



Iron sulphate roasting for extraction of lithium from lepidolite



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ARTICLE INFO

Article history:

Received 10 April 2013

Received in revised form 7 September 2013

Accepted 29 September 2013

Available online 17 October 2013

Keywords:

Lithium
Lepidolite
Roasting
Leaching
Iron sulphate

ABSTRACT

Iron sulphate roasting and water leaching were investigated to extract lithium from lepidolite in this study. HSC modelling was used to simulate the process of roasting lepidolite with $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ and CaO . Based on HSC, three-dimensional models were derived to predict the effect of temperature, SO_4/Li and Ca/F molar ratios on the production of S- and F-containing gases (SO_2 , SO_3 , HF) and Li species (Li_2SO_4 , LiKSO_4) during roasting. It is believed that temperature and SO_2/SO_3 gases controlled the extraction of lithium from lepidolite during roasting while more soluble Li sulphate species determined the recovery of lithium during leaching. Using optimum parameters selected from HSC, roasting tests were conducted to produce calcines for leaching. Optimum roasting conditions were experimentally determined as 850 °C, 1.5 h, and SO_4/Li and Ca/F molar ratios of 3:1 and 1:1, respectively. Roasting in a closed environment led to more Li extracted than with an open system. Leaching the calcines obtained from the open and closed systems with a water/calcine mass ratio of 1:1 at room temperature for 1 h yielded leach liquors containing ~7.9 g/L Li and ~8.7 g/L Li, corresponding to ~85% and ~93% extractions of Li from lepidolite, respectively.

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1. Introduction

Lithium plays an important role in many industries, especially in high-tech applications. Lithium has been used in the production of ceramics, glass, lubricating greases, electronics, medicine, rechargeable batteries, etc. The global lithium production has expanded from 34,100 tonnes in 2011 to 37,000 tonnes in 2012 (USGS, 2013), representing an increase of nearly 10% within a year. The demand of lithium is predicted to increase steadily due to a high requirement in the manufacturing of electronic devices such as smartphones, laptop computers, and power tools. According to Roskill Information Services Ltd. (Roskill, 2009), an annual increase in lithium demand of 7% is projected from 2011 to 2025.

Sociedad de Quimica Minera de Chile SA (SQM), Rockwood Lithium in Chile, FMC Corp. in Argentina, and Galaxy Resources Ltd. in Australia are known as the biggest manufacturers of lithium around the world. Of these, the companies from South America have produced lithium carbonate from salar brines, which are abundant in this region. Containing 0.06–0.15% Li, salar brines are considered as the more common raw materials for the lithium production. Other mineral resources have also been recently developed or evaluated to extract lithium such as spodumene (by Galaxy Resources Ltd., WA, Australia), china clay (by Goonvean Ltd., Cornwall, UK) (Siame and Pascoe, 2011) and zinnwaldite wastes from tin–tungsten mines in Cinovec, the Czech

Republic (Jandová et al., 2010). Compared to brines, processing of lithium minerals is technically more difficult as extra operations are required, including beneficiation to produce a concentrate containing 1–3% Li and roasting in sulphate or carbonate to extract Li into water-soluble species before lithium can be solubilised into solutions. Conditions and results of major published studies on lithium recovery from ores are listed in Table 1.

During the treatment of lithium bearing minerals, ore concentrates of spodumene, lepidolite or zinnwaldite were first mixed with additives containing sodium sulphate (Jandová et al., 2009; Kondás and Jandová, 2006; Luong et al., 2013; Siame and Pascoe, 2011; Yan et al., 2012a,b) or calcium carbonate (Jandová et al., 2010) before being roasted at high temperatures (850–1050 °C) in order to decompose such hard rocks and convert Li to soluble species such as Li_2SO_4 , LiKSO_4 , and $\text{Li}_2\text{NaK}(\text{SO}_4)_2$. Calcines produced were then leached in water at different solid to liquid ratios and temperatures from which stage Li was extracted. There was no discussion on the potential release of gases (such as SO_2 and HF) in the latest studies, except from an early investigation by the US Bureau of Mines (Crocker et al., 1987).

Sulphation roasting has been applied in extracting Ni and Mo from lateritic ores (Guo et al., 2009; Wang and Wang, 2010) or Cu from sulphide minerals (Güntner and Hammerschmidt, 2012). Galaxy Resources is using sulphuric acid to crack Li from its pre-roasted silicate host (spodumene) and produce Li_2SO_4 for leaching in its plant (Galaxy Resources Ltd., 2008, 2010). The conversion of refractory or hard-to-leach metal oxides to sulphate is believed to be due to the reaction with SO_3 gas as suggested by Li et al. (2010,

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Table 1

Experimental profiles and results of several published studies on processing of Li from mineral sources.

Mineral tested	Luong et al. (2013)	Siame and Pascoe (2011)	Yan et al. (2012a)	Yan et al. (2012b)	Jandová et al. (2009)	Jandová et al. (2010)	Sitando and Crouse (2012)
	Lepidolite	Zinnwaldite	Lepidolite	Lepidolite	Zinnwaldite	Zinnwaldite	Petalite/lepidolite
Li %	2.55	0.96	2.00	2.00	1.4	1.21	1.9
K %	9.31	7.94	6.50	6.50	6.62	10.97	0.37
F %	3.69	3.36	4.46	4.46	Unreported	Unreported	0.01
Best roast temp, °C	1000	850	850	880	950	825	1100 (1st stage)
Roasting time, h	0.5	1	0.5	0.5	1	1	2
Additives	Na ₂ SO ₄	Na ₂ SO ₄	Na ₂ SO ₄ , K ₂ SO ₄ , CaO	Na ₂ SO ₄ , CaCl ₂	CaSO ₄ , Ca(OH) ₂	CaCO ₃	H ₂ SO ₄ (300 °C, 2nd stage)
Best leach temp, °C	85	85	Ambient	Ambient	90	90–95	50
Leaching time, h	3	0.5	0.5	0.5	0.5	0.5	1
Water/calcline ratio	2–18:1	10:1	2.5:1	0.8:1	10:1	5:1	7.5:1
Max. Li Extract, %	~90	>90	~90	~90	96	~90	97.3
Liquor Li concentration, g/L	1–3	Expected to be ~1	4.39	8.53	0.69	0.4	5.7

2011). In their studies, SO₃ produced from the decomposition of K₂S₂O₈ reacts with metals existing in sodium aluminosilicate (albite), calcium aluminosilicate (anorthite) minerals, etc. and converts them to soluble metal sulphates.

Recent literature studies used sodium sulphate to extract Li from its silicate hosts. However at >950 °C, a glass phase was formed making it difficult to recover calcines for leaching (Luong et al., 2013). The high sodium content in leach liquors would also create difficulty during lithium recovery using ion exchange for the production of high purity lithium products. Various calcium salts were used to control the release of fluoride into the leach liquors (Table 1), without reference to its presence in the gaseous phase during roasting.

Thus, the aim of this study was to use iron sulphate (with/without CaO addition) as the additive mixed with lepidolite to extract lithium during roasting. The formation of gases (SO₂, SO₃) during the decomposition of FeSO₄·7H₂O was studied as one of the key issues for extracting lithium from the concentrate to form soluble species. The role of CaO in reducing the release of F into gaseous or soluble forms was also investigated in this study. Thermodynamic modelling based on the HSC programme (Outotec, 2011) was conducted to predict different reactions encountered during roasting and identify the effect of temperature, SO₄/Li and Ca/F molar ratios on Li recovery. Leaching parameters including water/calcline mass ratio, leaching duration and temperature were also tested.

2. Experimental

2.1. Materials

A lepidolite ore (0.7% Li) collected from the BOAM mine (Gyeongsangbuk-do, Korea) was used as the material for this study. In most processes producing lithium from ores, beneficiation is required to upgrade the Li content to a concentration of ~1–3% Li before being intensively treated. Handpicking of the lepidolite grains (purple in colour) was therefore undertaken to yield a concentrate containing 1.79% Li before subjecting it to roasting tests. Its chemical composition is presented in Table 2 in which the content of Li was determined by ICP-AES after digestion of the concentrate whereas the other metal components were measured by X-Ray Fluorescence (XRF) analysis.

2.2. Equipment and chemicals

XRF analysis (using Rigaku ZSX Primus II) was used to determine the chemical compositions of the concentrate, calcines and residues after leaching whereas their structures were determined by X-Ray Diffraction (XRD) analysis (Cu-tube60kv 50 Ma, Phillips). Cation concentrations of leach liquors were measured by ICP-MS (Agilent A5500) whereas anions (SO₄, F) were analysed using Ion Chromatography (ICS-2000, Dionex). A muffle furnace was used for batch-roasting. Heating mantles and hot plates were used for leaching experiments. All chemicals used in the study were of analytical grade.

2.3. Experimental techniques

In this study, lithium was extracted from lepidolite via roasting and then recovered by water leaching. During roasting, the concentrate was mixed with iron sulphate and calcium oxide at different molar ratios of SO₄/Li (1:1 to 3.5:1) and Ca/F (0.5:1 to 2:1). They were thoroughly mixed before being poured into ceramic crucibles and then placed into the preheated muffle furnace for roasting. Two types of roasting were carried out including a closed system in which the crucibles were slightly covered by caps to ensure the gases liberated from the decomposition of FeSO₄·7H₂O do not quickly escape into the furnace atmosphere. The other was an open system in which there was no cap used. Various durations from 0.5 to 2 h were selected for roasting. A temperature range of 800–950 °C was set for the tests. After roasting, the calcines were cooled to laboratory temperature (20–22 °C) before being manually ground. Finely ground samples were then subjected to water leaching to determine the leachability of Li species.

Leaching was conducted at various temperatures ranging from 20 °C to 85 °C for 1 h. Magnetic bar stirrers were used for tests carried out at laboratory temperature whereas heating mantles with motored stirrers were used for high temperature leaching. A correctly weighed calcine sample (10–30 g) was introduced into a known amount of water (30–100 g) at liquid to solid mass ratios varying from 1:1 to 10:1. Preliminary tests showed that a steady state was reached after 15 min. All leaching experiments were conducted in duplicate.

Table 2

Composition (%) of the lepidolite concentrate by XRF and ICP-AES.

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	P ₂ O ₅	Na ₂ O	K ₂ O	MgO	CaO	MnO	Rb ₂ O	Cs ₂ O	Li	F
57.7	22.9	0.17	0.34	0.80	9.15	0.47	1.91	0.13	0.81	0.25	1.79	3.08

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