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Microwave-assisted Decomposition of K-feldspar at Low Temperature in Acids System

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Abstract

Extraction of potassium from K-feldspar under microwave-assisted at low temperature was studied in this paper. The mineralogical characteristics of raw K-feldspar and residues after microwave pre-treatment and reaction under different conditions were determined by X-ray diffraction (XRD), scanning electron microscopy (SEM). The results showed that under the same experimental conditions, microwave power had significant effects on K-feldspar characteristics. There was a clear change in peak shape when the power was 600W or higher. Potassium dissolution efficiency by microwave pretreatment of feldspar was similar to microwave digestion reaction process. The potassium dissolution efficiency was as high as 80% in microwave-assisted process under conditions such as: K-feldspar and phosphorite was 0.8:1, calcium phosphate and calcium fluoride mass ratio of 1:3, temperature 160°C, reaction time 150 min. Microwave-assisted extraction method was demonstrated to be an excellent alternative for short reaction time and higher extraction efficiency.

1. Introduction

K-feldspar (KAlSi_3O_8), a valuable insoluble potash ore, is widely used in many applications, such as wastewater treatment [1], carbon dioxide capture [2, 3] and preparation of various potassium compounds [4]. One of the most attractive characteristics of K-feldspar is its higher content of K_2O which can be used in agriculture [5, 6]. However, plants can not directly absorb insoluble potash ores, research of extracting potassium from insoluble feldspar has potential economic value and social benefits. For converting insoluble potassium into soluble potassium can alleviate the current shortage of potash status. In recent years, several authors have investigated the feasibility of potassium extraction from K-feldspar mainly including pressure heat method, high temperature decomposition, low temperature decomposition and microbial decomposition method [7-9]. K-feldspar is difficult to be decomposed because of the frame-like structure of aluminum silicate with the characteristics of high chemical stability. Therefore, the process of extracting potassium often requires high temperature and pressure conditions which may significantly increase the chemical costs and energy consumption. In order to destabilize the structural of K-feldspar in relatively low temperature, acid-extractable and alkali solution were often chosen to extract potassium [9-11]. Su et al. [12] studied the hydrothermal stability of microcline in KOH and NaOH solutions, and successfully synthesized zeolite A and kalsilite using the Si-Al components from K-feldspar.

Hydrothermal decomposition of potassium feldspar was often carried out in 200~250°C. Compared with alkali system, acid system had more advantages such as lower temperature, significantly reducing energy consumption. The use of microwave has gained a lot of attention in recent times because of its unique characteristics such as rapid and uniform heating, quick reaction times, low cost and energy saving [13-14]. Microwave-assisted chemistry has emerged as a rapidly growing field for the preparation of inorganic materials [15].

Although there are some studies on potassium extraction from K-feldspar at low temperature in acids systems, there have been limited studies on microwave-assisted extraction method. The aim of this research was to investigate the advantages of microwave on potassium extraction process. Microwave pretreatment and extraction reaction processes were carried out and the effects of microwave power and temperature on potassium extracting efficiency were also assessed. In addition, mineralogical characteristics of pretreated potassium feldspar and residues after reactions were analyzed.

2. Materials and Method

2.1. Materials

The raw K-feldspar used in this work was obtained from Wenxi County in Shanxi province, China. The chemical composition of the K-feldspar was analyzed by X-ray fluorescence spectrometer (Bruker AXS, S8TIGER, Germany). In Table 1, content of K₂O was 13.41% and content of K was calculated as 11.13%. Through crushing and screening, particles of potassium feldspar were between 100 to 500 mesh. Calcium phosphate, sulfuric acid, calcium fluoride and deionized water were used for potassium extraction. All reagents used were of analytical grade.

Table 1. Compositions of K-feldspar.

Composition	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	Na ₂ O	K ₂ O
Content (%)	63.23	17.70	0.355	0.394	3.18	13.41

2.2. The Microwave Pretreatment and Extraction Reaction Processes

The potassium feldspar was pretreated by microwave radiation (2450MHZ) with microwave power 300-700W for 15 min at different particle size. The mixture of 0.8g K-feldspar, 0.25g calcium phosphate and 0.75g calcium fluoride, 10ml sulfuric acid (70%) were placed in a Teflon chamber (100ml) fitted into a stainless steel pressure vessel and heated in an oven from 100 to 180°C. As a comparison in the experiment by microwave digestion reaction, K-feldspar without pretreatment and reactants were mixed together and placed in microwave digestion system supplied by Italy MILESTONE model ETHOS. After reaction from 60min to 300min, products were cooled and taken out and then diluted

to 1L volumetric flask. The residue was filtered through filter paper and natural drying for mineral identification and morphology analysis. Mineral identification was carried out by power X-ray diffraction (XRD) with CuKα radiation (D/MAX-Ultima IV, Japan). Morphology of K-feldspar and residues was observed by scanning electron microscopy (SEM FEI, Quanta 200, Czech).

2.3. Dissolution of K-feldspar

The dissolution of potassium ions was analyzed with an ICS-1500 (Dionex, USA) ion chromatography system. The potassium dissolution rate (%) was calculated according to the following formula:

$$N = \frac{CV}{1000M \times 11.13\%}$$

Where C (mg/L) represents the measured potassium ion concentration, V (L) represents the constant volume and M (g) is the quantity of potassium feldspar.

3. Results and Discussion

3.1. Effects of Microwave Pretreatment on Potassium Feldspar Characteristics

To investigate the effects of microwave power on potassium feldspar characteristics, the potassium feldspar was pretreated by microwave radiation with power from 300W to 700W for 15 min with particle size 100 mesh. Figure 1 showed XRD patterns of potassium feldspar and microwave treated potassium feldspar. The results of XRD proved the diffraction peaks changed significantly when microwave power increased 600W or higher. Figure 2 showed effects of potassium feldspar XRD patterns under microwave 600W in different particle sizes. From this experiment, it can be seen that the effects of microwave pretreatment on potassium feldspar particle size becomes less significantly.

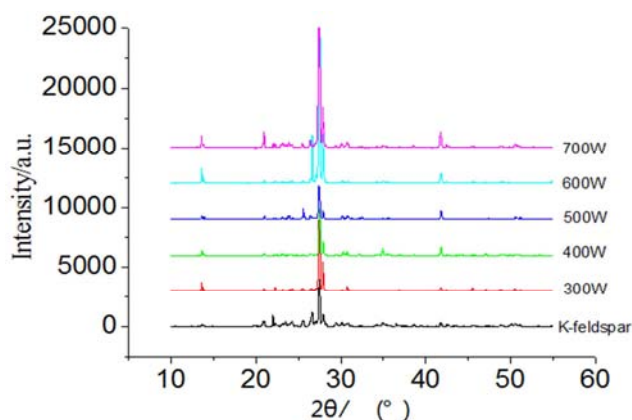


Figure 1. XRD patterns of K-feldspar in different microwave powers.

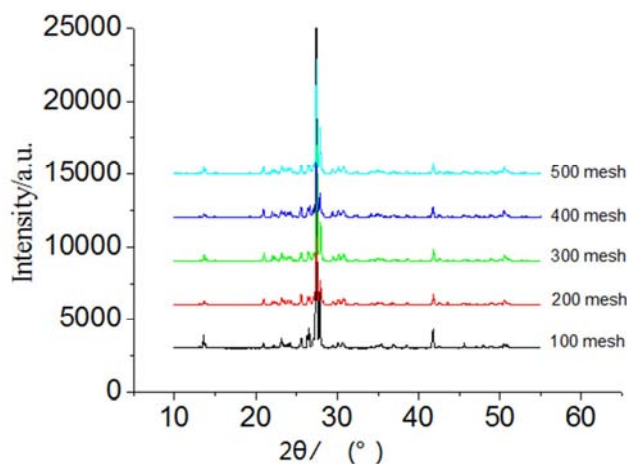


Figure 2. XRD patterns of K-feldspar in different particle sizes.

3.2. Effects of Operational Conditions on Potassium Dissolution Rate

3.2.1. Effects of Particle Size on Potassium Dissolution Rate

Effect of the particle size of K-feldspar on potassium dissolution rate was investigated at 160°C, reaction duration 2.5 h in traditional process and microwave-assisted processes (Figure 3). The potassium dissolution rate gradually increases with the decrease of particle size due to the larger solid-liquid contact area, which accelerated the leaching rate of K from the K-feldspar. However, further decrease in the particle size from 200 to 500 mesh does not lead to significant increase of potassium dissolution rate. This is probably due to the slightly increasing solid-liquid contact area. Hence, a particle size of 200 mesh was adopted for future tests.

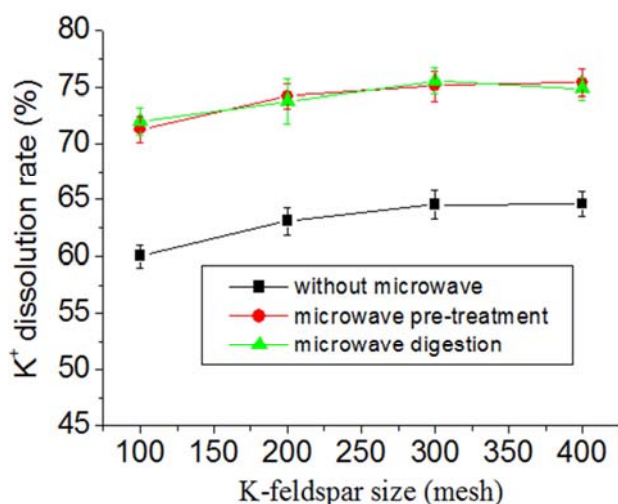


Figure 3. Effects of particle size on potassium dissolution rate.

3.2.2. Effects of Reaction Temperature on Potassium Dissolution Rate

Like most chemical reactions, the potassium dissolution rate increases as the temperature is raised. As shown in Figure 4, when K-feldspar particle size was 200 mesh,

increasing reaction temperature from 120°C to 180°C resulted in an increasing trend of potassium dissolution rate. At microwave-assisted processes, temperature impact on potassium dissolution rate was more significant, changing from 70% at 120°C to 80% at 180°C. At traditional process, however, temperature had less impact on potassium dissolution rate. It appears that the potassium dissolution rate is fast at higher temperatures. A positive impact of temperature on potassium dissolution rate is similar to the result in alkali system of extracting potassium [9]. For higher dissolution rate and lower energy considerations, the most suitable reaction temperature was 160°C.

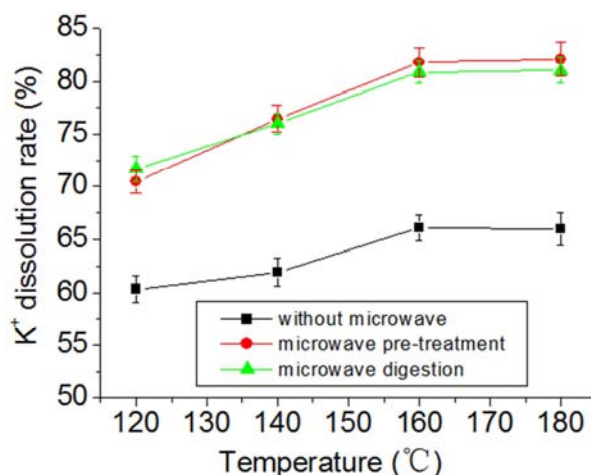


Figure 4. Effects of reaction temperature on potassium dissolution rate.

3.3. Potassium Feldspar Decomposition Residue Analysis

The crystallographic structure of the residues of potassium feldspar was determined by XRD. Figure 5 showed XRD patterns of residues after traditional reaction, microwave pretreatment and microwave digestion, respectively. The results of XRD proved the diffraction peaks changed significantly after potassium feldspar decomposition in three processes. XRD analysis confirmed the formation of calcium pyrophosphate and Na-feldspar in decomposition residues while SEM (Figure 6) studies confirmed different shapes of residues in three processes. K-feldspar surface formed a new solid product layer in the potassium extraction process assisted with microwave, which was different from traditional reaction process. The SEM images at different reaction processes showed that the reaction processes assisted with microwave were different from the traditional reaction, because the decomposition on the surface of the K-feldspar was different. In microwave reaction processes, the surface of the K-feldspar formed large amount of dense holes orderly. However, in the traditional reaction process the surface formed irregular bulk material. This result is in good agreement with the theory motioned in the literature that the microwave irradiation induces a volumetric, simultaneously and inside-out heating [16].

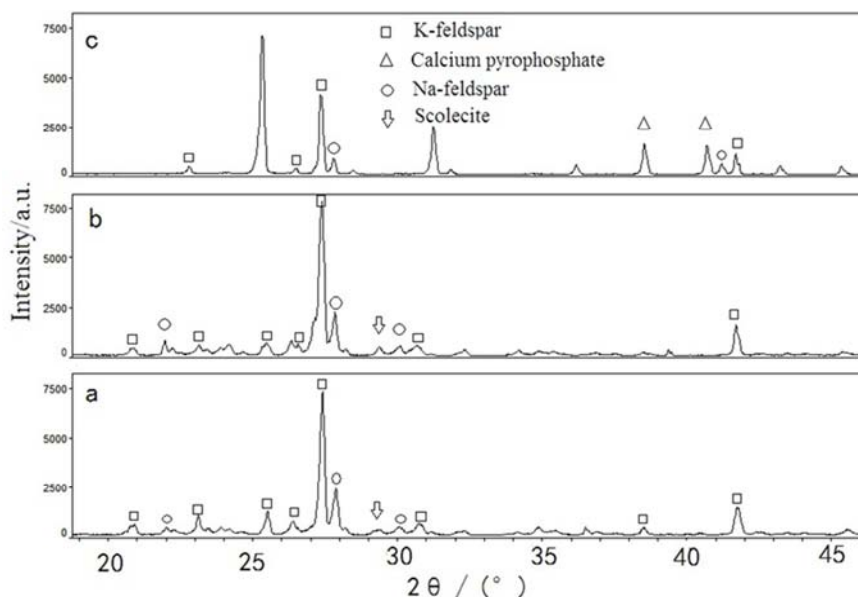


Figure 5. XRD patterns of residues (a: traditional reaction, b: microwave pretreatment, c: microwave digestion).

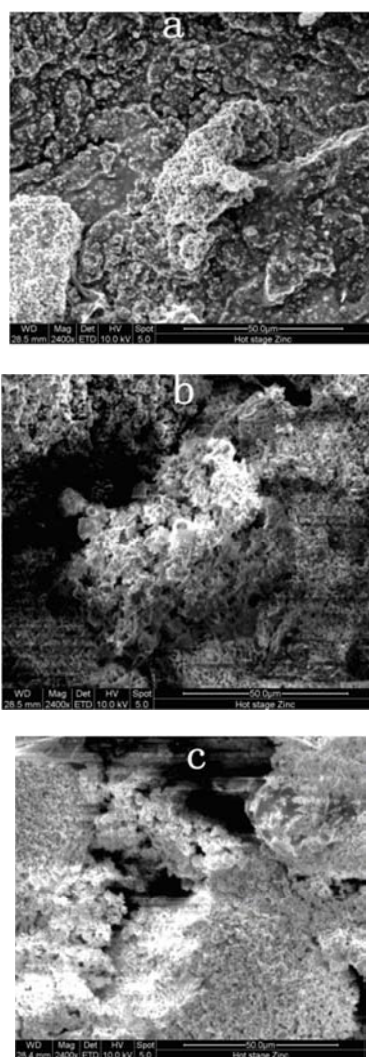


Figure 6. SEM images of residues (a: traditional reaction, b: microwave pretreatment, c: microwave digestion)

4. Conclusions

Microwave-assisted decomposition of K-feldspar at low temperature was studied in this paper. The experimental results indicated that potassium dissolution rate was up to 82% under the following conditions: reaction temperature 160°C and reaction time 150min. There was a clear change in peak shape when the power was 600W or higher. Increasing reaction temperature caused the increase of potassium dissolution rate.

XRD analysis confirmed the formation of calcium pyrophosphate and Na-feldspar in decomposition residues. Compared with traditional process, the dissolution rate of potassium was much higher in microwave pre-treatment and microwave digestion processes, indicating that the decomposition of potassium feldspar was more efficient under microwave-assisted condition.

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