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Recovery of cesium from saline lake brine with potassium cobalt hexacyanoferrate-modified chrome-tanned leather scrap adsorbent

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G R A P H I C A L A B S T R A C T

Chrome-tanned leather scrap is made up of collagen fibers with substantial -COOH, -OH and -CONHR. After hydrolysis with NaOH, the amounts of -OH and -COOH increase, and they exist in the form of sodium salt. Co^{2+} replaced Na⁺ and chelated with -OH, -COOH and -CONHR. Subsequently, K₄Fe(CN)₆·3H₂O reacted with Co^{2+} loaded on AHLS to form KCoFC. During the process of adsorption, Cs⁺ was exchaged with K⁺ located in the body center of KCoFC on AHLS.



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ABSTRACT

Potassium cobalt hexacyanoferrate was modified on alkali-hydrolyzed leather scrap (AHLS/KCoFC) to prepare a novel adsorbent for cesium (Cs⁺) adsorption. AHLS/KCoFC was characterized with SEM, FT-IR, XRD, and XPS to affirm the correct modification. Batch experiments were performed to investigate the adsorption performance of AHLS/KCoFC for Cs⁺ under various conditions, including pH, initial concentration, contact time and temperature. The equilibrium data was fitted well with Langmuir model and the maximum adsorption capacity was 36.75 mg g⁻¹ at pH 6.0, 298.15 K and concentration 100 mg L⁻¹. Thermodynamic and kinetic studies showed that the adsorption process was spontaneous and endothermic, the pseudo-second-order kinetic model fitted accurately the data, and the adsorption process was controlled by intraparticle diffusion, but it was not the only rate-limiting step. Moreover, selective adsorption was investigated systematically. It was found that AHLS/KCoFC could be employed to the recovery of Cs⁺ form saline lake brine, and the adsorption efficiency was achieved 97.33%, suggesting AHLS/KCoFC is potential application advantage as an efficient adsorbent.

1. Introduction

Cesium has been widely applied to electronics, defense industries, fiber optical communication system, organic synthesis catalysts and night-vision equipment due to its unique characteristics, such as high chemical activity and excellent photoelectric property [1–5]. In recent years, cesium has been playing an important role in the development of efficient energy devices. Studies have showed that the thermal efficiency of the magnetic fluid power generation device with cesium compounds as the ionization source is 70%, which is twice as much that

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of the traditional thermal power plant. However, a 600 Mw magnetic fluid power generation device requires annually 3175 t Cs₂CO₃ [6]. Therefore, the demand for cesium is enormous. To the best of our knowledge, the distribution of cesium is not concentrated in nature, and the extraction f cesium from solid ore requires sophisticated techniqueand the purity is not desirable. In addition, cesium resources in oreshave been reduced over time due to unreasonable exploitation. Thus, the research on the extraction of cesium from other resources, such as oilfield, seawater and saline lake brine, is attracting more and more attention [7–9]. In fact, there are many saline lakes in China, and surveys found that the concentration of cesium in the saline lake were about several times or hundreds as much that of any other sources [10]. It is predictable that saline lake brine will become the main source of cesium resources in the future. However, the uptake of cesium from saline lake brine has been facing a big challenge for a long time because cesium coexists with substantial alkali and alkaline earth metal (sodium, potassium, lithium, rubidium, calcium and magnesium) in salt lake brine [11].

Some available methods of cesium extraction from saline lake brine, including solvent extraction [12,13], precipitation [14,15] and adsorption [7,16–20], have been employed. Among them, adsorption is preeminent as an efficient, convenient and cleanseparation method. However, the excellent selectivity of adsorbent is critical. Inorganic exchanger based on potassium metal hexacyanoferrate has been reported to be selective adsorbent for cesium adsorption and has gained an increasing attention of researchers [21]. Potassium metal hexacyanoferrate has a structural formula of K_xM_y[Fe(CN)₆], where M represents divalent transition metal (Fe, Ni, Cu, Zn and Co) Potassium metal hexacyanoferratehas a face-centered cubic structure, the Fe (II) is surrounded by cyanide groups to form octahedra and transition metal ion is located at the corner of the cube, and the exchangeable K⁺ occupies the cube center in order to balance the negative charge of the crystal structure [22]. Numerous studies have shown that the order of adsorption selectivity is Co > Ni > Cu > > Zn > > Fe, and adsorption capacity of potassium metal hexacyanoferrate is in the order of Cu > Ni > Zn > Co > Fe [23–25]. Thus, in the terms of the adsorption selectivity and adsorption capacity, potassium cobalt hexacyanoferrate (KCoFC) was preferentially considered as an adsorbent for cesium adsorption from the complex saline lake environment. However, the major drawback of KCoFC is nano-size, which is not conducive to the separation of adsorbent from the solid-liquid system. Furthermore, its application in fixed-bed columns is limited due to the low permeability, column blockage, and head pressure loss. In view of its undesirable properties, it appeared that KCoFC was immobilized on suitable materials, such as silica, zeolite, polymer and biopolymer [26-30]. Among them, the biopolymer by virtue of its cheapness, less pollution, and regeneration is used as a support of powder adsorbent.

Leather industry has created significant benefits for the national economy in several developing countries, but its enormous solid waste byproduct has produced a serious environmental problem [31]. According to statistics [32], the equivalent of approximately 10% of the quality of the raw hide is discarded in environment as solid waste such as shavings and trimmings, constituting disposal problems and environmental nuisance. In addition, chromium (III) is usually used as tanner agent to produce wet blue leather [33]. It is possible that chromium (III) in leather shavings is oxidized to mutagenic chromium (IV) in a complex natural environment [34,35]. To solve the pollution problem, it is an interesting idea that to use solid wastes from leather industry as an effective adsorbent to remove the hazardous substance from the environment. For instance, Oliveira and Onenc [36,32] used chromium-containing solid waste as adsorbent to remove organic contaminants, Cr (VI) and phenol from aqueous solution. Beyond that, leather, as a natural polymer, is comprise of collagen fibers with threedimensional helical structure, which supplies many functional groups (-OH, -COOH and -CONHR) that can chelate with cobalt ions in the raw material for the synthesis of KCoFC [37]. Thus, it is possible for

leather to act as a support of KCoFC.

In this study, potassium cobalt hexacyanoferrate-modified chrometanned leather scrap adsorbent (AHLS/KCoFC) was prepared by two steps. The adsorbent was characterized by SEM, XRD, XPS and FT-IR. Adsorption properties of the adsorbent for Cs^+ were investigated under various experimental conditions, such as pH, temperature, contact time and initial concentration of Cs^+ . Moreover, in order to discuss the adsorption mechanism, the equilibrium capacity, kinetics, competitive adsorption experiments with co-existing ions were investigated. Remarkably, the adsorbent was used to recover Cs^+ from the brine of Qarham playa to evaluate its potential application.

2. Materials and methods

2.1. Materials and reagents

Chrome-tanned leather scrap was collected from the National Engineering Laboratory for Clean Technology of Leather Manufacture of Sichuan University, China. Cobalt nitrate hexahydrate (Co $(NO_3)_2$ ·6H₂O), potassium ferrocyanide trihydrate (K₄Fe(C)₆·3H₂O), CsCl, NaOH, HCl, KCl and other reagents were purchased from Chengdu Kelong Chemical Reagent Co., Ltd., China. 1 mg mL⁻¹ cesium stock solution was from National Center for Analysis and Testing of Nonferrous Metals and Electronic Materials. All the reagents used for the study were of analytical grade without further purification.

2.2. Prepraration of adsorbent

2.2.1. Preparation of AHLS

Chrome-tanned leather scrap was cut into $0.2 \times 0.2 \text{ cm}^2$ particles. These particles were soaked with deionized water for 12 h and further dried in air at 50 °C, and the granular leather scrap was obtained (GLS). As described by Yan [38], alkali-hydrolyzed leather scrap (AHLS) was prepared by adding 1 g GLS into 150 mL conical flask containing 100 mL of 0.05 mol L⁻¹ NaOH solution to hydrolyze for 4 h at 60 °C. Then, the AHLS was washed with deionized water to neutral and freezedried for 24 h in vacuum.

2.2.2. Preparation of AHLS/KCoFC

The KCoFC was modified on AHLS by two steps. The processes as follow: a weighed amount of $Co(NO_3)_2$ ·6H₂O (0.1–5 g) was completely dissolved in 50 mL deionized water to form a homogeneous solution, and 0.5 g AHLS was added with continuous stirring for 24 h at 25 °C. After that, the prepared mixture was filtered and residue was washed repeatedly with deionized water for removal excess reactant. The sample was freeze-dried for 24 h in vacuum to receive the AHLS modified with Co²⁺ (AHLS-Co).

0.5 g AHLS-Co was suspended in 50 mL solution containing required amounts of K_4 Fe(CN)₆·3H₂O (1–5 g), and the obtained suspension was shaken for 24 h at 25 °C. Whereafter, the suspension was filtrated and washed repeatedly with deionized water to remove excessive K_4 Fe (CN)₆ and adjust pH to neutral. After freeze-drying, the adsorbent, AHLS modified with KCoFC (AHLS/KCoFC), was obtained.

2.3. Characterization

The surface morphology images and elemental of samples were obtained by scanning electron microscopy coupled with energy dispersive X-rayanalysis (SEM-EDX, Ultra 55, Carl Zeiss, Germany). To clarify the structure of samples, the fourier transform infrared spectroscopic analysis (FT-IR, Nicolet-5700, PerkinElmer, USA) was performed with KBr tablet method in frequency range of 400–4000 cm⁻¹. Moreover, X-ray diffraction (XRD) data of samples were collected using a X'Pert-Pro powder X-ray diffractometer with Cu K_{α} radiation. Further, X-ray photoelectron spectroscopy (XPS, ESCALAB250, Thermo Fisher Corportation, USA) was employed to obtain the elements binding

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