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High-efficient recovery of chromium (VI) with lead sulfate

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ABSTRACT

Lead sulfate was chose to precipitate chromium (VI) based on the difference of the solubility between lead sulfate and lead chromate. The effects of parameters on the precipitation efficiency of chromium including reaction temperature, reaction time, and initial pH of solution and dosage of lead sulfate were investigated. Results showed that the initial pH of solution and dosage of lead sulfate had big influence while reaction time and reaction temperature had little. The concentration of chromium (VI) could reduce from 0.2 mol/L to 0.0015 mmol/L (0.08 mg/L) at pH value of 13.90 and the dosage of lead sulfate as $n(PbSO_4)/n(K_2CrO_4)=4$. The XRD result of precipitation was consistent with the result predicted by Visual MINTEQ software and the precipitation was composed of $PbCrO_4$, $PbSO_4$ and other oxides containing lead. Otherwise, XRF and ICP were used to analyze the residual lead (II) in the filtrate and results indicated that the concentration of Pb (II) in the filtrate was acceptable.

1. Introduction

Chromium is an important metal used for metal plating, leather tanning, metal corrosion inhibition, and pigment productions [1–3]. Chromium (VI) discharged from industrial activities including electroplating, petroleum refining, alloy manufacturing and battery production [4–9]. It is known that chromium (VI) species such as dichromate ($Cr_2O_7^{2-}$) and chromate ($HCrO_4^-$, CrO_4^{2-}) could cause a series of problems for humans and animals because they could form non-biodegradable compounds that affect aquatic life and cause serious health issues such as digestive tract cancer, anemia, neurological damage, circulatory shut down and death [10–12]. In China, it has become one of the most commonly detected contaminants in groundwater due to improper storage and disposal practices. Chromium (VI) has been considered as a hazardous contaminant and there is increasing interest in developing effective measures to remove chromium from chromium-contaminated wastewater.

Many treatment technologies for removal of heavy metals from wastewater have been conducted [13]. Adsorption methods based on the high porosity, specific surface and high surface activity of adsorbents are used to remove chromium (VI) species from wastewater [14–16], while it is only applicable for treatment of low concentration wastewater. Membrane filtration methods seriously suffer from lack of resistance of the membranes to the harsh conditions typically found in industry wastewaters [17]. Ambient chromium mainly exists in the oxidative states of chromium (VI) and chromium (III). The chromium (III) species are relatively stable and have low solubility and mobility in environmental conditions. In contrast, the chromium (VI) species are highly soluble and mobile and also more poisonous than chromium (III). Mostly, chromium (VI) species are reduced to chromium (III) species and then treated with other technologies, like adsorption or precipitation [18–21], but occupied with high dosage of reducing agent and acid or alkaline, also large amount of chromium-containing sludge.

In this paper, chemical precipitation was chosen to recover chromium from the solution. Lead sulfate was selected as precipitation agent to precipitate chromium (VI) based on the difference of the solubility constant between lead sulfate and lead chromate, for which was 1.6×10^{-8} and 2.8×10^{-13}, respectively. The effects of parameters on the precipitation efficiency of chromium including initial pH of solution, reaction temperature, reaction time and dosage of lead sulfate were studied.

2. Experimental

2.1. Materials

All the reagents were analytical grade, including sulfuric acid, potassium dichromate, sodium hydroxide, lead sulfate used for precipitation reaction and phosphoric acid and ammonium ferrous sulfate, hexamethylenetetramine, potassium permanganate, and *N*-phenylanthranilic used in the chemical analysis.

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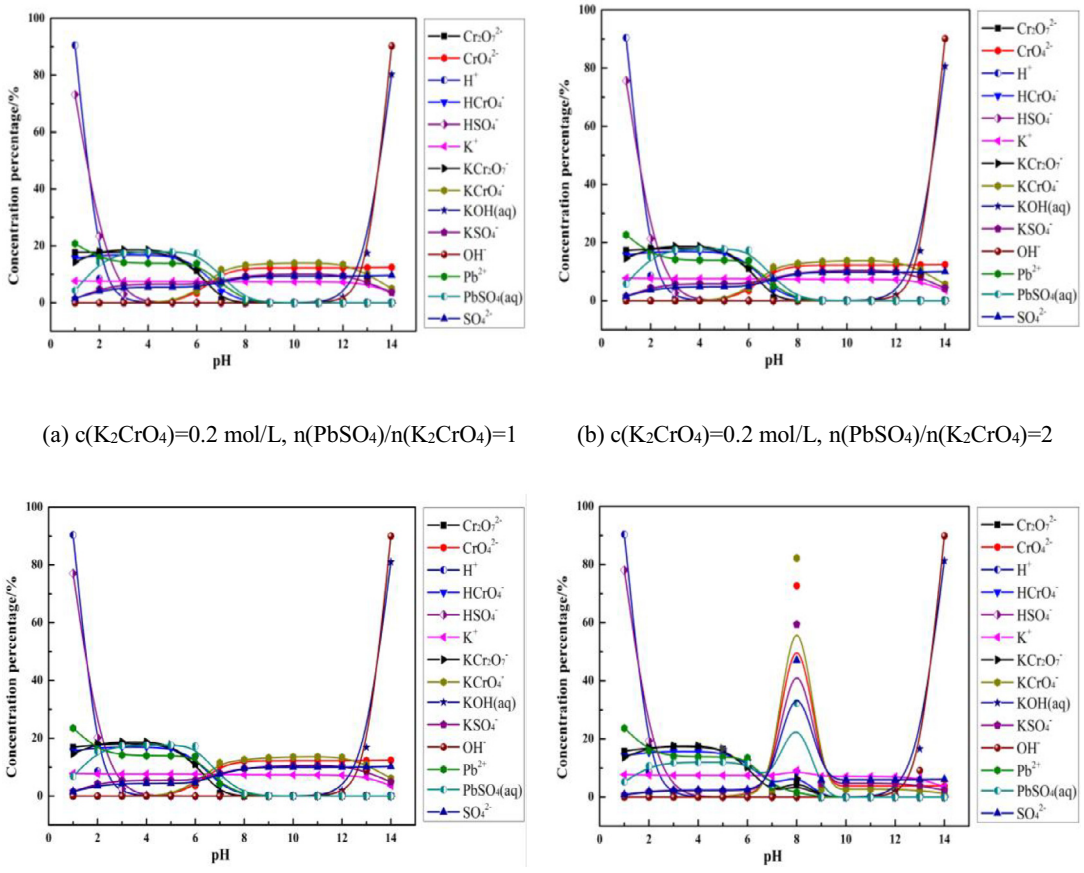

(a) c(K_2CrO_4)=0.2 mol/L, n($PbSO_4$)/n(K_2CrO_4)=1

(b) c(K_2CrO_4)=0.2 mol/L, n($PbSO_4$)/n(K_2CrO_4)=2

(c) c(K_2CrO_4)=0.2 mol/L, n($PbSO_4$)/n(K_2CrO_4)=3

(d) c(K_2CrO_4)=0.2 mol/L, n($PbSO_4$)/n(K_2CrO_4)=4

Fig. 1. Effect of pH value on species of the solution of K^+–CrO_4^{2-}–Pb^{2+}–SO_4^{2-}.

The chromium solution for the experiment was prepared by dissolving potassium dichromate in deionized water. The deionized water used in the experiments was produced by a water purification system (HMC-WS10).

2.2. Apparatus and procedures

All experiments were performed in a glass beaker with a thermostatic mixing water bath pot.

A predetermined amount of potassium dichromate and deionized water was added to the beaker to produce a homogeneous solution under constant stirring. Subsequently, the pH value of the solution was adjusted with sulfuric acid and sodium hydroxide. The solution was heated to a predetermined temperature. Next, the lead sulfate was added to the beaker. After the required reaction time had lapsed, the filtrate was separated from the precipitation through vacuum filtration.

Titration with ammonium ferrous sulfate is used to determine the concentration of chromium in the filtrate [22]. Precipitation efficiency of chromium is calculated using the following formula:

$$\eta = \frac{(C_1 \times V_1 - C_2 \times V_2)}{C_1 \times V_1} \times 100\% \quad (1)$$

where η is the chromium precipitation efficiency, %; C_1 is the total chromium concentration in the chromium aqueous, g/L; V_1 is the volume of chromium aqueous, mL; C_2 is the total chromium concentration in the filtrate after the reaction, g/L; and V_2 is the volume of filtrate after the reaction, mL.

3. Results and discussion

3.1. Technology principle

3.1.1. The composition in the solution of K^+–CrO_4^{2-}–Pb^{2+}–SO_4^{2-}

The composition in the K_2CrO_4 solution was calculated by Visual MINTEQ software, including the presence form, the dissolution and equilibrium and the saturated state of the solid. The calculating conditions were set with concentration of K_2CrO_4 solution at 0.2 mol/L, the dosage of lead sulfate was set as molar ratio at n ($PbSO_4$)/n (K_2CrO_4) = 1, n ($PbSO_4$) /n (K_2CrO_4) = 2, n ($PbSO_4$)/n (K_2CrO_4) = 3, n ($PbSO_4$)/n (K_2CrO_4) = 4, reaction temperature of 25 °C, pH varied from 1 to 14. The activity coefficient of the charged materials was calculated with Davies equation [23]. The results were shown in Fig. 1.

The results shown in Fig. 1 indicated that the main species in the solution were $Cr_2O_7^{2-}$, CrO_4^{2-}, H^+, $HCrO_4^-$, HSO_4^-, K^+, $KCr_2O_7^-$, $KCrO_4^-$, SO_4^{2-}, KSO_4^-, OH^-, and Pb^{2+}. The chromium (VI) was mainly existed in form of $Cr_2O_7^{2-}$, $HCrO_4^-$ and $KCr_2O_7^-$ at pH < 7, and then converted to $KCrO_4^-$ and CrO_4^{2-} as pH value increased. When the pH value of solution was below 7, the lead in the solution existed in Pb^{2+} and $PbSO_4$ (aq) which was poisonous and harmful to the environment. When the pH value increased above 8, the concentration of Pb^{2+} and $PbSO_4$ (aq) was nearly 0. Therefore, the precipitating process of chromium with lead should be reacted at high pH value in order to avoid the environment pollution caused by lead. Otherwise, the results showed that the dosage of lead sulfate had little influence on the species of the solution according to the results shown in Fig. 1.

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