

Removal of Aluminum from Leaching Solution of Lepidolite by Adding Ammonium

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Lepidolite is a kind of low-grade lithium ore $(3.5-4.0\%\ \text{Li}_2\text{O})$ which contains more than 22% Al_2O_3 and 8% K_2O . Removal and utilization of the Al and K will be very important for the economic extraction of lithium from this ore. In this paper, a novel method is proposed to remove Al from the leaching solution of lepidolite by adding ammonium from $(NH_4)_2SO_4$. The results indicate that adding NH_4^+ could remove more aluminum and have a smaller loss of lithium in comparison to adding K^+ for removing aluminum in the leaching solution. The Al removal reached up to 80.44% at $(K^+ + NH_4^+)/Al^{3+}$ molar ratio of 0.95, while the amount of Li^+ (99.84%) still remained in the leaching solution. In addition, the phase equilibrium in the quaternary K_2SO_4 - $Al_2(SO_4)_3$ - $(NH_4)_2SO_4$ - H_2O system at 278.2 K was investigated to support the above conclusion. The results verify that the addition of ammonium could remove more Al^{3+} by easily forming potassium alum $(KAl(SO_4)_2\cdot12H_2O)$ and ammonium alum $(NH_4Al(SO_4)_2\cdot12H_2O)$.

INTRODUCTION

Lithium has been widely used for powering electric vehicles and portable electronic devices, and therefore the exploitation and utilization of lithium have encountered unprecedented challenges. 1-4 Nowadays, more than 80% of lithium produced in the world comes from brine.⁵ However, the global distribution of lithium brine is dramatically uneven,6,7 which may cause intense competition for lithium supplies due to the mass production of electric cars. ^{8–10} Therefore, developing the technology of extracting lithium from solid lithium ore will be important to meet the demand for lithium. Among the solid lithium minerals, pegmatite is still a dominant resource used for extracting lithium for the preparation of high-purity lithium carbonates. 11-13 Lepidolite, a type of pegmatite, has an ideal formula of KLi_{1.5}Al_{1.5}AlSi₃O₁₀F₂, ¹⁴ and its distribution is much wider than that of lithium brine. 15 However, the content of Li₂O in lepidolite is relatively low (3.5–4.0 wt.%) comparing with that of spodumene (5.5–6.0 wt.%). ¹⁶ Using traditional methods to extract lithium often incurs higher costs due to the inefficient utilization of other metals contained in the lepidolite. Furthermore, the high energy consumption for destroying the compact matrix of lepidolite in the extraction process limits its application. Thus, novel methods that are more effective and economic are required for industrial-scale application of lepidolite. In recent years, researchers have been seeking new ways to extract lithium from lepidolite, and some novel processes have been proposed, which can be primarily devided into acid methods and alkaline methods. ^{16–20} However, there seems to have been no discussion in the literature on the removal and recovery of Al and K during the process of lithium extraction from lepidolite.

In our previous study, 21 the fluorine chemical method, a kind of acid method, was proposed for extracting lithium from lepidolite. Firstly, H_2SO_4 and HF (generated from the reaction of H_2SO_4 and H_2SiF_6) were used to erode lepidolite. The reaction slurry was then roasted to release fluorine at $250^{\circ}C$ (523.15 K), and the residues after roasting were treated by water leaching that caused the elements of Li, Al, K, Rb and Cs in lepidolite to be converted

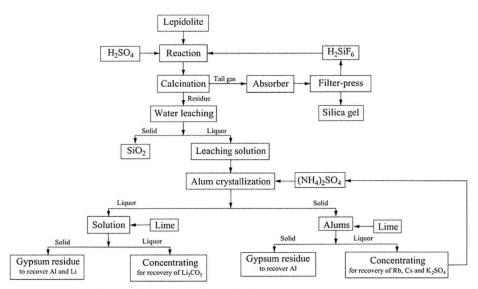


Fig. 1. Flow chart for the removal of aluminum and other procedures in lithium extraction from lepidolite in our pilot test.

into soluble sulfates in the leaching solution.²² This kind of novel acid method is comparable with extracting lithium from brine and is expected to ease the tension between the uneven distribution and explosive demand for lithium in the future.⁵ Nevertheless, these sulfates, especially Al₂(SO₄)₃, should be removed economically before lithium precipitation as the colloidal Al(OH)₃ crystals can firmly adsorb Li⁺ ions.²³ What is more, the comprehensive utilization of these sulfates after being removed from the leaching solution could enhance the profitability during the lithium extraction from lepidolite.

In the process of direct alum crystallization proposed in previous literature, aluminum could be removed by forming potassium alum (KAl $(SO_4)_2 \cdot 12H_2O)$ using a leaching solution that contains a significant amount of K⁺. ^{22, 24} However, the original K⁺/Al³⁺ molar ratio of the leaching solution in lithium extraction from lepidolite was 0.40, rather than the chemical stoichiometry 1:1 of potassium alum, which resulted in the aluminum not being removed completely by direct crystallization along with K^+ . The preliminary investigation indicated that the removal % of Al^{3+} increases with the addition of K2SO4. In this method, it also increased the possibility of forming double-salt KLiSO₄ which will decrease the lithium recovery. 24 Furthermore, the higher price of K₂SO₄ will increase the cost of lithium extraction from lepidolite. Therefore, other chemical additives should be considered to remove the remaining aluminum while simultaneously having no negative effect on lithium recovery.

In this study, a novel method is presented to remove aluminum by introducing ammonium from $(NH_4)_2SO_4$ into the leaching solution obtained in the process of lithium extraction from lepidolite, because $(NH_4)_2SO_4$ has more significant salting-out effect with $Al_2(SO_4)_3$ to form ammonium alum

NH₄Al(SO₄)₂·12H₂O.²⁵ At the same time, the K⁺, Rb+ and Cs+ in the leaching solution can also form corresponding alums with Al₂(SO₄)₃. After alum crystallization, the lithium in the liquor and the aluminum, potassium, rubidium, cesium and ammonium in the alums can be recovered in the following procedures.^{5,22} Figure 1 shows the flow chart of the removal of aluminum and other procedures in lithium extraction from lepidolite in our pilot test. Additionally, the phase equilibrium in the quaternary systems K₂SO₄-Al₂(SO₄)₃-(NH₄)₂SO₄-H₂O at 278.2 K was also investigated, which could theoretically verify the proposed method. This research could provide basic data and theory instruction for removal and recovery of aluminum and potassium from the leaching solution of lepidolite.

EXPERIMENTAL

Materials

The leaching solution used in this study was obtained after the procedures of water leaching which are part of the fluorine chemical method for extracting lithium from lepidolite in the pilot test (Fig. 1). The major components of the leaching solution are listed in Table I, and the primary materials are listed in Table II. The salts were dissolved by ultrapure water (electrical conductivity $< 0.055~\mu s~cm^{-1}$) in the addition process and phase equilibrium study.

Procedures and Methods

Addition Process of NH_4^+ and K^+

The leaching solution of lepidolite consisted mainly of $Al_2(SO_4)_3$, K_2SO_4 and Li_2SO_4 , while the Al^{3+} and K^+ can be removed by forming alums of $KAl(SO_4)_2\cdot 12H_2O$ and $NH_4Al(SO_4)_2\cdot 12H_2O$. The

Table I. Major components of the leaching solution of lepidolite

Component	$\underline{\text{Li}_2\text{SO}_4}$ $\underline{\text{Rb}_2\text{SO}_4}$		$\overline{ ext{Cs}_2 ext{SO}_4}$	$Al_2(SO_4)_3$	K_2SO_4	
Concentration (g/L) $c \pmod{L}$	$28.16 \\ 0.256$	$4.81 \\ 0.018$	$0.82 \\ 0.002$	$156.15 \\ 0.457$	$31.81 \\ 0.184$	

Table II. Main material descriptions

Materials	$\text{Li}_2 \text{SO}_4 \cdot \text{H}_2 \text{O}$	$\mathbf{Al_2(SO_4)_3}$ ·18 $\mathbf{H_2O}$	K_2SO_4	$(NH_4)_2SO_4$	
Purity (mass fraction)	≥99.0%	≥99.0%	≥99.0%	≥99.0%	
Supplied resource	a	a	a	a	

a Tianjin Fu Chen Chemical Reagents Factory, China.

solubility of Li_2SO_4 increases with a temperature decrease, while the solubility of $\text{Al}_2(\text{SO}_4)_3$, K_2SO_4 , $\text{KAl}(\text{SO}_4)_2$ and $\text{NH}_4\text{Al}(\text{SO}_4)_2$ decrease with a temperature decrease. Therefore, a lower temperature of the solution can enhance the removal of Al^{3+} and K^+ . Moreover, the hydrolysis of aluminum is an endothermic reaction, thus the lower temperature could inhibit the hydrolysis of Al^{3+} . Therefore, cooling was used for the forming of alums by considering the economic costs of power consumption, when the temperature of the solution was reduced from 333.2 K to 278.2 K.

In order to compare the effect of the addition of NH₄⁺ and K⁺ on aluminum removal in the leaching solution, various amounts of (NH₄)₂SO₄ and K₂SO₄ were added into the sample leaching solution (400 mL for each experiment). The solution was then cooled from the original temperature $(T_0 = 333.2 \text{ K})$ to terminal temperature $(T_{\rm t} = 278.2 \text{ K})$ by placing it into a shaking incubator (BS-100; Shanghai Boxun Industry and Commerce) with constant temperature of 278.2 K \pm 0.1 K and shaking frequency of 150-180 Hz. When the composition of the liquid phase remained unchanged, which meant that the equilibrium of alum crystallization was reached, the solution was filtrated. The concentrations of Li₂SO₄ and Al₂(SO₄)₃ in the filtrate were then measured. The reactions during crystallization process can be represented as:

$$M_2SO_4 + Al_2(SO_4)_3 + 12H_2O \rightarrow 2MAl(SO_4)_2 \cdot 12H_2O \downarrow$$

$$(1)$$

M stands for NH₄⁺, K⁺, Rb⁺ and Cs⁺.

Phase Equilibrium Investigation

In order to clearly reveal the actual process of alum formation in the leaching solution by adding ammonium, the phase equilibrium in the typical system of the solution K₂SO₄-Al₂(SO₄)₃-(NH₄)₂SO₄-

H₂O at 278.2 K was investigated. The known mass ratios of K₂SO₄, Al₂(SO₄)₃·18H₂O and (NH₄)₂SO₄ were partly dissolved into Erlenmeyer flasks, which were then placed into an incubator (BS-100) and shaken at 150-180 Hz to reach equilibrium. The equilibrium was not reached until the composition of the liquid phase remained unchanged. The temperature was regulated at 278.2 K \pm 0.1 K. The equilibrated liquid and solid were partially taken and weighed by a standard analytical balance (BS224S; Sartotius) with a 220-g capacity and a 0.0001-g resolution. A certain amount of the liquid (~5 mL) was immediately measured and diluted to a suitable concentration to determine the concentrations of Li+, Al3+, K+ and NH4+. The density data of the liquid phase were also measured with a gravity bottle. The partial residue was analysed by powder x-ray diffraction (XRD; MiniFilex II; Rigaku). In this way, a series of system points were obtained to represent the solubility of the K₂SO₄-Al₂(SO₄)₃-(NH₄)₂SO₄-H₂O quaternary system at 278.2 K.

Analytical Method

isothermal equilibrium method The employed to measure the solubility of the K₂SO₄- $Al_2(SO_4)_3$ - $(NH_4)_2SO_4$ - $H_2O.^{27}$ The contents of K⁺, Li⁺ and NH₄⁺ were determined by an ion chromatograph (CIC-260; Qingdao ShengHan Chromatography Technology), and the uncertainty was less than 1%. The K+ concentration was also determined by gravimetric methods using sodium tetrapuenyl borate. The results were compared with data obtained by the CIC-260 with a relative error of roughly 3%. The Al³⁺ was determined by ethylene diamine tetraacetic acid (EDTA) complexometric titration with an accuracy of less than 0.3%. The SO_4^{2-} was measured by gravimetric methods using BaCl₂. Each final value was an average of three measurements.

Table III. Suitable content of NH₄⁺ and K⁺ added for the alum crystallization

No.a	$n_1^{\mathbf{b}}$	$C_{\rm o} ({\rm g/L})^{\rm c}$	$n_2^{ m d}$	$Q_{\rm t}({ m g/L})^{ m e}$	Li recovery (%)	Al removal %
1	0.40	28.16	0.40	58.71	99.94	62.40
2	0.40	28.16	0.73	45.88	99.83	70.62
3	0.40	28.16	0.84	37.88	99.78	75.74
4	0.40	28.16	0.95	33.49	99.72	78.55
5	0.40	28.16	0.73	42.22	99.90	72.96
6	0.40	28.16	0.84	35.77	99.86	77.09
7	0.40	28.16	0.95	30.54	99.84	80.44

^a1 Blank experiment, without ammonium and potassium added. 2, 3, 4 $\rm K_2SO_4$ added into the leaching solution. 5, 6, 7 $\rm (NH_4)_2SO_4$ added into the leaching solution. $\rm ^{c}C_0$ is the original concentration of $\rm Li_2SO_4$. $\rm ^{d}n_2$ is the $\rm (K^++NH_4^+)/Al^{3+}$ molar ratio in leaching solution after the addition of $\rm NH_4^+$ or $\rm K^+$. $\rm ^{e}Q_t$ is the $\rm Al_2(SO_4)_3$ equivalent concentration of the filtrate after the leaching solution crystallized which was calculated as the product of the $\rm Al_2(SO_4)_3$ concentration of the filtrate and the ratio of the volume of solution after and before alums crystallization.

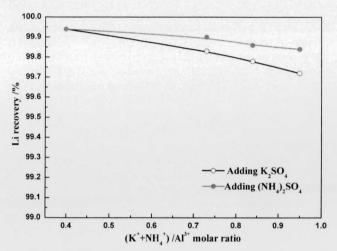


Fig. 2. Effect of the addition of NH₄⁺ and K⁺ on Li recovery.

85 80 75 80 65 60 0.4 0.5 0.6 0.7 0.8 0.9 1.0 (K'+NH, ')/Al'* molar ratio

Fig. 3. Effect of the addition of NH₄⁺ and K⁺ on Al removal.

RESULTS AND DISCUSSION

Effect of the Addition of NH₄⁺ on Li Recovery and Al Removal

The experimental results are presented in Table III and Figs. 2, 3. The initial concentration of $Al_2(SO_4)_3$ and pH value of the leaching solution was 156.15 g/L and -0.54, respectively. The alum was obtained after it was filtrated and aqueouswashed three times. The lithium contained in the filtrate is determined as Li recovery %, which represents the recovery of lithium during the alum crystallization process. The Al removal % was calculated to evaluate the efficiency of the removal of aluminum during the crystallization process.

Based on the results in Table III and Fig. 2, the Li recovery always reached above 99.5%, regardless of how much ammonium or potassium were added in the same conditions. Because the solubility of Li₂SO₄ increases when the temperature decreases, and Li₂SO₄ does not involve crystallization, ²⁶ therefore Li⁺ still existed in the solution and could be recovered as lithium salt in the following

procedures. The data only have small deviations for the entrainment loss of alum, and the Li recovery in the leaching solution with potassium addition is less than with ammonium for large amounts of K_2SO_4 thus increasing the formation possibility of double-salt $KLiSO_4$. Therefore, the proper ammonium addition could have no negative effect on the recovery of lithium.

From Table III and Fig. 3, we see that Al removal could reach 62.40% by alum crystallization, with the temperature reduced from 333.3 K to 278.2 K, and the Al removal generally increases with the addition of ammonium and potassium into the leaching solution. The Al removal of 78.55% was recorded when K_2SO_4 was added into the leaching solution at $(K^++NH_4^+)/Al^{3+}$ molar ratio of 0.95, while the removal of aluminum reached 80.44% when adding $(NH_4)_2SO_4$ with the same $(K^++NH_4^+)/Al^{3+}$ molar ratio. Therefore, the removal of aluminum with the ammonium addition is better than with potassium addition under the same conditions.

Table IV. Equilibrium data of the quaternary system K₂SO₄-Al₂(SO₄)₃-(NH₄)₂SO₄-H₂O at 278.2 K

	Jänecke index, J(b) ^a					Jänecke index, J(b)					
No.	J(K)	J(A1)	J(N)	J(H)	Solid phase ^b	No.c	$J(\mathbf{K})$	J(Al)	J(N)	J(H)	Solid phase
1,Q	0	65.32	34.68	209.99	NA + A	13,S	93.58	6.42	0	371.98	KA + KN
2	0.43	48.36	51.21	220.41	NA + A	14,P	0	13.02	86.98	420.87	NA + KN
3	1.76	42.67	55.57	248.37	NA + A	15,R	3.13	96.67	0	568.29	A + KA
4	13.09	20.66	66.25	305.85	NA + KA	16	5.68	67.32	27.00	540.21	A + KA
5	21.23	16.95	61.82	302.99	NA + KA	17	6.14	43.14	50.72	500.42	A + KA
6	30.73	13.57	55.70	321.44	NA + KA	$18,E_{2}$	8.54	28.64	62.82	473.87	A + KA + NA
7	60.71	4.46	34.83	425.03	NA + KA	19	15.98	6.18	77.84	262.12	NA + KA
$8,E_1$	62.10	3.37	34.53	309.12	KA + KN	20	27.48	6.01	66.51	252.13	NA + KA
9	66.73	3.21	30.06	321.53	KA + KN	21	33.98	5.83	60.19	267.13	NA + KA
10	70.86	3.72	25.42	331.95	KA + KN	22	38.75	5.12	56.13	274.22	NA + KA
11	73.53	4.05	22.42	336.12	KA + KN	23	46.35	4.52	49.13	281.14	NA + KA
12	84.77	5.14	10.09	342.52	KA + KN	24	48.59	4.80	46.61	295.31	NA + KA

 ^{a}J (b): Jänecke index of salt b. K $K_{2}SO_{4}$. N $(NH_{4})_{2}SO_{4}$. Al $Al_{2}(SO_{4})_{3}$. H $H_{2}O$. ^{b}A $Al_{2}(SO_{4})_{3}$ · $18H_{2}O$. NA $NH_{4}Al(SO_{4})_{2}$ · $12H_{2}O$. KA KAl $(SO_{4})_{2}$ · $12H_{2}O$. $^{c}E1$, E2 are co-saturated points of the quaternary system. O, P, Q, R are co-saturated points of the sub-ternary systems.

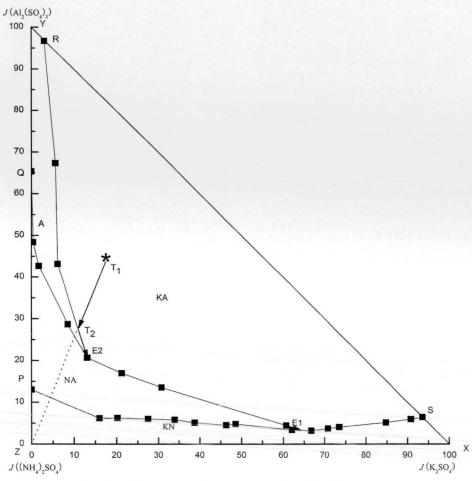


Fig. 4. Dry-salt diagram of the K_2SO_4 - $Al_2(SO_4)_3$ - $(NH_4)_2SO_4$ - H_2O quaternary system at 278.2 K.

Equilibrium in the Quaternary System K₂SO₄-Al₂(SO₄)₃-(NH₄)₂SO₄-H₂O at 278.2 K

The solubility data of the stable equilibrium in the K₂SO₄-Al₂(SO₄)₃-(NH₄)₂SO₄-H₂O quaternary system at 278.2 K are listed in Table IV. The solubility data of salt b are presented as J (b) (Jänecke index, g/100 g of dry salts). Figure 4 shows the stable phase diagram of the quaternary system.

The phase diagram (Fig. 4) of the quaternary system at 278.2 K presents the information of four crystallization regions, one single salt phase area, four univariant curves and two co-saturated points. YRE₂Q is the single salt phase Al₂(SO₄)₃·18H₂O. The crystallization regions of RE₂E₁S, QE₂E₁P and PZXS are KAl(SO₄)₂·12H₂O, $NH_4Al(SO_4)_2 \cdot 12H_2O$ and KNH_4SO_4 , respectively. Point T_1 (with symbol of *) is the original leaching solution under the $(K^+ + NH_4^+)/Al^{3+}$ molar ratio at 0.84. T₁T₂E2E1 is the liquid concentration curve for alum crystallization at isothermal conditions, which are located in the crystallization region of potassium alum ($KAl(SO_4)_2 \cdot 12H_2O$).

In Fig. 4, the crystalline regions of NH₄Al (SO₄)₂·12H₂O and KAl(SO₄)₂·12H₂O are larger than the crystallization areas of KNH4SO4 and Al₂(SO₄)₃·18H₂O. Thus, the solubility of NH₄Al (SO₄)₂·12H₂O and KAl(SO₄)₂·12H₂O are relatively small and can easily crystallize to remove more aluminum in the leaching solution of lepidolite. Moreover, the variation range of the mass fraction of Al₂(SO₄)₃ from E2 to E1 is larger for the saltingout effect with ammonium addition. The amount of ammonium could be added properly to change the solution composition along with E2E1. In this way, the concentration of Al₂(SO₄)₃ in the filtrate will be at a minimum based on the reverse principle of phase diagrams and the aluminum will be mostly removed by crystallization.

CONCLUSION

The addition of ammonium was demonstrated to be an efficient way to remove more aluminum in leaching solutions during the extraction of lithium from lepidolite. The removal of aluminum increased up to 80.44%, and the recovery of lithium simultaneously exceeded 99% when the (K++NH₄+)/Al³⁺ molar ratio was 0.95 in the leaching solution, which could enhance the profitability of lithium extraction from such low-grade lithium ore by the recovery of large amounts of aluminum to product valuable byproducts. The phase diagram of the K₂SO₄-Al₂(SO₄)₃-(NH₄)₂SO₄-H₂O quaternary system at 278.2 K verifies that aluminum could be removed mostly as ammonium alum (NH₄Al(SO₄)₂·12H₂O) for a remarkable salting-out effect of ammonium. Based on this study, a certain amount of ammonium

could dramatically accelerate the removal of aluminum and have no negative effect on the lithium recovery. Furthermore, the results can provide fundamental data for the addition of ammonium to remove aluminum in the leaching solution of lepidolite and other solutions with similar components.

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