

A chlorination roasting process to extract rubidium from distinctive kaolin ore with alternative chlorinating reagent



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ABSTRACT

A chlorination roasting process was proposed to recover rubidium from the distinctive rubidium-containing kaolin ore. The rubidium ions which existed in the alumina octahedron of the phyllosilicates structure could be extracted effectively by using CaCl_2 as chlorinating reagent due to formation of $\text{Ca}(\text{Al}_2\text{Si}_2\text{O}_8)$ phase in the chlorination process of kaolin ore, and the maximum extraction yield reached 96.95%. Additionally, the kinetics results of chlorination roasting of the kaolin ore showed that the extraction rate of rubidium was controlled by the chemical reaction, and the activation energy was found to be $40.13 \text{ kJ mol}^{-1}$ in the roasting temperature range of this study.

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1. Introduction

Rubidium finds wide applications in space technology, electronics industry, biological engineering and material science due to its high stability, easy ionization, excellent photoelectric characteristics, and extremely lively properties (Lenk et al., 2010). Total world demand for rubidium was estimated at about 2 to 4 tons per year, nevertheless, the increase in lithium exploration which may create new supplies leads to the expanded commercial applications (Thompson, 2011; Li et al., 2013).

Rubidium forms no minerals of its own, and the main commercially valuable sources of rubidium are natural brines of salt lake and the associated minerals, such as the lithium mica ($\text{KLi}_2\text{Al}(\text{Al,Si})_3\text{O}_{10}(\text{F,OH})_2$), the pollucite, and the zinnwaldite ($\text{KAl}(\text{Fe,Li})(\text{Si}_3\text{Al})\text{O}_{10}\text{F}_2$) (Vu et al., 2013; Buttermann and Reese, 2003). Recently, a large number of valuable rubidium containing kaolin ore (kaolin) has been found in the South China.

At present, rubidium is industrially extracted through two main processes: one is the acid decomposition method to extract rubidium from the lithium mica, rubidium containing pollucite and other minerals (Gao et al., 2014); and the other is the ion exchange or solvent extraction method to extract rubidium from brines (Pyper et al., 2006; Nisan et al., 2009; Yu et al., 2014). For the former process, the method requires expensive equipments, resulting in difficulty in

large-scale production. For the later process, the rubidium ions are extracted from brines by using an organic resin and an extraction agent. Very recently, various methods for extracting rubidium have been developed with special consideration for high extraction yield of rubidium. A chlorination roasting followed by water leaching process was used to extract rubidium from albite which was originated in the JiLin province of Northeast China; the rubidium oxide reacted with chloride ions to produce rubidium chloride (RbCl), and the extraction yield of rubidium could reach about 90% (Vu et al., 2013). The low-grade rubidium and cesium ores were also used to extract rubidium by a chlorination roasting process, and the maximum extraction yield of rubidium and cesium reached 97% and 99%, respectively (Jandova and Vu, 2008, 2009).

As one of the classic pyrometallurgical techniques, the chlorination roasting has been industrially applied to recover Ti, Zn and Nb from minerals with different chlorinating reagents such as HCl, NH_4Cl and CaCl_2 (Jaafar et al., 2011; Brocchi and Moura, 2008). In particular, rare earth was extracted by the chlorination roasting with NH_4Cl -MgO as the chlorinating reagents. MgO was pre-added into the bastnasite to deactivate fluorine to avoid formation of water-insoluble rare earth fluorides, and rare earth oxide of the MgO-added bastnasite reacted with HCl, which was produced from the decomposition of NH_4Cl under heating, to form rare earth chlorides (Chi and Zhang, 2004).

In this study, a novel chlorination roasting process with CaCl_2 or NaCl as an alternative chlorinating reagent was developed to extract distinctive rubidium-containing kaolin which was originated in the JiangXi province of South China. Key factors that influenced the

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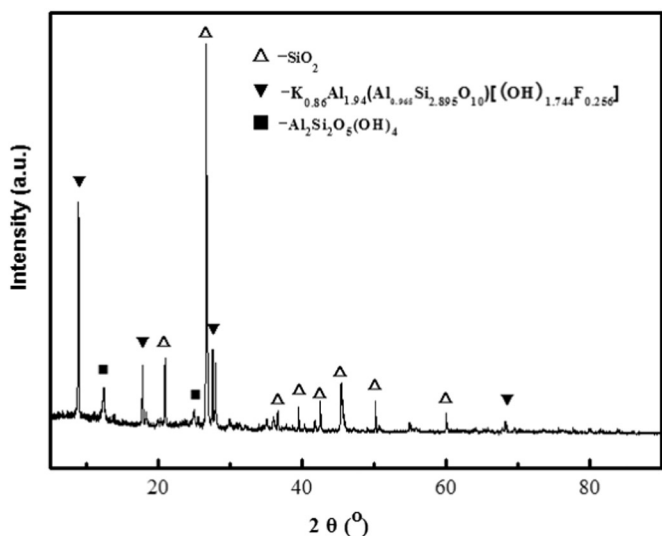


Fig. 1. XRD pattern of kaolin ore.

extraction yield of rubidium were studied, and the roasting kinetics was also investigated.

2. Experimental section

The kaolin, which was originated in the JiangXi province of South China, was crushed, ground and sieved to sizes lower than 100 μm . The treated kaolin powders thoroughly mixed with different chlorinating reagent (NaCl or CaCl_2) in a blending bottle, and then transferred into a corundum crucible. The crucible was put into a muffle furnace, the temperature was raised and the sample was kept at the preset temperature (600, 700, 800 and 900 $^\circ\text{C}$) for different periods of time (10, 20, 30, 40, 50 and 60 min), depending on the experiment requirements; subsequently the furnace was naturally cooled to ambient temperature. The roasted powders were taken out, ground, and leached by distilled water at 60 $^\circ\text{C}$ for 3 h under stirring condition, and then filtered to remove the soluble components. The filter cake was leached by distilled water several times to completely remove soluble components, and then dried to measure the residual rubidium. The rubidium content of filtrate was analyzed to calculate extraction yield of rubidium, which could be expressed as follows:

$$\text{Rb}\% = \frac{C \times V}{P \times M} \times 100\% \quad (1)$$

where Rb% is extraction yield of rubidium; C represents the rubidium concentration of the leaching solution; V is the volume of leaching solution; P is the mass fraction of rubidium in kaolin; M is the quantity of kaolin in the roasting process.

The phases of kaolin and roasted powders were analyzed by X-ray diffraction (XRD, APD-10X, Philips, Netherlands). The XRD analysis was carried out with a Rigaku diffractometer using $\text{CuK}\alpha$ radiation. The chemical compositions of kaolin and roasted powders were analyzed by X-ray fluorescence (XRF, Lab-X3500, Oxford, British). The chemical analyses of rubidium were performed by inductively coupled plasma-optical emission spectroscopy (ICP-OES, PE Optima 5300DV,

Table 1
Chemical composition of kaolin ore (wt%).

Si	Al	K	Fe	Na	F	Rb	O
23.669	14.487	3.931	0.571	0.561	0.979	0.219	55.178

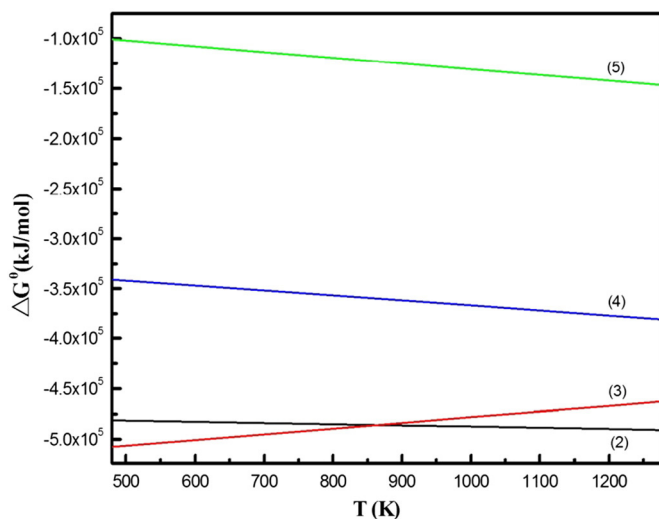


Fig. 2. Relationship between standard Gibbs free energy change of reactions (2)–(5) and temperature.

America). Differential scanning calorimetry (DSC) of kaolin mixed with different chlorinating reagent was performed at a heating rate of 10 $^\circ\text{C min}^{-1}$ with Al_2O_3 as the reference (TG-DSC, TGA/DSC 1, Switzerland).

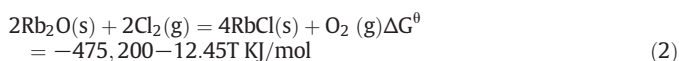
3. Results and discussion

3.1. Phase and compositions of kaolin

The kaolin belongs to the phyllosilicates structure, in which each basic unit layer structure is composed of two layered silicon oxygen tetrahedron and two alumina octahedron (Anandarajah, 1997). Fig. 1 shows the X-ray diffraction pattern of kaolin, where 2θ is the range of 5–90 $^\circ$. Peaks near $2\theta = 20.66^\circ$, $2\theta = 26.7^\circ$, $2\theta = 42.38^\circ$, $2\theta = 45.7^\circ$ and $2\theta = 50.35^\circ$ could be clearly indexed as a SiO_2 phase (quartz, JCPDS, No. 47-1144), analyzed by Jade 5.0, while the peaks at $2\theta = 9^\circ$, $2\theta = 17.69^\circ$, $2\theta = 27.60^\circ$ and $2\theta = 12.65^\circ$, $2\theta = 25.0^\circ$ were ascribed to $\text{K}_{0.86}\text{Al}_{1.94}(\text{Al}_{0.965}\text{Si}_{2.895}\text{O}_{10})[(\text{OH})_{1.744}\text{F}_{0.256}]$ (Muscovite-2M1, JCPDS, No. 06-0263) and $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ (kaolinite-1Md, JCPDS, No. 29-1488) phases, respectively. Rubidium-containing phases were not detected, indicating that the rubidium ions probably existed in the lattice of the alumina octahedron. Table 1 summarizes the chemical compositions of kaolin. As shown in the table, the kaolin contained 60.04% SiO_2 , 30.84% Al_2O_3 , 5.98% K_2O , 0.83% Na_2O , 1.06% Fe_2O_3 and 0.24% Rb_2O , based on the calculating standard of oxide. Overall, XRD and XRF results showed that the kaolin contained SiO_2 as the main phases and $\text{K}_{0.86}\text{Al}_{1.94}(\text{Al}_{0.965}\text{Si}_{2.895}\text{O}_{10})[(\text{OH})_{1.744}\text{F}_{0.256}]$ and $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ as the minor phases, and the content of rubidium reached 0.219%.

3.2. Thermodynamic analysis

Thermodynamically, the kaolin, which consists of SiO_2 , $\text{K}_{0.86}\text{Al}_{1.94}(\text{Al}_{0.965}\text{Si}_{2.895}\text{O}_{10})[(\text{OH})_{1.744}\text{F}_{0.256}]$ and $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ phases, can be conceived as the mixture of single oxide, such as SiO_2 , Al_2O_3 , K_2O , Na_2O , Fe_2O_3 and Rb_2O . In the chlorination roasting process of kaolin, the Rb_2O transforms to RbCl with an alternative chlorinating reagent such as Cl_2 , HCl , CaCl_2 and NaCl , and the possible chlorination reaction of kaolin with the chlorinating reagent can be listed as reactions (2)–(5). The relationships between standard Gibbs free energy of these reactions and temperature are plotted in Fig. 2.



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