



A novel D2EHPA-based synergistic extraction system for the recovery of chromium (III)



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H I G H L I G H T S

- Cr (III) was recovered by D2EHPA–isooctanol synergistic extraction system.
- Influence factors in synergistic extraction process were studied in detail.
- Stripping of Cr (III) from the synergistic extraction system was investigated.
- Mechanism on Cr (III) extraction by mixed extractant was studied by UV–vis, FT-IR.

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A synergistic solvent extraction method for recovering chromium (III) was proposed using the mixed extractant consisting of di (2-ethylhexyl) phosphoric acid (D2EHPA) and isooctanol. Based on the synergistic effect and the price of extractant, isooctanol was selected as the effective synergistic extractant in the D2EHPA-based extraction system. The chromium (III) extraction was found to increase and then decrease with increasing isooctanol concentration. The effects of D2EHPA concentration, initial pH, contact time, temperature, and anions concentration on the synergistic extraction of chromium (III) were investigated. The comparative study on the stripping of chromium from the loaded D2EHPA–isooctanol and the loaded D2EHPA was carried out. Based on the experiment results and the analysis of UV–vis and FT-IR spectra, the mechanism on the synergistic extraction of chromium (III) by D2EHPA–isooctanol mixed extractant was discussed.

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1. Introduction

Chromium is widely used in many industries, such as electroplating, wood preservation, leather tanning, textile, metallurgy, and pigments [1]. Large amounts of chromium-containing waste water and sludge are discharged from various industries, causing environmental problems. In the waste water or the leaching liquor of sludge, chromium occurs in two common valence states, i.e. Cr (VI) and Cr (III). Cr (VI) is highly toxic to humans and animals because of its strong oxidizing nature and carcinogenic effect [2,3]. Cr (III) is less toxic than Cr (VI) and is regarded as an essential trace element required for humans, so the reduction of Cr (VI) to Cr

(III) is usually performed in the treatment of wastewater [4,5]. However, accumulated Cr (III) is reported to be also toxic to the living creatures because it is not easily absorbed, besides Cr (III) can be oxidized to the more toxic Cr (VI) under some conditions. Therefore, the discharge of Cr (III) is also regulated [6]. There have been increasing concerns on the removal of Cr (III) to meet the discharge standard, moreover, it brings economic benefit if chromium is recovered [7]. Various methods have been studied for the removal of heavy metals from the aqueous solution, such as solvent extraction, precipitation, ion exchange, electro-Fenton process, photo-Fenton's process, vermiremediation, adsorption, and membrane process [8–17]. Solvent extraction is widely applied due to its mild reaction condition, continuous operation, and high recovery.

Various extractants have been employed for the removal of the anionic, neutral, and cationic species of Cr (III). The anionic species of Cr (III) were extracted using amine extractants e.g. Aliquat 336,

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TOA from alkaline media, ethylenediaminetetraacetic acid media, and thiocyanic acid media [18–20]. The neutral species of Cr (III) were extracted using neutral extractants e.g. TOPO, TBP, and polyethoxylated alcohols from perchloric acid, nitric acid media [21–23]. The cationic species of Cr (III) were extracted using acidic extractants e.g. D2EHPA, PC-88A, and Cyanex 272 from sulfuric acid media, hydrochloric acid media, and nitric acid media [24–31].

The recovery of chromium (III) using D2EHPA, an easily available and relatively cheap acidic extractant, has been investigated in different systems. The chromium (III) recovery by liquid–liquid extraction was conducted using D2EHPA, and the factors in the extraction and stripping were studied [26,27]. The removal of chromium (III) by three-liquid-phase extraction method was carried out using D2EHPA, and the effects on the partitioning behavior were investigated [29]. The chromium (III) separation was performed by ACM and PIM using D2EHPA as an ion carrier, and the conditions of chromium (III) transport were optimized [24,25,28]. In the previous studies, D2EHPA was employed as the single extractant for chromium (III) extraction. The single D2EHPA extraction system suffered from some drawbacks, such as small loading capacity of chromium (III) and slow phase separation. Synergistic solvent extraction, which could improve the metal recovery or selectivity, caused increasing attention. Thus, the effect of mixing different extractants with D2EHPA on chromium (III) extraction was studied and isooctanol was selected as the effective synergistic extractant in this paper. The extraction of chromium (III) from the aqueous solution using a D2EHPA–isooctanol synergistic system has not been reported.

In this study, the extractant composed of D2EHPA and isooctanol in kerosene was employed to extract chromium (III) from the aqueous solution. The influences of various parameters on the extraction and stripping of chromium (III) were investigated. An obvious synergistic effect was observed, and the mechanism of chromium (III) extraction using the D2EHPA–isooctanol extractant was studied.

2. Experimental

2.1. Reagents and instruments

D2EHPA was purchased from the Sinopharm Chemical Reagent Co., Ltd, and isooctanol was supplied by the Xilong Chemical Co., Ltd. In all experiments, 50% D2EHPA was ammoniated by aqueous ammonia (25–28%, Xilong Chemical Co., Ltd). All extractants were used without further purification. Feed solutions containing chromium (III) were prepared by dissolving $\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$ ($\geq 99.0\%$, Sinopharm Chemical Reagent Co., Ltd) in distilled water. The pH of the aqueous phase was adjusted using hydrochloric acid or sodium hydroxide. Other chemicals used were of analytical reagent grade. The concentration of chromium in the aqueous phase was measured by inductively coupled plasma-optical emission spectroscopy (ICP-OES, Optima 5300DV, Perkin-Elmer). The pH of solution was measured using a pH meter (S220, Mettler). UV–vis spectra were measured using a UV–visible spectrophotometer (UV-9100 B, LabTech). FT-IR spectra were measured using a FTIR spectrometer (Spectrum GX, Perkin-Elmer).

2.2. Procedure

Extraction experiments were carried out in the stoppered conical flasks using the mechanical agitation at a speed of 200 r/min, and phase separation was performed using the separatory funnels. After phase separation, the concentrations of chromium in raffinate were analyzed with ICP-OES (the wavelength for chromium determination was 267.72 nm). The concentrations of chromium

in the loaded organic phase were calculated according to mass balance.

The loaded organic phase and stripping reagent were mixed in the stoppered conical flasks using the mechanical agitation at a speed of 200 r/min, and phase separation was performed using the separatory funnels. After phase separation, the concentrations of chromium in the stripping solution were analyzed with ICP-OES.

2.3. Treatment of data

The extraction (E), distribution ratio (D), and stripping (S) were calculated as follows:

$$E = \frac{V_{\text{Org.}} C_{\text{Org.}}}{V_{\text{F}} C_{\text{F}}} \times 100\% = \frac{D}{D + \frac{V_{\text{Aq.}}}{V_{\text{Org.}}}} \times 100\% \quad (1)$$

$$D = \frac{C_{\text{Org.}}}{C_{\text{R}}} \quad (2)$$

$$S = \frac{V'_{\text{Aq.}} C'_{\text{Aq.}}}{V_{\text{Org.}} C_{\text{Org.}}} \times 100\% \quad (3)$$

where C_{F} , $C_{\text{Org.}}$, C_{R} and $C'_{\text{Aq.}}$ are the concentrations of chromium in feed solution, organic phase, raffinate and stripping solution, respectively. V_{F} , $V_{\text{Org.}}$, $V_{\text{Aq.}}$ and $V'_{\text{Aq.}}$ are the volumes of feed solution, organic phase, aqueous phase and stripping solution, respectively.

3. Results and discussion

3.1. Solvent extraction of chromium (III)

3.1.1. Selectivity of extractant on the extraction of chromium (III)

The extraction efficiency of different types of extractants on chromium (III) extraction was studied. As shown in Table 1, the acidic extractant could extract chromium (III) from the acidic solution, whereas the extraction of chromium (III) by neutral extractant and basic extractant was negligible. This was due to the cationic forms of chromium (III) ions and the cation-exchange mechanism of acidic extractant. In the acidic solution, the forms of chromium (III) species were Cr^{3+} , $\text{Cr}(\text{OH})^{2+}$, and $\text{Cr}(\text{OH})_2^+$ [29]. The chromium (III) extraction efficiency of acidic extractant was in the following order: D2EHPA > PC88-A > Cyanex 272. The acidity of D2EHPA is stronger than that of PC88-A and Cyanex 272, therefore, the hydrogen atom in P–O–H bond of D2EHPA is easily substituted by Cr^{3+} , $\text{Cr}(\text{OH})^{2+}$, and $\text{Cr}(\text{OH})_2^+$. Hence, D2EHPA was employed as the cheap and effective extractant for chromium (III) extraction.

Table 1

Effect of extractant on the extraction of chromium (III), experimental conditions: 10 vol.% extractant in kerosene, initial pH of 1.63, chromium concentration of 3.40 g/L, contact time of 30 min, and temperature of 15 °C.

Type of extractant	Extractant	Chromium extraction (%)
Acidic extractant	D2EHPA	49.7
	PC88-A	46.5
	Cyanex 272	41.9
Neutral extractant	TBP	0
	MIBK	0
	2-octanol	0
	Cyanex 923	0
Basic extractant	Aliquat 336	0

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