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ORIGINAL ARTICLE

Recovery of rare earths and niobium from a potash feldspar ore beneficiation tailings



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KEYWORDS

Rare earths; Niobium; Potash feldspar ore beneficiation tailings comprehensively recovery **Abstract** In this paper, a hydrometallurgy process was investigated for recovering rare earths (REs) and niobium (Nb) from a potash feldspar ore beneficiation tailings. Firstly, the REs and Nb could be leached from potash feldspar ore beneficiation tailings by sulfuric acid roasting-water leaching process, and the effects of the roasting temperature, roasting time, sulfuric acid addition, leaching temperature, ratio of solid to liquid and leaching time for REs and Nb leaching rate were investigated, and the REs and Nb leaching rate could reach to 89.9% and 80.6% respectively at the optimum test conditions. Then, the REs was removed from the leaching liquor by adding HF, the precipitation rate could reach to 91.53%. The rare earths fluoride (REF₃) with the purity 60.51% was obtained, and the REs recovery rate reached to 70.6%. Finally, the Nb was recovered by solvent extraction with methyl isobutyl ketone (MIBK), Nb extraction rate could be reached 99.59%. The loaded organic phase could be stripped easily, nearly all niobium was stripped using 0.5 M sulfuric acid as strip liquor. The Nb₂O₅ products with the Nb₂O₅ content 87.61% was obtained by ammonia precipitation-roasting process, and the recovery Nb was 76.31% in the whole process.

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1. Introduction

Rare earths (REs) and niobium (Nb) are important strategic resources which are widely applied in the fields of optical, catalysis, superconductor, magnetism, luminescence, etc (Maestro and Huguenin, 1995; Wang et al., 2009). The demand for REs and Nb is increasing with the development of industry, and the exploitation of low grade REs and Nb ores had been encouraged, too (He et al., 2006; Omneya and Mohamed, 2002; Zhou et al., 2005; Zhang et al., 2014; Lv et al., 2012; Humphries, 2010). In China, large quantities of Nb and REs exists in form of multimetallic ore (Zhang et al., 2014; Lv et al., 2012), and this kind of resources contain the characteristics of low rare earth and niobium content, fine mineral dissemination size, difficulty monomer dissociation, which leaded to difficulty in recovery of REs and Nb.

A approach "sulfuric acid decomposition-leaching-selective hydrolysis-diatilling and calcining" for recovering Nb and REs from the Bayan Obo tailings were investigated, and the crude Nb_2O_5 products (60.67 wt% Nb_2O_5) and the composite RE oxides (88.65 wt% RE_xO_y) were obtained (Zhang et al., 2014). LIU Yong et al. reported a method for recovering rare earth and niobium from rare metal ores (Liu et al., 2011). This method took the polymetallic ore containing rare earth and niobium as the research object, niobium and rare earth were leached aqueous solution by the method of sulfuric acid roasting and water leaching, then the leaching solution was heated and boiled to form precipitation containing iron, niobium and titanium, and the rare earth still remained in the solution. The iron, niobium and titanium was recovered as ferroniobium alloy and titanium slag by magnetic separation and melted. The rare earth was recovered as rare earth oxide by oxalic acid precipitation and calcination.

Shaanxi Lueyang Anlingouxantaiba area is rich in syenite niobiumtantalum ore which was associated with rare earth resources (Zhang and Wang, 2013). Feldspar concentrate and Feldspar floatation tailings concentrate containing tantalum and niobium could be obtained through beneficiation from this kind of resources (Song et al., 2014). However, how to recovery rare earths and niobium from this kind of feldspar beneficiation tailings was rarely reported.

In this paper, a potash feldspar ore beneficiation tailings which contained niobium 0.24% and the RE_xO_y 0.78% in Shaanxi Lueyang Anlingou area was obtained, and a hydrometallurgical process "sulfuric acid decomposition-leaching-HF deposited-extraction-stripping-ca leining" for the REs and Nb recovery from potash feldspar beneficiation tailings was developed, and the different parameters on the leaching rate, extraction and stripping rate were investigated in detail, and The crude Nb₂O₅ products (87.61 wt% Nb₂O₅) and the composite REF₃ (60.51 wt% REF₃) were obtained.

2. Experimental section

2.1. Materials and reagents

The potash feldspar ore beneficiation tailings samples with the granularity of -0.074 mm accounted for 70% used in this study were obtained from Shaanxi Lueyang Anlingou area

(China), and the major metal elements was listed in Table 1. A variety of RE elements are present in potash feldspar ore tailing, Table 2 shows the content of RE elements in potash feldspar ore beneficiation tailings. From the Table1 and Table 2, we could see that the content of Nb₂O₅ and RE_xO_y in the potash feldspar ore beneficiation tailings were 0.24% and 0.78% respectively, which were lower than the Bayan Obo tailings and tailing of beneficiated complex rare metal associated ore (Zhang et al., 2014, Liu et al., 2015). And comparing the Bayan Obo tailings, there were more types of rareearth elements in the potash feldspar ore beneficiation tailings. Therefore, how to recovery rare earth and niobium from the potash feldspar ore beneficiation tailings with lower rare earth and niobium contents was a challenge. The tributyl phosphate (TBP) was obtained from Sinopharm Chemical Reagent Co. Ltd, China. Methyl isobutyl ketone MIBK, hydrofluoric acid (40 wt% HF) and Sulfuric acid (98 wt% H₂SO₄) were produced by Beijing Yili Chemical Co. Ltd, China.

2.2. Decomposition and leaching

Complex low-grade REs or Nb minerals were commonly decomposed by sulfuric acid roasting (Zhang et al., 2014; Wu, 2001; Liu et al., 2015), after which the REs and Nb would become soluble sulfates and be easily leached. In this paper, potash feldspar ore beneficiation tailing (20 g) and a certain amount of H_2SO_4 (98 wt%) were placed into a crucible and stirred well. Then, the crucible was placed into muffle burner and heated at 200 °C–300 °C for 1–4 h. Next, the decomposition sample was added to de-ionized water in a flask under mixing with a solid-to-liquid ratio of 1:2–1:10, leaching temperature 40 °C–90 °C, leaching time 0.5 h–4 h. Finally, the leaching solution was separated from the residue by filtration using a vacuum pump and a filter.

$$\eta_M^L = \frac{\mathbf{m} \cdot W_M - \mathbf{m}^1 \cdot W_M^1}{\mathbf{m} \cdot \mathbf{W}_M} \times 100\% \tag{1}$$

The leaching ratio was calculated as Eq. (1), where M is the element in the tailings; η_M^L is the leaching ratio of M, %; W_M is the concentration of M in tailings,%; m is the weight of tailings, g; W_M^1 is the concentration of M in leaching residue, %; m¹ is the weight of leaching residue, g.

2.3. Precipitation of RE elements

Rare earth in sulfuric acid solution could be recovered as rare earth fluoride precipitation by adding HF acid (Li et al., 2013). In this study, HF (40% concentration) was added to the leaching solution under stirring, the effect of HF addition and stirring time for the REs precipitation rate were investigated. The

Table 1 The metal elements of potash feldspar ore beneficiation tailings.							
Elements	Al ₂ O ₃	K ₂ O	SiO ₂	Fe _(total)	TiO ₂		
Content(%)	15.22	6.06	40.66	14.31	4.14		
Elements	Na ₂ O	Nb ₂ O ₅	Ta ₂ O ₅	$RE_{x}O_{y}$			
Content(%)	0.67	0.24	0.0081	0.78			

Table 2 The Rare earth element distribution in potash feldspar ore beneficiation tailings (g/t).

Elements	La ₂ O ₃	CeO ₂	Pr ₂ O ₃	Nd ₂ O ₃	Sm_2O_3	Eu ₂ O ₃	Gd_2O_3	Tb_2O_3
Content	1950	2750	480	1240	175	285	445	15
Elements	Dy ₂ O ₃	Ho ₂ O ₃	Er ₂ O ₃	Tm ₂ O ₃	Yb ₂ O ₃	Lu ₂ O ₃	Y ₂ O ₃	
Content	312	45	146	80	20	20	20	

gel type precipitation was gotten from the liquor by centrifugation at 5000 r/min for 10 min, and then the gel type precipitation was neutralized by 10% KOH solution, after washing with de-ionized water to remove KF, a light yellow precipitation enriching rare earth fluoride was gotten.

$$\eta_R^P = 100\% - \frac{C_R^1 \cdot V^1}{C_R \cdot V} \times 100\%$$
⁽²⁾

The precipitation ratio of REs was calculated as Eq. (2), where R is the REs in the leaching solution; η_R^P is the precipitation ratio of REs, %; C_R is the concentration of REs in leaching solution, mg/L; V is the volume of the leaching solution, ml; C_R¹ is the concentration of REs in precipitation filtrate, mg/L; V¹ is the volume of the precipitation filtrate, ml.

2.4. Extraction and stripping of Nb

The organic phase was MIBK or TBP, and the aqueous phase was the leaching solution after RE precipitation. Equilibrium distributions of Nb between organic and aqueous phases under different conditions were determined by transferring the two phases into a 60 ml separating funnel and mixing with a mechanical shaker for a certain time at room temperature.

$$\eta_N^E = 100\% - \frac{C_N^1}{C_N} \times 100\%$$
(3)

The extraction ratio of Nb was calculated as Eq. (3), where N is the Nb in the precipitation filtrate; η_N^E is the extraction ratio of Nb, %; C_N^1 is the concentration of Nb in raffinate, mg/L; C_N is the concentration of Nb in precipitation filtrate.

In the stripping experiments, 10 ml of loaded organic phase was taken and contacted with 5 ml of stripping solution (different concentrations of sulfuric acid) in separating funnels, followed by mechanical shaking to reach equilibrium.

$$\eta_N^S = 100\% - \frac{C_N^1}{C_N} \times 100\% \tag{4}$$

The stripping ratio of Nb was calculated as Eq. (4), where N is the Nb in the Loaded organic phase; η_N^S is the stripping ratio of Nb, %; C_N^1 is the concentration of Nb in stripping solution, mg/L; C_N is the concentration of Nb in loaded organic solution.

3. Results and discussion

3.1. The effect of sulfuric acid roasting conditions on the REs and Nb leaching ratio

In general, the roasting temperature, sulfuric acid addition and roasting time are important factors which influence the decomposition of ore during the roasting process. Fig. 1, Fig. 2 and Fig. 3 showed the effect of roasting temperature, roasting time

and sulfuric acid addition on RE and Nb leaching ratio respectively under the leaching conditions of leaching temperature 80 °C, the ratio of liquid to solid 6 and leaching time 2 h. Fig. 1 showed that the RE and Nb leaching ratio increased with the temperature increasing when the roasting temperature was lower than 300 °C. However, the leaching ratios decreased clearly while the roasting temperature was above 300 °C because of the decomposition of sulfuric acid (Zhang et al., 2014). And this variation was similar to the sulfuric acid decomposition for BayanObo tailings. Fig. 2 showed that the REs and Nb leaching ratio increased with the sulfuric acid addition while the ratio of sulfuric acid to potash feldspar ore tailings was lower than 2, and the REs and Nb leaching ratio no longer increased when the ratio of sulfuric acid to potash feldspar ore tailings was more than 2, which implied that the excess sulfuric acid did not improve the REs and Nb leaching. Fig. 3 showed that the REs and Nb leaching ratio increased with the roasting time extending, however, the REs and Nb leaching ratio increased slowly when the roasting time was longer than 2 h. Considering the energy consumption and cost, the roasting temperature, roasting time and sulfuric acid addition were chosen 300 °C, 2 h and 2 times quality sulfuric acid to potash feldspar ore tailings respectively.

3.2. The effect of leaching conditions on the REs and Nb leaching ratio

Leaching process was also important for the REs and Nb leaching, under fixed roasting conditions, the effect of leaching temperature, leaching time and the ratio of liquid to solid for the REs and Nb leaching rate were investigated, and the results



Fig. 1 The effect of roasting temperature on REs and Nb leaching ratio.



Mass ratio of sulfuric acid to potash feldspar ore benification tailings (g/g)

Fig. 2 The effect of sulfuric acid additon on REs and Nb leaching ratio.



Fig. 3 The effect of roasting time on REs and Nb leaching ratio.

were shown in the Fig. 4, Fig. 5 and Fig. 6 respectively. Fig. 4 showed that the leaching temperature increasing promoted the REs and Nb leaching ratio greatly when the leaching temperature was lower than 80 °C, however, continuously increasing temperature had little effect on the REs and Nb leaching ratio. Zhang, B et al. investigated the effect of the leaching temperature on the extraction ratio of REs and Nb elements from Bayan Obo tailings within 20 °C-90 °C (Zhang et al., 2014). And the results showed that the leaching ratios increased with increasing leaching temperature within 20 °C-60 °C, above 60 °C, the variation in the leaching ratios was insignificant, which was similar with the result of the effect of leaching temperature in this investigated. And the Fig. 5 showed that the REs and Nb leaching ratio increased with leaching time increasing within 0.5 h -2h, and above 2 h, the variation in the REs and Nb leaching ratios was insignificant. Fig. 6 showed that the REs and Nb leaching ratio increased with the increase of the liquid-solid ratio, and the leaching ratio increased insignificantly while the liquid-solid ratio was higher than 6. Liu, Y et al. studied the effect of liquid-solid ratio for recovering rare earth and niobium from rare metal ores within 3:1-10:1 (Liu et al., 2011). And the results showed that the leaching ratios of rare earth increased from 83.96% to



Fig. 4 The effect of leaching temperature on REs and Nb leaching ratio.



Fig. 5 The effect of leaching time on REs and Nb leaching ratio.



Fig. 6 The effect of liquid–solid ratio on REs and Nb leaching ratio.

85.03% with increasing liquid–solid ratio within 3:1-8:1, the general variation was similar with the result of the effect of liquid–solid ratio in this investigation. Therefore, the optimum leaching temperature, leaching time and liquid–solid ratio were chosen 80 °C, 2 h and 6 respectively.

At the optimum roasting and leaching conditions, the REs and Nb leaching ratio could reach to 89.9% and 80.6% respectively, and the major metal elements in leaching solution was shown in Table 3. As seen in the Table 3, the total amount of REs and Nb in leaching solution was about 692.36 mg/L and 218.75 mg/L, while the content of Fe³⁺, Al³⁺ and Ti⁴⁺ were 12.7 g/L, 6.78 g/L and 2.45 g/L, respectively.

3.3. Recovery of REs by fluoride precipitation method

The common REs precipitation method contains fluoride precipitation method and oxalate precipitation method etc (Zhang et al., 2014; Wang et al., 2000). And the fluoride precipitation method is applicable to the separation REs from the solution containing Nb because of the following Nb extraction step needed the fluoride ions. The HF addition is the key factor for the REs precipitation. The effect of HF addition on the RE precipitation rate was investigated and the results was shown in Fig. 7. Fig. 7 showed that the REs precipitation ratio increased with the HF addition increasing sharply when the HF condition was low than 2.5 mol/L. But, it should be noticed that the precipitate weight was increased with the HF addition increasing because of the AlF₃ which was slightly soluble in water would be precipitated, which would result in the content of REs in the precipitate dropping. So, the HF addition was chosen 1.25 mol/L, and the REs precipitation ratio could reach to 78.5%. The precipitate was washed with deionized water to neutral, then the precipitate was dried at 80 °C, and the rare earth fluoride (REF₃) with the purity 60.51% was obtained. The major impurities in the crude product REF₃ were potassium (K), aluminum (Al), titanium (Ti), silicon (Si) and calcium (Ca), the concentration of K, Al, Ti, Si and Ca were 19.44%, 2.25%, 1.71%, 1.54% and 2.27% respectively. The high potassium content was mainly due to the use of 10% KOH solution potassium for neutralization of the gel type rare earth fluoride precipitation.

3.4. Recovery of Nb by extraction

MIBK, TBP and n-octanol are the main extraction agents for niobium extraction (Yang et al., 2013; Zhu and Cheng, 2011; Bhattacharyya and Ganguly, 1984; El-Hazek et al., 2012). In this paper, the comparative experiments were done using TBP and MIBK as extractant for the Nb extraction, and the results was shown in Fig. 8. Fig. 8 showed that MIBK had



Fig. 7 The effect of HF addition on REs precipitation ratio.

higher Nb extraction ratio in this study, and the Nb extraction ratio increased sharply while the HF content was low than 2.5 mol/L, and then the Nb extraction ratio increased insignificantly with the addition more HF. The effects of extraction time on the Nb extraction ratio was studied, and the results was shown in Fig. 9. From the Fig. 9, it could be seen that MIBK extracted niobium quickly and would reached to equilibrium in 2 min. The extraction phase ratio of aqueous phase vs organic phase(A/O) was conducted, and the experiments result was shown in Fig. 10, which showed that the Nb extraction ratio decreased with the increase of A/O ratio, and the Nb extraction ratio reached to 87.2% while the A/O ratio was 1:1. In order to improve the extraction rate of niobium, two-stage countercurrent extraction was investigated, and the results showed that Nb extraction ratio could reach to 100% when the A/O was 4/1.

The stripping of Nb was carried out with sulfuric acid, the effect of sulfuric acid concentration on the stripping rate was shown in Fig. 11. Fig. 11 showed that the stripping rate of Nb was almost 100% when the sulfuric acid concentration was 0.5 mol/L.

3.5. Nb₂O₅ produciton

 Nb_2O_5 could be obtained by the method of ammonia precipitation and roasting (Pan, 1991).In this paper, the niobium was precipitated by adding ammonia water under room temperature with stirring, while the pH of the solution was adjusted to 8, the white precipitation was obtained, and the niobium was almost completely precipitated. Then, the white precipita-

Table 3 The major metal elements in leaching solution.									
Elements	Al	К	Si	Fe	SO_{4}^{2-}	Ti	Na		
Content(g/l)	6.78 Nb	4.01 Ta	0.085 PEs	12.70	177	2.15	0.078		
Content(mg/l)	218.75	0.81	692.36						



Fig. 8 The MIBK and TBP for the Nb extraction.



Fig. 9 The effect of extraction time for the Nb extraction.



Fig. 10 The effect of extraction phase ratio (A/O) for the Nb extraction.



Fig. 11 The effect of H_2SO_4 concentration for the Nb stripping.



Fig. 12 The process chart of recovery of REs and Nb from potash feldspar ore beneficiation tailings.

tion was calcined at 800°C for 3 h, the Nb₂O₅ product was obtained with the content of Nb₂O₅ is 87.61%. The major impurities in the crude product Nb₂O₅ were calcium (Ca) and titanium (Ti), the concentration of Ca and Ti were 4.86% and 0.26% respectively.

3.6. Presentation of a comprehensively recovery method

Based on the above-mentioned results, a hydrometallurgy process was presented for comprehensively recovery REs and Nb from potash feldspar ore tailings, as shown in Fig. 12. Firstly, the potash feldspar ore beneficiation tailing was decomposed by sulfuric acid roasting. Secondly, the decomposition sample was leached using water. Then, the rare earth was recovered as rare earth fluoride precipitation by HF acid precipitating, and the REF₃ with the purity 60.51% was obtained. The Nb was recovered by extraction and stripping method. Nb₂O₅ could be obtained by the method of ammonia precipitation and roasting, the Nb₂O₅ product was obtained with the content 87.61%. The total recovery rate of REs in this process could reach to 70.6%, and the total recovery rate of Nb could reach to 76.31%, so the REs and Nb could be recovered effectively from the potash feldspar ore tailings. The observed lower purity for REF₃ was attributed to significantly low concentration of REs in the leaching solution, which resulting in high impurities in the rare earth fluoride precipitation process. And using of 10% KOH solution potassium for neutralization of the gel type rare earth fluoride precipitation leaded to the high potassium content in the REF₃ product. The studies for the increasing the content of niobium and rare earth in the potash feldspar ore beneficiation tailings and exploring a better way for the neutralization of the gel type rare earth fluoride precipitation are ongoing, and the details of the overall design and optimization of the process will be reported in a future publication.

4. Conclusions

A feasible process was employed for recovering REs and Nb from potash feldspar ore beneficiation tailings, which was operated as follows.

- (1) By sulfating roasting at 280 °C and subsequent leaching at 80 °C, the 89.9% of REs and 80.6% of Nb were leached into the solution.
- (2) Under the condition of 1.25 mol/L hydrofluoric acid, REs was precipitated with the precipitation rate of 78.5%. At this time, titanium, aluminum and potassium almost do not precipitate.
- (3) The crude Nb₂O₅ products (87.61 wt% Nb₂O₅) and the composite RE fluoride (60.51 wt% REF₃) were obtained eventually. The total recovery rate of REs and Nb in this process reached up to 70.6% and 76.31% respectively.

CRediT authorship contribution statement

Wang Wei: Writing – original draft, Writing – review & editing. Liu Lin: Investigation. Liu Hongzhao: Writing – review & editing. Wang Hongliang: Investigation. Cui Hongmin: Writing – review & editing, Data curation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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