical experiments and practice. Among these

ion exchangers, resin is the most common. A lot of resins have been used to remove heavy metals from aqueous solutions such as chelating resins [9,10], cation-exchange resin [11] and basic anion resin [12]. Recently, with the rapid development of material technology, many commercial resins such as Lewatit SP-112 [13], Diaion CR11, Amberlite IRC 86 [14], Amberlite IRC 748 [15], D401 [16] have been used to reduce heavy metals from aqueous solution due to their strong metal exchange capacity. Among these commercial resins, cation-exchange resins containing sulfonic group as active sites such as Amberlite IRC 748 [17] and Davankow resins [18] have been successfully applied for the recovery of heavy metals. 732-CR is a gel-type polystyrene, sulphonated strong cation-exchange resin, and it has same structure and components with Amberlite IR 120. In previous studies, it was indicated that Amberlite IR 120 may be used for the removal of heavy metals (Cu^{2+} , Zn^{2+} , Ni^{2+} , Cd^{2+} , Pb^{2+}) at certain conditions in H^+ form [19–21]. It is durable, insoluble and cost-efficient, and has an excellent ability to remove many heavy metals. However, information is insufficient on the fundamentals of using 732-CR in Na^+ form to remove lead from aqueous solution.

The objectives of this study were to: (1) evaluate the performance of 732-CR resin for Pb^{2+} sorption under different conditions; (2) investigate kinetics, the ion-exchange characteristics of 732-CR resin for Pb^{2+} removal and the related mechanisms; and (3) perform column studies to evaluate performance of 732-CR resin for lead removal under dynamic flow conditions.

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Table 1
Characteristics of 732-RC.

Characteristics	Value
Functional group	Sulfonic acid
Physical form	Opaque, claybank beads
Ionic forms as shipped	Na ⁺
Total exchange capacity	≥168 mg/g (Na ⁺ form) ^a
Particle size	0.3–1.2 mm
True density in wet state	1.23–1.28 g/mL
Bulk density in wet state	0.75–0.85 g/mL
pH range	1–14 (depending on application)
Water content	46.0–52.0%
Surface area	1.066 m ² /g

^a Using adequate NaOH solution to treat raw resin in H⁺ type, then HCl was used to titrate redundant NaOH. The total exchange capacity was calculated by the equation $(C_{\text{NaOH}} \times V_{\text{NaOH}} - C_{\text{HCl}} \times V_{\text{HCl}})/m$, where m is dry weight of resin.

2. Materials and methods

2.1. Materials in experiments

Commercial synthetic resin 732-CR in sodium form used in the experiments was supplied by Sinopharm Chemical Reagent Co. Ltd (Shanghai, China). The physicochemical properties are shown in Table 1. The resin was washed with deionized water to remove impurities and dried at 40 °C to constant weight. The dried resin was stored in a glass bottle for further experimental studies.

All the reagents used in the experiments are analytical grade. Pb(NO₃)₂ was purchased from Sinopharm Chemical Reagent Co. Ltd. The standard stock solution of 5000 mg/L Pb²⁺ was prepared by dissolving 7.992 g Pb(NO₃)₂ in 1 L of deionized water. The other lower concentrations were obtained by diluting the stock solution freshly for each experiment. Moreover, the acid battery wastewater containing Pb²⁺ (Table 2) was kindly provided by Wuhan Intepower Co. Ltd, China, and used in column experiments.

2.2. Analytical methods

A WFX-110 Model Atomic Absorption Spectrophotometer (AAS) (Beijing Beifen-Ruili Analytical Instrument (Group) Co. Ltd., Beijing, China) operating with an air-acetylene flame was used to analyze the concentration of Pb²⁺ in solutions. The pH measurements were performed with LIDA pH meter (LIDA Instrument, Shanghai, China). BS-IEA model thermo stated shaker (Jierui Electrical Co. Ltd., Changzhou, China) was used for the batch experiments.

2.3. Batch ion-exchange experiments

The influence of the resin dosage for ion-exchange experiments was performed in the polypropylene centrifuge tube. A fixed mass of 0.01–0.06 g resin was added to each tube, and 80 mL of 300 mg/L Pb²⁺ solution was transferred to the tube at pH 4.0. The solutions were agitated in the shaker at 25 ± 1 °C for 24 h, which is adequate to achieve equilibrium [22–24]. Then the resin was separated and the liquid was analyzed by the AAS to determine the residual Pb²⁺ concentration in the liquid phase.

The effect of contacting time and initial concentration on the removal of Pb²⁺ ions were studied in the range of 5–300 min and 40–320 mg Pb²⁺/L at pH 4.0, respectively. In these experiments, 0.05 g resin was mixed with 80 mL target solution and agitated at

25 ± 1 °C. The experiments for the removal of Cu²⁺ and Ca²⁺ were conducted at the initial concentration of 100 mg/L, contacting time 5–300 min, resin dosage 0.04 g, temperature 25 ± 1 °C and pH 4.0. The experiments about the pH effect were performed by varying pH from 2.0 to 8.0. HNO₃ (2.5 M) and NaOH (2.5 M) were used to adjust the initial pH. In addition, the isotherm experiments were studied at different temperatures (288 K–308 K). All the experiments were conducted in duplicate.

2.4. Column experiments

For the column experiments, ion-exchange of Pb²⁺ in a continuous flow system was carried out in a glass column (internal diameter = 1.5 cm and height = 20 cm), packed with approximately 2.0 g resin. The glass wool was added to the top of the column for the flow distribution. The initial Pb²⁺ concentration of the synthetic solution and industrial wastewater was 50 mg/L and 44.14 mg/L, respectively. The solutions were percolated through the column at a constant flow rate of 10 mL/min by using a peristaltic pump (Longer Precision Pump Co. Ltd., Baoding, China) in the down-flow mode. The effluent solution was collected at different time and analyzed as described above.

Regeneration of the resin was performed by passing 1 mol/L NaOH through the column at a flow rate of 10 mL/min for 1 h. After the regeneration, the column was rinsed with deionized water until the eluting water achieved neutral. The regeneration resin was reused for the next cycle.

2.5. Characterization of resin surface functional groups

For the characterization of 732-CR, Fourier transform infrared spectrometer (FTIR-ATR, VERTEX 70, Bruker Corporation) was used to measure the surface functional groups on the resins before and after ion-exchange experiments. The samples were placed on the scanning spot directly and scanned in a frequency range of 550–4000 cm^{−1}.

3. Results and discussion

3.1. Effects of resin dosage and pH on ion-exchange tests

Fig. 1(a) shows the relationship between resin dosage and ion-exchange capacity. The maximum ion-exchange capacity was observed for the resin dosage of 0.5 g/L. It is apparent that the ion-exchange capacity of lead increased with increasing resin amount from 0.125 to 0.5 g/L. It can be due to the increase in resin exchange sites available for the sorption of lead ions [25]. Then there is a slight decline with an increase in resin amount from 0.5 to 0.75 g/L. This trend can be attributed to the formation of clusters of exchanger particles resulting in decreased effective surface area [26]. Therefore, 0.5 g/L was considered as the optimum dose and was used in further experiments.

As shown in Fig. 1(b), the exchange capacity enhanced gradually at the pH range of 2.0–4.0. This may be due to the high H⁺ ion concentration, which reverses ionization of Na⁺ on resin surface. There is a sharp increase in exchange capacity at pH of 5.0 to 8.0. It can be described as the precipitation of Pb(OH)₂ [27]. Hence, the remaining experiments were carried out at pH 4.0.

3.2. Effects of time and initial concentration on ion-exchange tests

Kinetic studies were conducted with the varied initial Pb²⁺ concentration from 50 mg/L to 200 mg/L. Fig. 2 shows that the exchange capacity for 732-CR on Pb²⁺ increases with time. For the initial concentration of 50 mg/L, the ion-exchange process reaches

Table 2
Properties of the effluent from lead acid battery industry.

Temperature (°C)	27.5
pH	5.54
COD _{Cr} (mg/L)	54
Pb ²⁺ concentration (mg/L)	44.14

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