



Intensification of potassium leaching from phosphorus-potassium associated ore with lauryl alcohol



Jun-Feng Zhou^a, Cun-Cheng Liu^a, Jia-Yu Ma^a, Yuan-Hang Qin^{a,*}, Zai-Kun Wu^b, Li Yang^b, Tie-Lin Wang^b, Wei-Guo Wang^b, Cun-Wen Wang^{b,*}

^a Key Laboratory of Green Chemical Process of Ministry of Education, School of Chemical Engineering and Pharmacy, Wuhan Institute of Technology, Wuhan 430205, China

^b Key Laboratory of Novel Reactor and Green Chemical Technology of Hubei Province, School of Chemical Engineering and Pharmacy, Wuhan Institute of Technology, Wuhan 430205, China

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ABSTRACT

In the present work, experiments have been performed for leaching potassium from phosphorus-potassium associated ore in hydrochloric acid/fluorite system. Surfactant or phosphoric acid extractant was added into the leaching system to enhance the leaching efficiency. The results show that alcohol chain length has great influence on potassium leaching and among the additives investigated, lauryl alcohol exhibits the largest promoting effect on potassium leaching and the leaching fraction of potassium can be increased from 89.5% to 97.8% with the addition of 1 mL of lauryl alcohol into 80 mL of leaching solution. The enhanced leaching observed in the presence of lauryl alcohol can be attributed to the hydrophobicity mechanism. The kinetic analysis shows that there are two distinct stages in the leaching process and the kinetics of both stages follows the classical shrinking core model with chemical reaction being the rate-controlling step.

1. Introduction

Nitrogen (N), phosphorus (P) and potassium (K) are the three essential nutrients for plant growth and crop production [1,2]. Potassium plays important roles in plant cellular homeostasis by contributing to charge balance, osmotic adjustment and enzyme catalysis [3]. Unfortunately, the soluble potassium resources for potassium fertilizer use are severely deficient in China. Insoluble potassium resources, mainly in the form of potash feldspar (KAlSi_3O_8), are rich in China and the exploration of these insoluble potassium resources for potassium fertilizer use is of great significance.

Recently, 0.8 billion tons of phosphorus-potassium associated ore containing 5–12% P_2O_5 , 5–10% K_2O and 45–55% SiO_2 was discovered in Yiling district of Hubei Province, China. The ore mainly consists of fluorapatite, potash feldspar, quartz, pyrite and dolomite. The separation of potassium (potash feldspar) and phosphorus (fluorapatite) in the ore by the conventional flotation method to produce K fertilizer and P fertilizer is not economically viable because of the low floatation efficiency and low phosphorus content. Considering the natural symbiotic association of phosphorus and potassium in the ore, the comprehensive use of phosphorus and potassium for preparing PK fertilizer has more economic advantages [4].

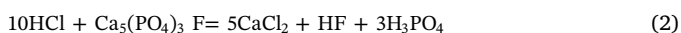
The biggest obstacle limiting the economic utilization of potash feldspar is the extremely stable Al-Si-O tetrahedron structure of potash feldspar, whose efficient decomposition determines whether potassium can be efficiently extracted. Over the past decades, many processes, including roasting immersion process [5,6], low-temperature acid leaching process [7–9], microbial decomposition process [10], high-temperature calcination process [11–14], hydrothermal process [15], and milling-assisted ion exchange and decomposition process [16] have been developed to extract potassium from potash feldspar. However, high energy consumption and/or low leaching fraction of potassium are still the critical issues need to be addressed.

In order to decrease the energy consumption and improve potassium leaching, substantial research effort has been devoted to intensifying the low-temperature acid process, which uses HF to decompose potash feldspar for potassium leaching and has the merits of relatively low energy consumption and high leaching fraction of potassium. In our previous work, ultrasound was used to intensify the low-temperature leaching process and the leaching fraction of potassium was increased from 87% in the absence of ultrasound to 94% in the presence of ultrasound [4]. However, the energy consumption of the ultrasound-assisted leaching process is relatively high and other intensifying methods need to be explored.

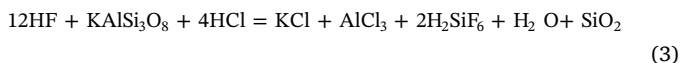
* Corresponding authors.

E-mail addresses: qyhsir@qq.com (Y.-H. Qin), wangcw0120@163.com (C.-W. Wang).

The decomposition of potash feldspar by low-temperature hydrochloric acid process proceeds through two consecutive steps. In the first step, HF was generated through the reaction of HCl with fluorite (CaF₂) and fluorapatite in the phosphorus-potassium associated ore.



In the second step, soluble potassium was obtained through the reaction of HF with potash feldspar in the phosphorus-potassium associated ore.



It can be seen from Eq. (3) that the generated SiO₂ may precipitate on the surface of unreacted potash feldspar and impede its decomposition. One possible way to alleviate the precipitation is to make the generated SiO₂ hydrophobic. In addition, the phosphoric acid generated by the reaction of HCl and fluorapatite (Eq. (2)) could react with Ca²⁺ and Mg²⁺ (from fluorapatite, fluorite and dolomite) to generate phosphate salt precipitations on the surface of potash feldspar and impede the leaching of potassium. One possible way to alleviate the precipitation is to extract the generated phosphoric acid from the aqueous phase during the leaching process so as to prevent the generation of phosphate salt precipitations.

In this work, low-temperature hydrochloric acid process was used to extract potassium from the phosphorus-potassium associated ore. Two kinds of additives, surfactant and phosphoric acid extractant, were respectively added into the leaching media with the aim to improve the leaching fraction of potassium. Surfactant such as sodium dodecyl sulfate (SDS) was expected to improve potassium leaching by adsorbing onto the surface of SiO₂ to improve its hydrophobicity. Phosphoric acid extractant such as butanol (C-4), n-amyl alcohol (C-5) and n-heptanol (C-7) was expected to improve potassium leaching by extracting phosphoric acid from the aqueous phase.

2. Materials and methods

2.1. Materials

Phosphorus-potassium associated ore used in the experiment was obtained from Yiling district of Hubei Province, China. The chemical composition of this ore analyzed by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES, IRIS Advantage Radial, Thermo Fisher Scientific) is shown in Table 1.

The characterization of the mineralogical composition of this ore via X-ray Diffraction (XRD, D8 Advance, Bruker) reveals that potassium and phosphorus are existed mainly in the form of potash feldspar and fluorapatite, respectively (Fig. 1). Calcium fluoride (CaF₂), hydrochloric acid (HCl, 37 wt%), butanol (C-4), n-amyl alcohol (C-5), n-heptanol (C-7), butyl carbitol (BC), lauryl alcohol (C-12), tetradecanol (C-14), cetanol (C-16), tri-n-butyl phosphate (TBP), di(2-ethylhexyl) phosphoric acid (D2EHPA), and sodium dodecyl sulfate (SDS) of analytical grade were supplied by Sinopharm Chemical Reagent Co., Ltd.

Table 1
The chemical composition of phosphorus-potassium associated ore (wt.%).

| Composition | Content (wt.%) | Composition | Content (wt.%) |
|--------------------------------|----------------|--------------------------------|----------------|
| SiO ₂ | 51.46 | MnO | 0.03 |
| Al ₂ O ₃ | 13.85 | CO ₂ | 0.90 |
| K ₂ O | 9.33 | F | 0.45 |
| CaO | 7.85 | MgO | 0.98 |
| P ₂ O ₅ | 5.85 | SO ₃ | 4.70 |
| Na ₂ O | 0.23 | BaO | 0.25 |
| Fe ₂ O ₃ | 3.32 | Cr ₂ O ₃ | 0.02 |
| TiO ₂ | 0.85 | Cl | 0.04 |

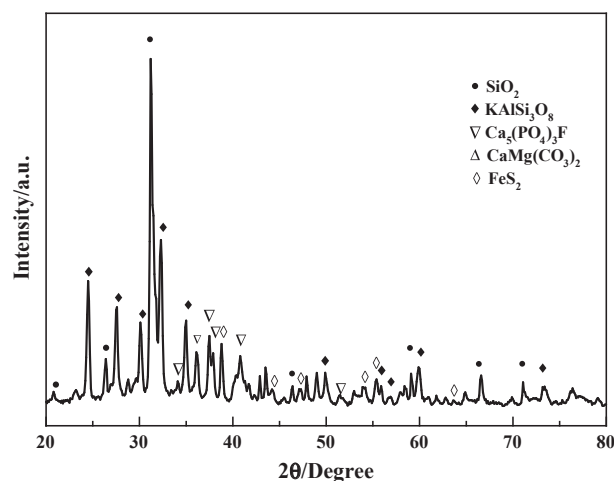


Fig. 1. XRD pattern of phosphorus-potassium associated ore.

2.2. Experimental procedure

Phosphorus-potassium associated ore was crushed and grinded to pass an 80-mesh sieve. In a typical leaching experiment, ore (20 g) and CaF₂ (4 g) were thoroughly mixed and then added into a preheated solution of hydrochloric acid (22.5 wt%, 80 mL) and extractant/surfactant (1 mL or 1 g) contained in a three-necked round-bottom glass flask (250 mL) equipped with a magnetic stir bar (200 rpm), a reflux condenser and a thermometer with an accuracy of ± 0.1 °C. To investigate the effect of reaction temperature on potassium leaching, the reaction media were preheated to the desired temperature (60–90 °C). During the leaching period, 1 mL of the medium was taken out at selected time intervals by using a syringe with a 0.22-μm membrane to determine the leaching fraction of potassium, which was performed by an Atomic Absorption Spectrophotometry (AAS, 4530 F, Shanghai Precision and Scientific Instrument Co., Ltd., China). After 6 h of leaching, the slurry was immediately filtered, and the resultant filtrate was analyzed by AAS and the filter residue was washed, dried and then subjected to XRD, Scanning Electron Microscopy (SEM, JSM-5510LV, JEOL), X-ray Fluorescence Spectrometry (XRF, EDX-8000, Shimadzu) and wettability analysis, which was performed by dispensing a water droplet of 4 μL onto the surface of residue and the contact angle was measured with a KRÜSSDSA100 contact angle system. The leaching fraction of potassium (*x*) was calculated based on the following equation,

$$x = m_o/m_i \quad (4)$$

where *m_i* is the mass of potassium in the ore, and *m_o* is the mass of potassium in the filtrate. All *x* data presented in this work were the average value of three repeated analysis of samples.

3. Results and discussion

3.1. Effect of surfactant/extractant addition on potassium leaching

The popular surfactant SDS with the aim to make the generated SiO₂ hydrophobic and the low-cost alcohol extractant C-4, C-5 or C-7 with the aim to extract phosphoric acid from the leaching solution were respectively added to the leaching system consisting of 20 g of ore, 4 g of CaF₂, and 80 mL of hydrochloric acid (22.5 wt%). The reaction temperature was controlled at 90 °C and after 6 h of reaction, the leaching fractions of potassium with and without additives were determined and the results are shown in Fig. 2.

It can be seen that the surfactant SDS has positive effect while the alcohol extractants have negative effect on potassium leaching. The low leaching fraction of potassium with the addition of extractant may

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