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# Selective recovery of vanadium and scandium by ion exchange with D201 and solvent extraction using P507 from hydrochloric acid leaching solution of red mud

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# HIGHLIGHTS

• Selective separation of V and Sc from acid solution using D201 and P507, respectively, was studied.

• The main parameters of adsorption and extraction of V and Sc were studied in detail.

• A flow sheet was proposed for the recovery of V and Sc from red mud acid leaching solution.

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# ABSTRACT

D201 resin and P507 extractant diluted with sulfonated kerosene were used to respectively separate vanadium and scandium, and impurity ions from hydrochloric acid leaching solution of red mud. More than 99% of vanadium was selectively adsorbed from the hydrochloric acid leaching solution under the conditions of pH value of 1.8, volume ratio of leaching solution to resin of 10, and flow rate of 3.33 mL/ min. Maximum extraction and separation of scandium was observed from the acid leaching solution at an aqueous pH value of 0.2. More than 99% of scandium can be selectively extracted using 15% P507, 5% TBP at the aqueous solution/organic phase (A/O) ratio of 10:1 for 6 min. The loaded organic phase was washed with 0.3 mol/L sulfuric acid, wherein most impurities were removed. After the process of desorption or stripping, precipitation, and roasting, high-purity V<sub>2</sub>O<sub>5</sub> and Sc<sub>2</sub>O<sub>3</sub> were obtained. Finally, a conceptual flow sheet was established to separate and recover vanadium and scandium from red mud hydrochloric acid leaching solution.

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# 1. Introduction

Vanadium and scandium are important rare metals that have been widely used in emerging industries. Vanadium mainly exists in titano-magnetite and stone coal and scandium rarely exists as rich ore (Wang et al., 2009; Zhang et al., 2011; Zhu et al., 2012). Red mud is a solid waste from the tailings during alumina production from bauxite. It is estimated that more than 2.7 billion tonnes of the residue exists, increasing at 120 million tonnes per annum in the world (Wang et al., 2008). Red mud usually contains Ti, A1, Fe Na, Ca, Si, Sc, and V (Zhu et al., 2015a,b). For example, the red mud produced in China, Greece, and India contains 100–200 mg/kg of Sc and 0.1–0.4 wt% of V (Wang et al., 2013; Xu et al., 2016). The extraction of Sc, V, Ti, and other rare metals from red mud was investigated using concentrated sulfuric acid for high recovery of valuable metals. Sulfuric acid at a concentration of 6 mol/L or even 10 mol/L was needed and all valuable metal ions were dissolved into the leaching solution (Kima et al., 2015; Power et al., 2011; Klauber et al., 2011; Li et al., 2014; Borra et al., 2015). However, for the process of hydrochloric acid leaching at appropriate concentrations, Sc and V were





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dissolved into this leaching solution (Sneha et al., 2013; Huang et al., 2016), while Ti, as a valuable metal, still existed in the leaching residue of red mud. Therefore, it is important to further study the recovery of vanadium and scandium from hydrochloric acid leaching solution.

Ion exchange is usually used to separate vanadium from neutral and alkaline solution. However, in acidic solution, vanadium cannot be easily separated because of many metallic impurities in some resins (Zeng et al., 2009). Numerous researchers have studied and developed the solvent extraction for the separation of vanadium, scandium, and other ions from acid leaching solutions. The acidic extractants such as D2EHPA, N1923, N235, and Cyanex were used in previous studies (Zeng and Cheng, 2010; Yang et al., 2016; Sun et al., 2011; Zhu et al., 2015a,b; Jiang et al., 2015; Xie et al., 2014). However, these extractants can usually co-extract vanadium and scandium along with other metals such as iron and aluminum. Particularly, vanadium and scandium were difficult to separate by solvent extraction using P507 or P204 because of their high extraction rates. Furthermore, iron could obviously affect the extraction of vanadium and scandium even at high acidity (Li et al., 2012, 2013). Therefore, selective separation of vanadium and scandium from the acid leaching solution with many impurity ions is necessary.

In this research, ion exchange and solvent extraction experiments were combined to efficiently separate vanadium, scandium, and other metal elements. Some anion exchange resins and acidic extractants were used, and the behaviors of adsorption and extraction of V(V), Sc(III), Fe(III), Al(III), Ca(II), Na(I), and Mg(II) from the hydrochloric acid leaching solution of red mud were analyzed. The appropriate recovery technology and the parameters were studied for the separation of vanadium, scandium, and other metal ions according to the properties of the hydrochloric acid leaching solution of red mud. Finally, a conceptual process flow sheet for vanadium and scandium recovery from red mud was proposed.

# 2. Experimental procedure

## 2.1. Materials and methods

The acid leaching solution was prepared by leaching red mud with 3 mol/L of HCl and 0.5 wt% of NaClO3 at 50 °C for 12 h according to our previous studies. The chemical composition of the acid solution is shown in Table 1. The pH value of this solution is 1.47. The valence state of vanadium in the solution is V(V). Analytical grade resins D201,  $201 \times 7$ , and D452 were obtained from Shanghai Aike Resin Co., Ltd, China. The resins were first steeped and washed with 1 mol/L HCl for 24 h and then washed with distilled water to about a pH of 7.0. The washed resins were continued to steep and wash with 1 mol/L of NaOH for 24 h and then were washed with distilled water to about a pH of 7.0. The washed resins were steeped and washed with 1 mol/L of ethyl alcohol for 12 h. Finally, the resins were washed with distilled water until the pH of the solution reached 7 before use. 2-Ethyl hexyl phosphonic acid 2-ethyl hexyl ester (P507, ≥98%), Bis(2-ethylhexyl) phosphate (P204, ≥98%), primary amine N1923, tri-n-butyl

Table 1		
The chemical composition of leaching solution,	adsorption tail lic	uid, and raffinate

Composition	V	Sc	Fe	Al	Ca	Na	Mg
Leaching solution (mg/L)	127	15	464	1379	2645	3324	664
Adsorption tail liquid (mg/L)		15	453	1369	2638	3317	657
Raffinate (mg/L)		-	427	1297	2514	3295	643

phosphate (TBP,  $\geq$ 99%), and sulfonated kerosene (AR) were supplied by Dongda Chemistry Co., Ltd.

#### 2.2. Procedure of ion exchange and solvent extractio

The leaching solution was adjusted to the prospective pH value by adding 4 mol/L of  $H_2SO_4$  and 4 mol/L of NaOH solutions prior to ion exchange. Ion exchange experiments were performed using a glass column ( $\Phi$  1 × 50 cm), where 20 mL of the pretreated resins were wet-packed. The operation was processed with 200 mL of the adjusted solution at 3.33 mL/min (except in the resin dosage and flow rate experiments). The adsorption tail liquid was collected, and the concentrations of the elements in the liquid were analyzed. The loaded resins were desorbed by 1.5 mol/L of NaOH solution.

The pH value of the adsorption tail liquid was adjusted by adding 4 mol/L of H<sub>2</sub>SO<sub>4</sub> prior to solvent extraction. Extractants were prepared using 15% P507, P204, or N1923 (V/V), 5% TBP, and 80% sulfonated kerosene. In a 250-mL volumetric flask, 50 mL of the adjusted adsorption tail liquid and 5 mL of the organic solutions (except in A/O ratio experiments) were mixed and shaken using a mechanical shaker for 6 min (except in time experiments). The raffinate and organic phases were separated using separating funnels. The loaded organic phase was washed with H<sub>2</sub>SO<sub>4</sub> solution at an A/O ratio of 2 for 8 min with a mechanical shaker. The washed aqueous and organic phases were also separated using separating funnels. Finally, the washed organic phases were stripped with 2 mol/L NaOH solution.

#### 2.3. Chemical analyses and data treatment

The concentrations of the elements in acid solution were analyzed using inductively coupled plasma atomic emission spectrometry (ICP-AES). Furthermore, after ion exchange and solvent extraction, the concentrations of the elements in the aqueous phase were also analyzed using ICP-AES.

The adsorption rate ( $\eta$ ), extraction rate (R), and washing rate (w) were calculated by Eqs. (1)–(3), respectively.

$$\eta = (n-m)/n \times 100\% \tag{1}$$

$$R = (\mathbf{m} - \mathbf{p})/\mathbf{m} \times 100\% \tag{2}$$

$$w = s/q \times 100\% \tag{3}$$

where n is the content of the corresponding element in the initial acid leaching solution (g), m is the content of the corresponding element in the adsorption tail liquid (g), p is the content of the corresponding element in the raffinate (g), s is the content of the corresponding element in the washed aqueous phases (g), and q is the content of the corresponding element in the organic phases (g).

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The characteristics and effect of the used resins and extraction agents.

Resins	Resin matrix	Resin typ	e	Function	group	Recovery of vanadium
D201 201 × 7 D452	Styrene monomer	Strong ba Weak bas	se e	N-(CH <sub>3</sub> ) <sub>2</sub> -N(CH <sub>3</sub> ) <sub>2</sub> -N(CH <sub>3</sub> ) <sub>2</sub>	C₂H₄OH ∙H₂O	99.5% 82.6% 76.4%
Extraction	agent		A/O	pН	Recovery and iron	of scandium
P507 (2-ethyl hexyl phosphonic acid 2-ethyl hexyl ester) P204 (Bis(2-ethylhexyl) phosphate) N1923 (Primary amine N1923)		10:1	1 0.2	99.3% an 92.4% an 46.5% an	d 8.4% d 37.9% d 29.4%	

# 3. Results and discussion

# 3.1. Vanadium separation with ion exchange

#### 3.1.1. Effect of resins' type on vanadium recovery

It is well known that the vanadium species in a solution contains cationic species, neutral species, and anion species according to solution thermodynamic (Zhou et al., 2011). The species of vanadium varies under different pH values. However, other metal ions are almost not present as anion species in acidic solution (Mu et al., 2011).

V(V) is mostly in the form of cationic species VO $\frac{1}{2}$  and anion species H<sub>2</sub>VO<sub>4</sub> at pH < 2.0 and exists in various kinds of anion species at pH > 2.0. Furthermore, the vanadium species existing in aqueous solution depends on the solution conditions such as pH value, Eh, and valence of vanadium. However, V(IV) can exist as cationic species and anion species in aqueous solution under different pH values, where the existence of HV<sub>2</sub>O<sub>5</sub> is at pH > 4.0. The pH value of hydrochloric acid leaching solution from red mud was about 1.5, in which the valence of vanadium was V(V). The characteristics and effect of treated resins on vanadium recovery at pH of 1.8 and flow rate of 3.33 mL/min are shown in Table 2.

The results in Table 2 indicate that D201 resin had a better adsorption performance of vanadium than the other two resins. The adsorption percentage of vanadium reached 99.5% using D201 resin at pH 1.8, whereas it was less than 85% using 201  $\times$  7 and D452 resins under the same pH condition. D201 is a strongly alkaline macroporous resin that shows a superior adsorption performance for vanadium in a weakly acidic solution. Thus, being an optimum type of resin for this study, D201 resin was used to investigate the effect of experimental parameters on vanadium separation.

#### 3.1.2. Effect of pH value on vanadium separation

The effect of the solution pH value on vanadium separation was investigated under the conditions of flow rate of 3.33 mL/min and volume liquid to resin ratio of 10. The results are shown in Fig. 1.

The results indicate that the pH value of the solution is an important factor for the separation and recovery of vanadium. The recovery of vanadium increased from 21% to 99% as the pH value increased from 0.6 to 1.8. The recovery of vanadium could reach 100% with further increase in the pH value. The adsorption percentage of iron is slightly increased with increasing pH value, whereas other metal ions are almost not adsorbed by D201 resin. D201 is an anion resin that adsorbs anions in an acid leaching solution. Vanadium appeared in the form of anions under these conditions, but other metal ions existed in cations in this acid leaching solution. Therefore, the selective recovery of vanadium from the acid leaching solution was made possible using D201 resin. Iron that existed as small crystals of ferric hydroxide was physically adsorbed by D201. The washing and delamination with distilled water on loaded D201 resin were performed to remove the iron impurities after vanadium adsorption. More than 99% iron was removed by washing with water.

#### 3.1.3. Effect of flow rate on vanadium separation

The effect of flow rate on vanadium separation was studied under the conditions of pH value of 1.8 and liquid to resin volume ratio of 10. The results are shown in Fig. 2(A).

The recovery of vanadium increased from 60% to 99% with decreasing flow rate from 13.33 to 3.33 mL/min. When the flow rate was less than 3.33 mL/min, the recovery of vanadium was almost constant. The prolonged adsorption time resulted in increased iron adsorption by about 5%, whereas it has almost no influence on other metal ions. Therefore, 3.33 mL/min was selected as the optimal flow rate.

#### 3.1.4. Effect of resin dosage on vanadium separation

The effect of D201 resin dosage on vanadium separation was studied under the conditions of pH value of 1.8 and flow rate of 3.33 mL/min. The results are indicated in Fig. 2(B).

The recovery of vanadium decreased very slowly with increasing volume ratio of leaching solution to resin. The



Fig. 1. Effect of initial pH on metal ions' recovery with D201 resin.



Fig. 2. Effects of resin dosage and flow rate on metal ions' extraction with D201 resin.

phenomenon became obvious with the increase in volume ratio of leaching solution to resin from 10 to 14, where the recovery of vanadium decreased from 99% to 84%. However, the adsorptive property of the other metal ions was almost not affected with increasing volume ratio of leaching solution to resin. Therefore, the optimal volume ratio of leaching solution to resin was 8–10.

# 3.2. Scandium separation with solvent extraction

The feeding liquid for solvent extraction is the adsorption tail liquid by adjusting the pH value. The adsorption tail liquid was obtained under the conditions of pH value of 1.8, volume ratio of leaching solution to resin of 10, and adsorption time of 60 min. The chemical composition of the adsorption tail liquid is listed in Table 1.

# 3.2.1. Effect of extractants on scandium recovery

The effect of extraction type on the separation and recovery of scandium was determined by shaking tests under the conditions of organic solution consisting of 15% (V/V) extraction agent, 5% (V/V) TBP, and 80% sulfonated kerosene, A/O ratio of 10, pH value of 0.2, and equilibrium time of 6 min. The results are shown in Table 2.

The results show that P507 exhibited better extraction and separation performance for scandium and iron than the other two resins under the same conditions. The extraction percentage of scandium reached 99.3% using P507 at a pH value of 0.2, whereas the extraction percentage of iron was only 8.4%. The extraction percentage of scandium was 92.4% and 46.5% using P204 and N1923, respectively, under the same conditions, and the extraction percentage of iron was 37.9% and 29.4%, respectively. Therefore, the co-extraction of iron was more obvious with P204 and N1923. P507 is a selective extraction reagent that is used at low pH value of the



Fig. 3. Effect of initial pH on metal ions' extraction with P507.



Fig. 4. Effect of P507 concentration on metal ions' extraction.

solution. Being an appropriate extraction agent for this study, P507 was used to investigate the effect of experimental parameters on the separation of scandium.

ratio of 10 for 6 min. The results are shown in Fig. 3.

The results show that the extraction of scandium increased obviously from 83% to 99% in the pH range of 0-0.2 and was stable at pH value more than 0.2. However, the extraction of iron increased from 8% to 20% in the pH range of 0.2-1.0 and presented remarkable improvement from 20% to 52% with increasing pH value from 1.0 to 1.4. The extraction percentage of aluminum was observed to increase slowly with increasing pH value from 0 to 1.4

The effect of pH value on the separation of scandium was determined by shaking tests in the aqueous pH value range of 0-1.4 under the conditions of 15% (v/v) P507 and 5% (v/v) TBP with an A/O

and reached 4.2% at the pH value of 0.2. The extraction performance of the other metal ions almost did not change in the current pH value range. Therefore, 0.2 was selected as the optimal pH value.

#### 3.2.3. Effect of P507 concentration on scandium separation

The effect of increasing the P507 concentration (v/v) from 5% to 25% on scandium separation was investigated under the conditions of the solution pH value of 0.2, A/O ratio of 10, and equilibrium time of 6 min. The results are presented in Fig. 4.

The recovery of scandium increased from 74% to 99% with increasing P507 concentration (v/v) from 5% to 15% and showed no remarkable improvement at P507 concentration (v/v) more than 15%. Furthermore, the extraction of iron and aluminum increased with increasing P507 concentration. The recovery of iron and aluminum was nearly 20% and 10%, respectively, with 20% P507, whereas it was only 8% and 4%, respectively, with 15% P507. The other metal ions were almost not extracted by P507 with increasing concentration. Therefore, the optimal P507 concentration (v/v) was

determined to be 15% for extraction of more scandium with less impurity.

# 3.2.4. Effect of the A/O ratio on scandium separation

The effect of increasing A/O ratio from 4 to 14 on scandium separation was studied under the conditions of P507 concentration (v/v) of 15%, TBP concentration (v/v) of 5%, solution pH value of 0.2, and equilibrium time of 6 min. The results are shown in Fig. 5(A).

Fig. 5(A) indicates that the extraction of scandium was almost consistent with increasing A/O ratio from 4 to 10, whereas the extraction of scandium decreased from 99% to 82% with increasing A/O ratio from 10 to 14. The extraction of iron and aluminum also decreased with increasing A/O ratio. The extraction of iron decreased from 20% to 8% with increasing A/O ratio from 4 to 10, while the extraction of aluminum decreased from 10% to 4%. The extraction of iron and aluminum slightly decreased with increasing A/O ratio from 10 to 14. Similarly, the extraction of the other metal ions was almost constant with increasing A/O ratio. Therefore, the optimal A/O ratio was selected as 10.



Fig. 5. Effects of A/O and time on metal ions' extraction with P507.



Fig. 6. Flow sheet for vanadium and scandium recovery from red mud.

# 3.2.5. Effect of time on scandium separation

The effect of increasing equilibrium time from 2 to 10 min on scandium separation was investigated under the conditions of P507 concentration (v/v) of 15%, TBP concentration (v/v) of 5%, solution pH value of 0.2, and A/O ratio of 10. The results are indicated in Fig. 5(B).

The figure shows that the recovery of scandium increased from 78% to 99% with increasing equilibrium time from 2 to 6 min. The increase was not obvious when the equilibrium time was increased from 6 to 10 min. Similarly, the extraction of iron and aluminum

increased with increasing equilibrium time from 2 to 10 min, and the increase in iron extraction was obvious above the equilibrium time of 6 min. However, the extraction of the other metal ions was almost zero with increasing equilibrium time. Therefore, the optimal equilibrium time was determined to be 6 min.

# 3.2.6. Washing of the loaded organic phase

The raffinate was obtained under the conditions of P507 concentration (v/v) of 15%, TBP concentration (v/v) of 5%, solution pH

value of 0.2, A/O ratio of 10, and equilibrium time of 6 min. The chemical composition of the raffinate is listed in Table 1.

The results show that scandium was almost extracted under the above conditions. Further, some impurity ions were also extracted particularly iron and aluminum. The washing of loaded organic phase was carried out to efficiently separate scandium and impurity ions. The loaded organic phase was washed with sulfuric acid of different concentrations. Scandium was almost not washed with increasing sulfuric acid concentration from 0.1 to 0.5 mol/L. After washing rate of iron and aluminum was increased from 90% to 100%. The other impurity ions could be fully washed with this sulfuric acid concentration range. The optimal sulfuric acid concentration for 0.3 mol/L.

#### 3.3. Flow sheet of vanadium and scandium recovery

The loaded D201 resin was used to adsorb vanadium of new hydrochloric acid leaching solution. The saturation extent of adsorption of vanadium by D201 resin was 452 mg(V)/g (resin). Then, the saturated D201 resin was desorbed under the conditions of strippant of 3% NaOH and 8% NaCl, flow rate of strippant of 0.02 mL/min, and volume ratio of strippant to D201 resin of 4:1. Under these conditions, the desorption rate of vanadium was 97.5%, and the concentration of  $V_2O_5$  in the stripping solution was 12.3 g/L. Polymer ammonium vanadate was crystallized and precipitated using NH<sub>4</sub>Cl at 95 °C and pH value of 2.1. in which the precipitation rate of vanadium was 99.5%. After roasting the polymer ammonium vanadate at 520 °C for 20 min, the purity of V<sub>2</sub>O<sub>5</sub> was 99.4%, thus meeting the standard specification of requirement of more than 99% of V<sub>2</sub>O<sub>5</sub> grade. The loaded organic phase was used to continue to extract the scandium from the new adsorption tail liquid in three stages. The saturated loaded organic phase was stripped under the conditions of sodium hydroxide concentration of 2 mol/L, O/A ratio of 5, and equilibrium time of 8 min. The stripping rate of scandium was 95.4%, and the precipitate of  $Sc(OH)_3$  was obtained. Then, the precipitate of Sc(OH)<sub>3</sub> was dissolved in 3 mol/L of sulfuric acid solution and re-precipitate of Sc<sub>2</sub>(C<sub>2</sub>O<sub>4</sub>)<sub>3</sub> was obtained. The Sc<sub>2</sub>O<sub>3</sub> of 99.2% purity was obtained with roasting the re-precipitate at 800 °C for 60 min. From the above results, a conceptual flow sheet for recovery and separation of vanadium and scandium from hydrochloric acid leaching solution of red mud was proposed as shown in Fig. 6.

# 4. Conclusions

Selective separation of vanadium and scandium from hydrochloric acid leaching solution of red mud by using D201 resin and P507 extractant, respectively, was investigated. Vanadium could be selectively adsorbed over scandium, iron, aluminum, calcium, sodium, and magnesium from the hydrochloric acid leaching solution under the conditions of pH value of 1.8, volume ratio of leaching solution to resin of 10, and flow rate of 3.33 mL/min. More than 99% of vanadium was adsorbed, but only a minimal amount of scandium, iron, aluminum, calcium, sodium, and magnesium were adsorbed. Scandium could be selectively extracted over iron, aluminum, calcium, sodium, and magnesium from the adsorption tail liquid by using 15% P507 at the pH value of 0.2. More than 99% of scandium was extracted, together with extraction of less than 10% iron and aluminum. Almost all the loaded impurities in the organic phase were washed away with 0.3 mol/L sulfuric acid without scandium. High-purity V<sub>2</sub>O<sub>5</sub> and Sc<sub>2</sub>O<sub>3</sub> could be obtained with desorption or stripping, precipitation, and roasting. Finally, a conceptual flow sheet to recover vanadium and scandium from red mud hydrochloric acid leaching solution was proposed.

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