



Technical note

Extraction of lithium from lepidolite via iron sulphide roasting and water leaching

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ABSTRACT

In this study, lithium was recovered from lepidolite via iron sulphide (FeS)–CaO roasting and water leaching. The thermodynamics taking place during the roasting of lepidolite and the additives was simulated by using the HSC programme. The simulation indicates that SO₂/SO₃ formed from the oxidation of FeS plays an important role in extracting lithium from lepidolite. Roasting at 750 °C using FeS/Li and Ca/F molar ratios of 5:1 and 1:1, respectively, followed by leaching at 50 °C using water/calcine mass ratios of >5:1 could yield a maximum of 81% Li recovery (at <1 g/L Li concentration). It was found that 71.5% of S was liberated into the atmosphere, resulting in lower extraction of Li and more diluted leach liquors compared to those obtained during closed-system roasting of lepidolite and Fe sulphate mixes.

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1. Introduction

Lithium, the lightest metal, has been utilised in the manufacture of e.g. glass, ceramics, and lubricating greases since its discovery in 1817. Lithium presently plays an important role in the high-tech industry due to its application in many fields including pharmaceuticals, rechargeable batteries and electronics. The global production was estimated to be 35,000 tonnes lithium in 2013 whilst its worldwide consumption was forecast to be approximately 30,000 tonnes, representing an increase of 6% compared to 2012 (USGS, 2014). An annual increase in lithium demand of 9.7% was projected until 2017 according to Roskill Information Services Ltd. (Roskill, 2013).

Manufacturers have processed salar brines and spodumene ores as dominant feedstocks for the production of lithium and lithium chemicals around the world. Containing 0.06–0.15% lithium, salar brines are widely used to produce high-purity lithium compounds because of low operation costs and the simplicity in processing. Meanwhile, spodumene ores have been treated to preferentially yield technical-grade concentrates containing <6% Li₂O directly used in glass and ceramic manufacturing although chemical-grade concentrates used for high-purity lithium production are also generated. The largest global deposits of salar brines are found in the “Lithium Triangle”, a region located in the

Andes Mountains, South America, whereas the largest reserve of spodumene was found at Greenbushes, Australia (Kesler et al., 2012).

Acid-roasting and lime-roasting of spodumene have been used by many manufacturers to produce lithium chemicals of technical, battery grade (99.5%) or high-purity (>99.9%) lithium carbonate (Roskill, 2013; Galaxy Resources Ltd, 2012). Such a well-known acid-roasting method involves firstly decrepitation (at 1070–1090 °C) to convert α-spodumene to a more reactive β structure, followed by sulphation using sulphuric acid at 250 °C and subsequent leaching (at 90 °C) of the calcine obtained to extract lithium into solution (Galaxy Resources Ltd, 2011; Nemaska Lithium Inc, 2014). This process has been applied by Galaxy Resources Ltd to spodumene concentrates mined at the Mt. Catlin and Greenbushes deposits, Australia. The same process is currently proposed by Nemaska Lithium Inc. to treat its spodumene resource at Whabouchi, Canada. The lime-roasting process, on the other hand, relies on the roasting of spodumene and lime at 1030–1040 °C before leaching the yielded clinker using water to recover lithium. The scheme was commercialised by Foote Mineral Company (now Chemetall) to produce lithium hydroxide from the 1950s to 1984 (Roskill, 2009). Other routes used to extract lithium from spodumene via pressure leaching with soda ash (Chen et al., 2011) or chlorination roasting (Barbosa et al., 2014) were also proposed.

Studies on lithium recovery from various lithium-bearing ores containing minerals such as petalite, zinnwaldite and lepidolite have also been developed. In recent years, zinnwaldite wastes discharged from processing of tin-wolfram ores in Cinovec, the Czech Republic

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(Jandová et al., 2010) and china clay in Cornwall, the United Kingdom (Siame and Pascoe, 2011) were tested to extract lithium. Limestone, gypsum and quenched lime were used to liberate lithium from zinnwaldite concentrates via roasting, and Li was then leached using water (Jandová et al., 2009, 2010; Vu et al., 2013). The concentrates (1.21–1.4 wt.% Li) were first recovered from zinnwaldite wastes (0.21 wt.% Li) using dry magnetic and grain-size separations. Sintering of mixtures of the concentrates and the additives at different temperatures ranging from 825 °C to 950 °C, followed by water leaching of the yielded calcines at 90–95 °C resulted in >90% Li extractions. Siame and Pascoe (2011) first mixed a zinnwaldite concentrate with either gypsum or sodium sulphate as a feed for roasting at 1050 °C and 850 °C, respectively. The calcines obtained were then leached with water at 85 °C, resulting in lithium recoveries of 85% and 97%, of which sodium sulphate roasting was noted to give a higher yield of lithium.

Lepidolite, one of the major mineral resources of lithium, has been recently studied to produce high-grade lithium compounds. However, no commercial extraction plant has commenced up to now. Sodium and potassium sulphate salts were mixed with lepidolite for roasting to extract lithium in studies by Yan et al. (2012a,b). Sulphation roasting of lepidolite using Na₂SO₄ was subsequently conducted by Luong et al. (2013) in which the role of LiKSO₄ and LiF in controlling the release of lithium during leaching was emphasised. Recently, a scheme using FeSO₄·7H₂O to extract lithium from a Korean lepidolite ore was also examined (Luong et al., 2014). It was believed that SO₂/SO₃ gases generated from the decomposition of such ferrous sulphate play a key role in the extraction of Li from lepidolite via a gas–solid interaction. The presence of CaO or CaCl₂ as a secondary additive to reduce the amounts of HF gas liberation by forming CaF₂ was also reported in these studies.

Other, cheaper sources of SO₂/SO₃ such as iron sulphide and sulphur metal should be evaluated. This study, therefore, aims to investigate the use of FeS in recovering lithium from a lepidolite ore collected from the La Vi deposit, Vietnam. The role of CaO in capturing fluorine and enhancing the extraction of lithium during lepidolite roasting was also studied. Thermodynamic modelling based on the HSC program (Outotec, 2011) was conducted to predict different factors controlling the extraction of lithium during roasting, e.g. temperature and FeS/Li molar ratio.

2. Experimental

2.1. Materials

Lithium-rich granite samples from a prospecting area of the La Vi deposit (Quang Ngai, Vietnam) were used in this study. A lepidolite concentrate (particle size <75 μm) containing 1.55 wt.% Li was then produced using froth flotation. Its chemical composition is shown in Table 1. The XRD pattern of the concentrate indicates that it contains mainly lepidolite – K(Li,Al)₃(Al,Si)₄O₁₀(F,OH)₂ and albite – NaAlSi₃O₈ (Fig. 1a).

2.2. Equipment and chemicals

The chemical compositions of the concentrate, the calcines produced from roasting and the residues after leaching were determined by X-Ray Fluorescence (XRF) using a Philips PW2404 spectrometer, whereas their mineralogical composition was evaluated by powder X-Ray Diffraction (XRD) using a Bruker AXS D8 Advance diffractometer. Atomic

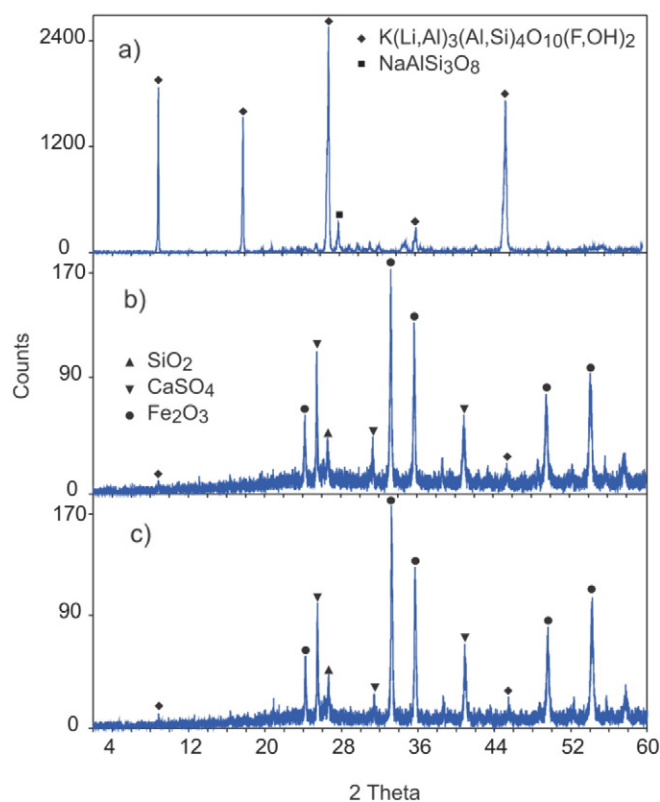


Fig. 1. XRD patterns of a) lepidolite concentrate; b) calcine after roasting at 750 °C for 1.5 h and with molar FeS/Li and Ca/F ratios of 5:1 and 1:1, respectively; c) residue after leaching at 50 °C for 2 h with a water/calcine mass ratio of 10:1.

Absorption Spectroscopy (AAS) and Ion Chromatography (IC) were applied to determine concentrations of cations and anions, respectively present in leach liquors. Differential Thermal Analysis (DTA) and Thermogravimetric Analysis (TGA) were conducted by using a Netzsch STA 449C instrument. A sealed tube furnace (Nabertherm R120/1000/12 with controller B180) was used for roasting tests, whereas hot plates were employed for the leaching experiments. All chemicals used in the study were of analytical grade.

2.3. Experimental techniques

Roasting and water-leaching were undertaken to extract and recover Li from lepidolite. During roasting, the concentrate was first mixed with reagent-grade iron sulphide (FeS) and calcium oxide (CaO) at various molar ratios of FeS/Li (1:1–7:1) and Ca/F (0:1–2:1). The mixtures were then poured into ceramic crucibles and placed in a tubular furnace for roasting at temperatures ranging from 700 °C to 850 °C. After roasting for 0.5–2 h, the resulting calcines were removed, cooled to ambient temperature and finely ground. Ground calcines were subsequently leached with water at 25–85 °C for 1–3 h using water/calcine mass ratios of 1:1–15:1 to release lithium into solution. Samples were taken at different time intervals and subjected to chemical analysis. Reproducibility of all results was estimated to be ±4% in several tests when roasting and leaching of the samples were done in duplicate.

3. Results and discussion

Roasting of lepidolite–FeS–CaO mixtures destroys the lepidolite structure and converts the mix into mainly Ca sulphate, hematite and quartz, as confirmed by XRD (Fig. 1).

Table 1

Chemical composition (wt.%) of lepidolite concentrate.

SiO ₂	Al ₂ O ₃	Fe	Mn	Mg	Ca	Na	K	Cs	Rb	Li	P	F
54.67	25.10	0.25	0.39	0.6	0.71	1.16	6.82	0.063	0.8	1.55	0.12	2.91

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