



Enhanced lithium leaching from lepidolite in continuous tubular reactor using $\text{H}_2\text{SO}_4 + \text{H}_2\text{SiF}_6$ as lixiviant

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Abstract: An acidic mixture of sulfuric and fluosilicic acid ($\text{H}_2\text{SO}_4 + \text{H}_2\text{SiF}_6$) was employed as lixiviant to enhance leaching of lithium from lepidolite. The H_2SiF_6 was obtained as a byproduct of anhydrous hydrofluoric acid production, aiming to provide HF molecules. It was found that the HF molecules were the main reaction component and played a key role in strengthening the dissolution of lepidolite. Different factors, including mass ratio of ore/ $\text{H}_2\text{SO}_4/\text{H}_2\text{SiF}_6$, concentrations of H_2SO_4 and H_2SiF_6 , leaching temperatures (40–80 °C) and time (15–75 min), were investigated. Moreover, an efficient tubular reactor was employed to improve this acid leaching system. Under the optimal conditions (ore/ $\text{H}_2\text{SO}_4/\text{H}_2\text{SiF}_6$ mass ratio of 1:0.8:1.6, 80 wt.% H_2SO_4 , 15 wt.% H_2SiF_6 , 80 °C, 15 min), 97.9% of Li, 96.4% of K, 97.6% of Rb, 96.7% of Cs and 81.4% of Al (mass fraction) were leached. Additionally, a two-step thermal process was proposed to remove fluorine of leaching slurry. This acid treatment using an acidic mixture of H_2SO_4 and H_2SiF_6 in a continuous tubular reactor shows potential as an alternative process to extract lithium from lepidolite.

Key words: lepidolite; lithium extraction; continuous tubular reactor; fluosilicic acid; fluorine chemical method

1 Introduction

With the rapid development of electric vehicles and novel energy storage devices, the demand for lithium (Li) has increased significantly, especially related to wide application on lithium-ion batteries (LIBs) [1–3]. Currently, lithium is mostly extracted from lithium-containing brines due to its low production cost. However, these brines are predominantly located in South America, such as Argentina, Bolivia and Chile [4,5]. To diversify the lithium source is important for lithium production, especially for high purity Li_2CO_3 products. Therefore, Li-bearing minerals, including

spodumene (which has realized commercial production) [6,7], lepidolite [8–12], and zinnwaldite [13], have been treated as important Li resources owing to their relatively wide global distribution.

Lepidolite ($\text{KLi}_{1.5}\text{Al}_{1.5}[\text{AlSi}_3\text{O}_{10}]\text{F}_2$) has received increasing attention due to its large reservation, especially in China. Different processes have been proposed to extract Li from lepidolite, which can be divided into acid [8–10], alkaline [11] or sulfates methods [12]. The sulfuric acid method has been reported as an efficient process to treat lepidolite. However, the industrial process has not yet been economically feasible due to its typically low lithium content. The lepidolite employed in this

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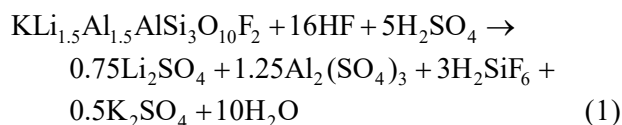
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study is obtained as flotation tailing of Ta and Nb ores from Yichun, Jiangxi province of China, resulting in a much more difficult process to extract lithium. Therefore, more effective methods need to be investigated to extract lithium from this typically low-grade lithium ore. Considering that 2–8 wt.% fluorine (F) is contained in lepidolite, an improved sulfuric acid process with the introduction of fluorine has been proposed to enhance the lithium extraction from lepidolite [14–16]. ROSALES et al [17] also reported an acid leaching using hydrofluoric acid (HF) to treat lepidolite. However, the lithium and other valuable elements were converted into insoluble fluorides, resulting in a much more difficult separation and purification process of Li products.

Previous investigation [18] shows that the HF molecules rather than F^- were the main reaction component involved in the acid treatment with introduction of F. The H^+ can accelerate the leaching via the protonation of the crystal lattices and convert the fluorides into soluble sulfates.

In our previous work, a fluorite (CaF_2)–sulfuric acid system has been employed to extract Li from α -spodumene [19]. Moreover, a mixture of sulfuric acid and hydrofluoric acid (H_2SO_4+HF) has been employed as lixiviant to leach Li from α -spodumene at 100 °C [20,21] and from lepidolite at 85 °C [14,15]. The leaching reaction between lepidolite and H_2SO_4+HF can be summarized as Reaction (1):



To diversify the fluorine source, H_2SiF_6 , a byproduct of anhydrous hydrofluoric acid production [22–24], was employed in this study to provide HF. The H_2SO_4 was introduced to convert the insoluble fluorides into soluble sulfates. The thermodynamic feasibility was first investigated that whether H_2SiF_6 can provide HF with H_2SO_4 . Preliminary experiments using only H_2SiF_6 as lixiviant were also performed to reveal reaction between lepidolite and H_2SiF_6 . Moreover, an efficient tubular reactor was employed to enhance the leaching of lithium from lepidolite. Subsequent heat treatment was then carried out for fluorine removal, which is important for further separation

and purification process.

2 Experimental

2.1 Materials

The lepidolite concentrate was obtained from Yichun, Jiangxi province of China. The ore sample was first ground using a jet mill and sieved to $<75 \mu m$ ($D_{50}=20.44 \mu m$, $D_{90}=55.86 \mu m$). The X-ray diffraction (XRD, MiniFlex II, Rigaku Co., Ltd.) analysis in Fig. 1 indicates that the ore sample mainly consists of lepidolite with some quartz and albite. The elemental analysis of the ore is given in Table 1.

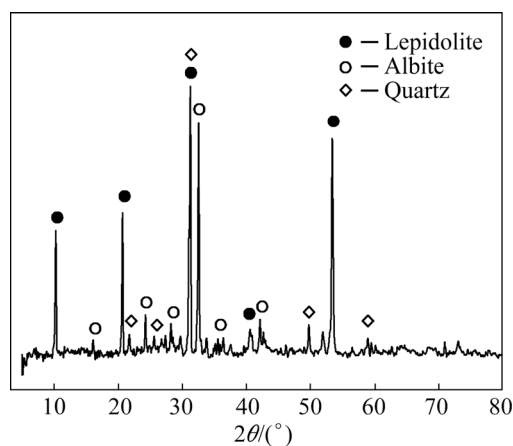


Fig. 1 XRD analysis of lepidolite ore

Table 1 Chemical analysis of lepidolite ore (wt.%)

Li ₂ O	K ₂ O	Al ₂ O ₃	SiO ₂	Rb ₂ O	Cs ₂ O	Na ₂ O	Fe ₂ O ₃	F
3.43	8.75	23.12	56.35	1.70	0.29	1.26	0.24	1.8

All chemicals used in this work were of analytical grade without further treatment. The hexafluorosilicic acid (H_2SiF_6 , 30 wt.%) was supplied by Xilong Scientific Co., Ltd., China. The concentrated sulfuric acid (H_2SO_4 , 98 wt.%) was supplied by Sinopharm Chemical Reagent Co., Ltd., China. Different concentrations of H_2SiF_6 and H_2SO_4 were prepared by diluting with predetermined amounts of deionized water.

2.2 Leaching process

The preliminary leaching experiments were firstly carried out in a closed polytetrafluoroethylene (PTFE) beaker. Different factors on leaching lithium were conducted: concentration of H_2SiF_6 (7.5–15 wt.%), H_2SiF_6 /ore mass ratio

(1.2–2.4), concentration of H_2SO_4 (50–90 wt.%), H_2SO_4 /ore mass ratio (0.6–1.0), leaching temperature (40–80 °C) and leaching time (15–75 min). Blank experiments using only H_2SiF_6 as lixiviant were also conducted. A tubular reactor (PTFE TR, inner diameter 8 mm, outer diameter 10 mm) was then employed to enhance this acid leaching system. The ore was first mixed with H_2SiF_6 and kept stirring continuously in a 1000 mL PTFE beaker. Then, the slurry of ore/ H_2SiF_6 and H_2SO_4 were simultaneously pumped into the tubular reactor with different mass ratios using two metering pumps (JLM1/10, Shanghai AQ Pump Co., Ltd., China). The resulted slurry was subsequently treated with the two-step heat treatment for fluorine removal as shown in Fig. 2. Then, the resulted slurry was washed using water/slurry ratio of 3:1 at 95 °C and stirred for 30 min. The obtained solution and residues were separated and analyzed, respectively.

2.3 Analytical methods

The leaching efficiency of lithium (L) was introduced to evaluate this enhanced acid leaching system as Eq. (2). Selective leaching of Li and leaching efficiency of valuable elements such as K, Al, Rb and Cs should also be considered for optimal conditions.

$$L = \frac{Q_L V}{m_{\text{ore}} w_{\text{Li, ore}}} \times 100\% \quad (2)$$

where Q_L is the lithium concentration in lixivium, g/L; V is the volume of lixivium, L; m_{ore} is the mass of ore sample, g; $w_{\text{Li, ore}}$ is the mass fraction of lithium in ore sample, %.

The lithium content was analyzed by atomic

absorption spectroscopy (AAS, AA-6800, Shimadzu). Other major elemental analyses of the liquid phase were analyzed by inductively-coupled plasma atomic emission spectrometry (ICP-AES, PS-6, Baird). Elemental content of solid samples was determined by X-ray fluorescence (XRF, model ZSX Primus II, Rigaku). The fluorine content was determined using a fluorine ion selective electrode (FISE, PF-1, LEICI). The utilization efficiency of fluorine (F_u , %) was calculated as Eq. (3) to determine the content of fluorine remained in residue out of that in original lepidolite sample. The removal efficiency of fluorine (F_r , %) during the heat treatment was calculated as Eq. (4):

$$F_u = \frac{F_{\text{ore}} - F_{\text{ins}}}{F_{\text{ore}}} \times 100\% \quad (3)$$

$$F_r = \frac{F_{\text{tot}} - F_{\text{ins}}}{F_{\text{H}_2\text{SiF}_6}} \times 100\% \quad (4)$$

where F_{ore} and F_{ins} are the masses of fluorine in ore and insoluble residue, respectively, g; F_{tot} is the total mass of fluorine in both ore and H_2SiF_6 added, g; $F_{\text{H}_2\text{SiF}_6}$ is the mass of fluorine in H_2SiF_6 added, g.

3 Results and discussion

3.1 Theoretical analysis

In our previous work, the feasibility using $\text{H}_2\text{SO}_4 + \text{HF}$ to extract lithium from lepidolite was discussed [14,15]. The introduced H^+ was mainly used to convert the fluorides into soluble sulfates. Here, H_2SiF_6 was employed as the substitute for hydrofluoric acid to provide more controllable HF molecules. Since HF is the main reaction

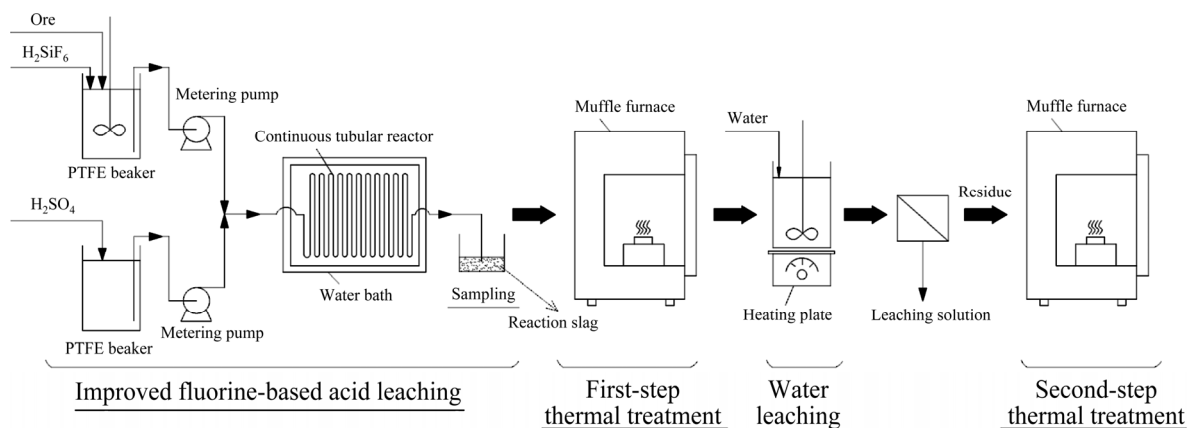
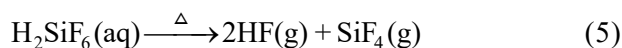


Fig. 2 Schematic diagram of leaching lithium from lepidolite using tubular reactor with $\text{H}_2\text{SO}_4 + \text{H}_2\text{SiF}_6$ as lixiviant

component involved in the leaching, the theoretical analysis that whether H_2SiF_6 can be used as the source of HF molecules was firstly carried out. The reaction of H_2SiF_6 to provide molecular HF occurred as



The thermodynamic analysis was calculated using HSC Chemistry software, 2006 version. Table 2 showed that the decomposition of H_2SiF_6 occurred spontaneously above 200 °C (473.15 K). Therefore, concentrated H_2SO_4 rather than diluted one was employed in this acid treatment, aiming to provide diluting energy for decomposition of H_2SiF_6 . This reaction also occurred in the manufacturing of HF [22–24].

Table 2 Thermodynamic analysis of decomposition of H_2SiF_6

T/K	$\Delta H/(\text{kJ}\cdot\text{mol}^{-1})$	$\Delta S/(\text{J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1})$	$\Delta G/(\text{kJ}\cdot\text{mol}^{-1})$
273.150	231.789	492.251	97.331
298.150	235.052	503.680	84.880
323.150	238.385	514.415	72.152
348.150	241.785	524.548	59.164
373.150	245.244	534.140	45.929
398.150	248.754	543.245	32.461
423.150	252.312	551.911	18.771
448.150	255.912	560.177	4.869
473.150	259.552	568.080	-9.235
498.150	263.228	575.650	-23.532
523.150	266.938	582.916	-38.015
548.150	270.678	589.901	-52.676
573.150	274.448	596.626	-67.508

3.2 Leaching with H_2SiF_6

The leaching results in Table 3 showed that the leaching temperature and leaching time slightly affected the leaching process under the investigated conditions. The XRD analyses of the insoluble residues in Fig. 3 showed that the diffraction peaks were basically same, indicating that no obvious reaction occurred between lepidolite and H_2SiF_6 at the leaching temperatures of 25–80 °C. Therefore, the lepidolite and H_2SiF_6 could be mixed and then pumped into the tubular reactor in future experiments.

Table 3 Factors and level of preliminary leaching experiment

No.	$w(\text{H}_2\text{SiF}_6)/\%$	$T/^\circ\text{C}$	t/min
1	20	25	15
2	20	25	90
3	20	80	15
4	20	80	90

$\text{H}_2\text{SiF}_6(\text{aq})/\text{ore mass ratio}=1.5$

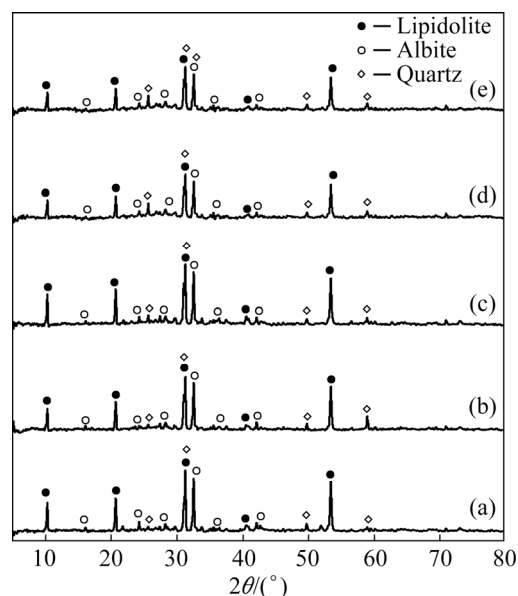


Fig. 3 XRD patterns of residues obtained from preliminary experiments: (a) Ore sample; (b) 25 °C, 15 min; (c) 25 °C, 90 min; (d) 80 °C, 15 min; (e) 80 °C, 90 min

3.3 Leaching with $\text{H}_2\text{SO}_4+\text{H}_2\text{SiF}_6$

Our previous investigation using $\text{H}_2\text{SO}_4+\text{HF}$ as lixiviant indicates that introducing fluorine showed a promising potential to extract lithium from lepidolite at a relative low temperature (85 °C) [14–16]. Here, H_2SiF_6 was used to provide HF. However, previous experiments showed that no obvious reaction occurred between lepidolite and H_2SiF_6 . Then, a mixture of concentrated H_2SO_4 and H_2SiF_6 was employed to leach lithium from lepidolite. Moreover, a tubular reactor was attempted to enhance this acid leaching of lithium. Effects of different factors on lithium leaching using $\text{H}_2\text{SO}_4+\text{H}_2\text{SiF}_6$ as lixiviant were investigated. Unless specifically explained, the leaching experiments were performed under the following conditions: ore/ $\text{H}_2\text{SO}_4/\text{H}_2\text{SiF}_6$ mass ratio of 1:0.8:1.6, H_2SiF_6 concentration of 15 wt.% and H_2SO_4 concentration of 70 wt.% at 50 °C for 15 min.

3.3.1 Effect of H_2SiF_6 concentration

The preliminary experiments showed that no obvious reaction occurred between lepidolite and H_2SiF_6 . However, the effect of H_2SiF_6 with the presence of concentrated H_2SO_4 needs to be investigated. Figure 4(a) showed that the lithium leaching efficiency increased from 93.5% to 98.0% with increasing the H_2SiF_6 concentration from 7.5 to 15 wt.%. The leaching efficiency decreased with

H_2SiF_6 concentration further increasing to 17.5 wt.%, which was caused by side reaction of more insoluble products like Li-F generated. To maximize the leaching efficiency of lithium, 15 wt.% H_2SiF_6 was chosen for further leaching experiments.

3.3.2 Effect of H_2SiF_6 /ore mass ratio

Along with the H_2SiF_6 concentration, the effect of H_2SiF_6 /ore mass ratio is also important for

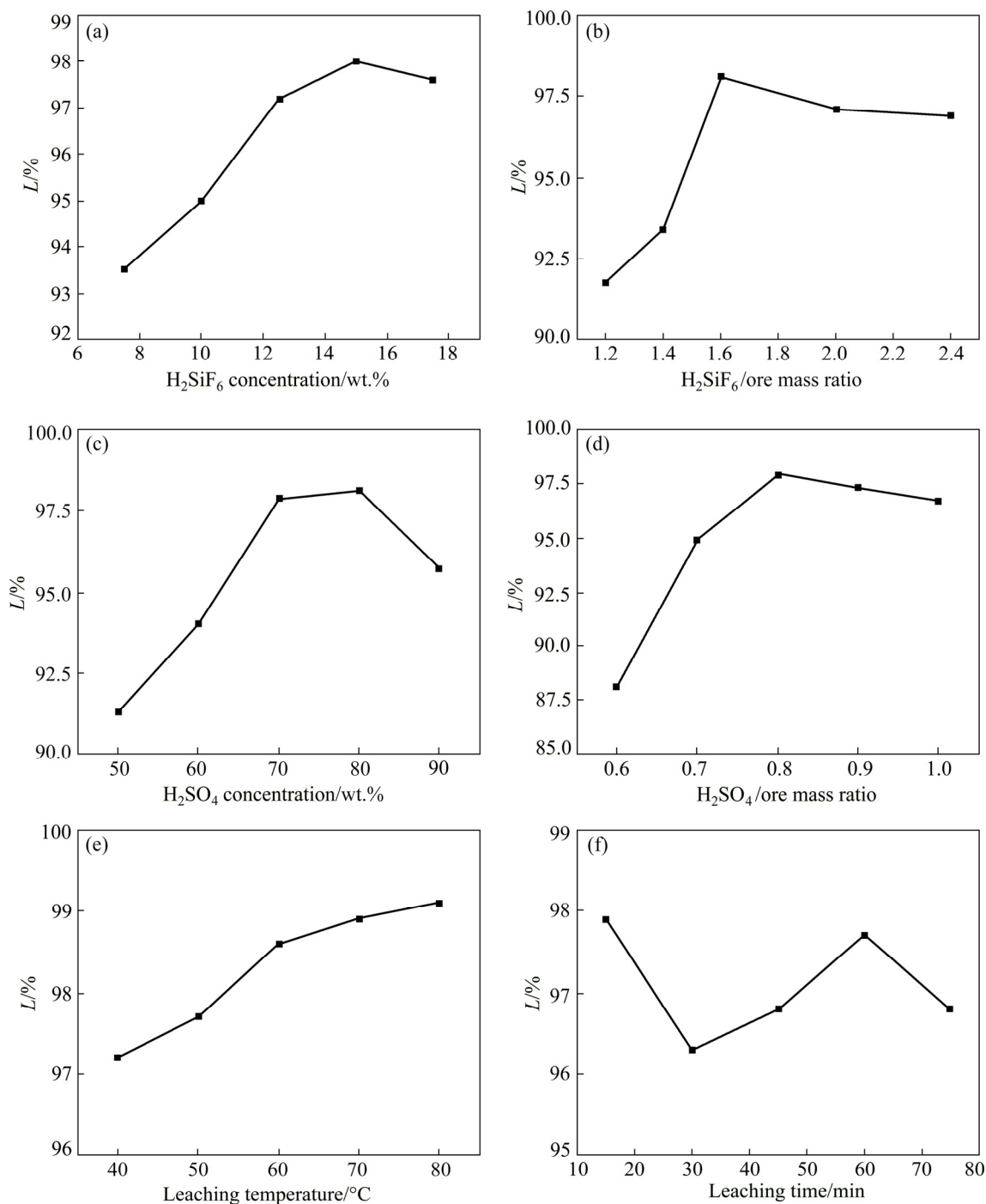


Fig. 4 Effects of different factors on leaching efficiency of Li: (a) H_2SiF_6 concentration; (b) H_2SiF_6 /ore mass ratio; (c) H_2SO_4 concentration; (d) H_2SO_4 /ore mass ratio; (e) Leaching temperature; (f) Leaching time

process optimization and utilization of fluorine. The results (Fig. 4(b)) indicated that leaching efficiency increased from 91.8% to 98.1% when the mass ratio of H_2SiF_6 (aq)/ore increased from 1.2 to 1.6. The leaching efficiency slightly decreased when the mass ratio of H_2SiF_6 (aq)/ore over 1.6. To avoid the formation of insoluble fluorides like Li-F , the mass ratio of H_2SiF_6 (aq)/ore of 1.6 was recommended for further experiments.

3.3.3 Effect of H_2SO_4 concentration

The extra energy supplied by diluting the concentrated H_2SO_4 could accelerate the reaction of H_2SiF_6 to release the HF molecules. However, the viscosity of concentrated H_2SO_4 is not beneficial for H^+ transport. Thus, effects of H_2SO_4 concentration (50–90 wt.%) were carried out. Figure 4(c) showed that the H_2SO_4 concentration had an obvious effect on leaching efficiency. The leaching efficiency increased by 7% when the H_2SO_4 concentration increased from 50 to 80 wt.%. While the leaching efficiency decreased with further increasing concentration over 80 wt.%. This could be attributed to the concentrated H_2SO_4 becoming too viscous for H^+ transport. To provide more input energy by diluting concentrated H_2SO_4 , the leaching experiments were performed with 80 wt.% H_2SO_4 .

3.3.4 Effect of H_2SO_4 /ore mass ratio

Figure 4(d) showed that the leaching efficiency increased considerably with increasing mass ratio of H_2SO_4 /ore, which reached a peak value of 97.9% at a ratio of 0.8. One reasonable explanation was that the concentrated H_2SO_4 released more heat to accelerate the decomposition of H_2SiF_6 to generate HF molecules, which is the main reaction component involved in the leaching. More importantly, the introduced H^+ converted insoluble fluorides like LiF ($K_{\text{sp}}=1.84\times 10^{-3}$) into soluble sulfates like Li_2SO_4 . Thus, the H_2SO_4 /ore mass ratio of 0.8 was considered as the optimal condition for further leaching experiments.

3.3.5 Effect of leaching temperature

Figure 4(e) indicated that the leaching temperature had a slight influence on the leaching of Li . The leaching efficiency increased from 97.2% to 99.1% with the temperature increasing from 40 to 80 °C. Thus, 80 °C was chosen for further leaching experiments, which was much lower than reported methods [8–12].

3.3.6 Effect of leaching time

The effect of leaching time using the tubular

reactor in Fig. 4(f) showed that the L reached 97.9% in 15 min, indicating a much shorter production cycle than reported methods. The traditional one usually consumed hours or even several days to achieve equivalent leaching efficiency [8–12,14]. This high efficiency could be owed to the negligible effect of back mixing using tubular reactor.

In summary, optimal conditions using the tubular reactor to enhance the leaching of Li from lepidolite are recommended as follows: ore/ H_2SO_4 / H_2SiF_6 mass ratio of 1:0.8:1.6, H_2SiF_6 concentration of 15 wt.%, H_2SO_4 concentration of 80 wt.% and leaching at 80 °C for 15 min.

3.4 Comparison among different reported acid methods

The sulfuric acid method has been considered as one of the most efficient processes to treat minerals. However, more efficient methods still need to be proposed due to the complex components and low Li_2O grade of lepidolite. Considering that 2–8 wt.% F in lepidolite and HF molecules can destroy silicate structure at much low temperature, and the fluorine additives were introduced to enhance the leaching of lithium from lepidolite recently. Comparison among reported acid methods is shown in Table 4.

The fine airtightness of the tubular reactor makes it more efficient to dissolve lepidolite. The efficient leaching of K , Al , Rb and Cs was also achieved as shown in Table 5, which was important for future utilization of lepidolite. The SEM images of the resulted residues in Fig. 5 showed that the crystal structure of lepidolite was destroyed. The results indicated that the introduction of F was a promising alternative process to accelerate the leaching of Li from lepidolite.

3.5 Heat treatment for fluorine removal

Based on the previous experiments, the leaching lithium from lepidolite was enhanced using $\text{H}_2\text{SO}_4+\text{H}_2\text{SiF}_6$ as lixiviant. However, the F remained in the acid leaching slurry should be removed for recycle use. Here, a two-step thermal process was proposed to remove fluorine of the resulted slurry.

3.5.1 First-step heat treatment of reaction slurry

The first-step heat treatment was conducted at 250 °C for 1 h using a PTFE reactor to remove the unreacted H_2SiF_6 . The leaching solution and

Table 4 Comparison among reported acid methods to extract lithium from lepidolite

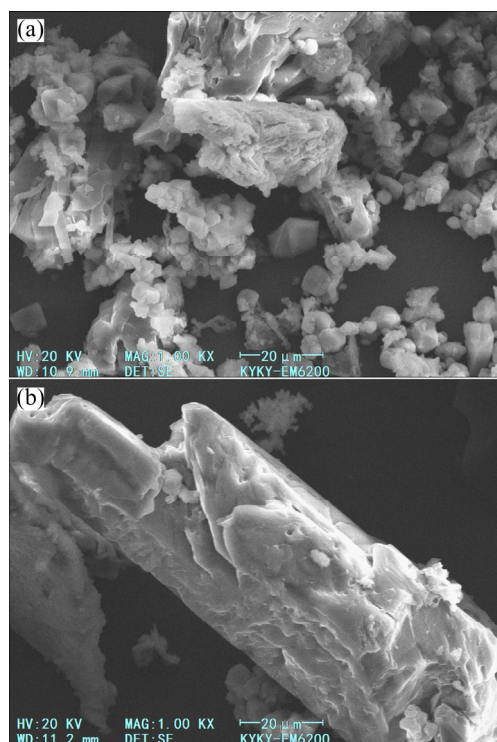
Method	Condition	Efficiency
Sulfuric acid (H_2SO_4)	Digested with concentrated H_2SO_4 at $\sim 300^\circ\text{C}$, 4 h	$> 90\%$ of Li recovered [5]
Hydrofluoric acid (HF)	123°C , 7 vol.% HF, 120 min	92% of Li recovered as LiF [17]
Hydrofluoric acid + sulfuric acid ($\text{HF}+\text{H}_2\text{SO}_4$) using stirred tank reactor	85°C , analytical pure HF, 3 h	98.6% of Li leached [14,15]
Fluorosilicic acid + sulfuric acid ($\text{H}_2\text{SiF}_6+\text{H}_2\text{SO}_4$) using stirred tank reactor	15 wt.% H_2SiF_6 , 80°C , 15 min	75.3% of Li leached
Fluorosilicic acid + sulfuric acid ($\text{H}_2\text{SiF}_6+\text{H}_2\text{SO}_4$) using tubular reactor	15 wt.% H_2SiF_6 , 80°C , 15 min	97.9% of Li leached

Table 5 Leaching efficiency with $\text{H}_2\text{SiF}_6 + \text{H}_2\text{SO}_4$ using tubular reactor (%)

Li	K	Rb	Cs	Al
97.9	96.4	97.6	96.7	81.4

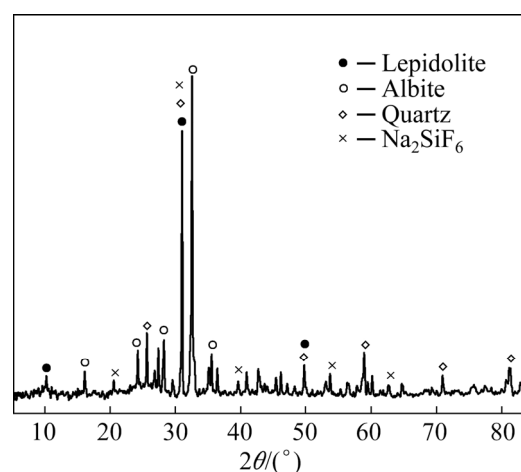
Table 6 Elemental analyses of insoluble residues after first-step heat treatment (wt.%)

Li	O	F	Na	K	S	Si	Al
0.079	45.53	4.72	0.96	0.52	7.32	31.23	4.12

**Fig. 5** SEM images of resulted insoluble residues after acid leaching using $\text{H}_2\text{SO}_4+\text{H}_2\text{SiF}_6$: (a) Generated insoluble substance; (b) Unreacted ore

corresponding insoluble residues were analyzed. The chemical analysis in Table 6 showed that there was still 4.72 wt.% fluorine in insoluble residues. Then, the second-step heat treatment was introduced for further fluorine removal of residues.

The XRD analysis (Fig. 6) indicated that the fluorine in residues mainly existed as Na_2SiF_6 . Combined with elemental analysis in Table 6, the insoluble residues were mainly Na_2SiF_6 and SiO_2 ,

**Fig. 6** XRD pattern of insoluble residue obtained after water leaching

which can be further utilized as white carbon black. Moreover, the quartz (SiO_2) and albite ($\text{NaAlSi}_3\text{O}_8$) were still present in insoluble residues, indicating that a selective leaching of Li was achieved under the optimal conditions.

3.5.2 Second-step heat treatment of insoluble residues

About 4.7 wt.% fluorine still remained in the insoluble residues. Then, the second-step heat treatment for 1 h was conducted for further fluorine removal. Effect of heating temperature on fluorine removal was investigated. Table 7 showed that the F content decreased with increasing temperature, which could be attributed to the decomposition of the generated fluorides like Na_2SiF_6 . When the temperature above 400°C , the $F_u > 0$ and $F_r > 100\%$,

respectively, indicating that the fluorine in H_2SiF_6 was completely removed and even the fluorine in lepidolite was removed. When the heating temperature reached 500 °C, 68.7 wt.% of fluorine in the ore was removed.

Table 7 Effect of temperature of second-step heat treatment on F removal

Temperature/°C	Fluorine content/%	$F_{\text{u}}/\%$ *	$F_{\text{r}}/\%$
100	4.23	−41.0	96.1
200	4.06	−35.4	96.6
300	3.93	−31.0	97.1
400	2.75	8.3	100.8
500	0.94	68.7	106.5

* Negative values mean that the fluorine removal of lepidolite is slower than that of residues, and positive values mean that the fluorine in lepidolite is even removed.

The second-step heat treatment at 500 °C resulted in an obvious decrease of the fluorine in residues from 4.72 wt.% to 0.94 wt.%, indicating that the fluorine in the insoluble residues or even in the lepidolite was removed, which was beneficial for downstream fluorine recovery or recycle use. Thus, an effective acid leaching of Li from lepidolite with fluorine additives was preliminarily set up using $\text{H}_2\text{SO}_4+\text{H}_2\text{SiF}_6$ as lixiviant.

4 Conclusions

(1) About 97 wt.% Li and 90 wt.% of K, Rb and Cs were leached using $\text{H}_2\text{SO}_4+\text{H}_2\text{SiF}_6$ as lixiviant at 80 °C for 15 min. Selective leaching of Li was achieved due to the fact that the lepidolite showed dissolving priority over quartz and albite.

(2) The fluosilicic acid (H_2SiF_6) was used to provide more controllable HF molecules by reacting with H_2SO_4 (80 wt.% in this study) since no obvious reaction occurred between lepidolite and H_2SiF_6 .

(3) The fluorine in insoluble residues, which mainly existed as Na_2SiF_6 was significantly decreased from 4.72 to 0.94 wt.% by stepwise heat treatment. The fluorine in lepidolite was also even removed. The tubular reactor can provide an alternative scheme to enhance the leaching of lithium from lepidolite.

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锂云母连续管式反应器中混酸 H₂SO₄+H₂SiF₆ 体系强化溶出锂

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摘 要: 采用硫酸(H₂SO₄)和氟硅酸(H₂SiF₆)的混酸浸出剂强化锂云母中锂的浸出。氟硅酸(H₂SiF₆)作为氢氟酸的副产物, 可以提供反应所需的氟化氢(HF)分子。而氟化氢是氟硅酸实际参与反应的主要成分, 对锂的强化溶出具有关键作用。探究矿石/硫酸/氟硅酸质量比、硫酸和氟硅酸浓度、浸出温度(40~80 °C)、浸出时间(15~75 min)等因素对锂浸出的影响。采用连续管式反应器进一步强化酸浸体系。确定酸浸最优条件为: 矿石/H₂SO₄/H₂SiF₆ 质量比 1:0.8:1.6、H₂SO₄ 浓度 80%(质量分数)、H₂SiF₆ 浓度 15%(质量分数), 97.9%的锂、96.4%的钾、97.6%的铷、96.7%的铯和 81.4%的铝(质量分数)在 80 °C 下仅需反应 15 min 即可得到有效浸出。此外, 提出两步热处理工艺对反应浆料进行脱氟。研究表明, 采用管式反应器加含氟混酸浸出剂(H₂SO₄+H₂SiF₆)可实现锂云母的高效提锂。

关键词: 锂云母; 提锂; 连续管式反应器; 氟硅酸; 氟化学法

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