

Extraction of lithium from micaceous waste from china clay production

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ABSTRACT

The granites of South-West England are a potential source of lithium which is generally found within the mica mineral, zinnwaldite. It is mainly found in the central and western end of the St. Austell granite. When kaolin extraction occurs in these areas a mica-rich waste product is produced which is currently disposed of in tailings storage facilities. In this study a tailings sample containing 0.84% Li_2O was upgraded by a combination of froth flotation, using dodecylamine as the collector, and wet high intensity magnetic separation (WHIMS) to 2.07% Li_2O . The concentrate was then roasted with various additives, including limestone, gypsum and sodium sulphate, over a range of temperatures. The resulting products were then pulverised before being leached with water at 85 °C. Analysis of these products by XRD revealed that the water-soluble sulphates, KLiSO_4 and $\text{Li}_2\text{KNa}(\text{SO}_4)_2$, were produced under specific conditions. A maximum lithium extraction of approximately 84% was obtained using gypsum at 1050 °C. Sodium sulphate produced a superior lithium extraction of up to 97% at 850 °C. In all cases iron extraction was very low.

Preliminary tests on the leach solution obtained by using sodium sulphate as an additive have shown that a Li_2CO_3 product with a purity of >90% could be produced by precipitation with sodium carbonate although more work is required to reach the industrial target of >99%.

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1. Introduction

Lithium is important for a number of uses, including production of batteries, glass and ceramics. It is also used in the production of aluminium, preparation of greases, rubbers, alloys and pharmaceuticals. In 2008 lithium battery production represented 70% of the total rechargeable battery market (USGS, 2010) which includes mobile phones, laptop computers and power tools. The use of lithium for batteries has been increasing by more than 20% per year (USGS, 2010). The use of lithium-ion batteries in hybrid electric vehicles, plug-in hybrid and pure electric vehicles could see further significant increases in lithium production. Forecasts indicate that the demand for lithium in the next 5 years is expected to increase by approximately 60% from 102,000 t to 162,00 t of lithium carbonate or equivalent (LCE), with batteries representing more than 40,000 t of the perceived growth (Hykawy, 2010).

The primary source of lithium is from continental brines which typically contain 0.06–0.15% Li followed by pegmatites. The principal lithium minerals from pegmatite, with their theoretical maximum lithium content, are shown in Table 1.

Most of the lithium minerals from pegmatite are used for glass and ceramic production. Lithium chemicals, such as lithium carbonate, are normally produced from brines because of the lower

costs involved. China does however produce some lithium carbonate using imported spodumene as a feed stock. In a future scenario in which brine deposits could not meet the demand for lithium carbonate the deficit would have to be made up by increasing the use of lithium minerals from pegmatite. Zinnwaldite is one mineral that could be exploited as a lithium source in the future. The high iron content of the mineral combined with the relatively low Li_2O content makes it relatively unattractive at the current time.

The processing of lithium minerals from pegmatites involves both comminution and physical separation techniques such as gravity concentration, froth flotation and magnetic separation (Bale and May, 1989; Amarante et al., 1999). A novel comminution technique involving the application of high voltage pulses has been shown to improve the liberation of spodumene (Brandt and Haus, 2010). Once a lithium mineral concentrate has been produced it is typically roasted followed by leaching the products with either acid or water. This is an energy intensive chemical process. The total cost of the process is significantly affected by the requirements for mining, fine grinding, physical separation, high temperature roasting and evaporation. A number of lithium extraction processes have been reported for spodumene, petalite, lepidolite (Wietelmann and Bauer, 2008; Dresler et al., 1998) and zinnwaldite concentrates (Alex and Suri, 1996; Jandova and Vu, 2008; Jandova et al., 2009, 2010). The method used for the extraction of lithium from zinnwaldite by Jandova and Vu (2008) and Jandova et al. (2009, 2010) involved roasting of the concentrate with

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Table 1
Lithium minerals from pegmatite (from Harben (2002) and USGS (2010)).

Mineral	Formula	% Li ₂ O	Resource
Spodumene	LiAlSi ₂ O ₆	8.0	Australia, China, Canada, Zimbabwe, Portugal and Finland
Petalite	LiAlSi ₄ O ₁₀	4.9	Zimbabwe, Namibia and Canada
Lepidolite	K(Li,Al) ₃ (Si,Al) ₄ O ₁₀ (OH,F) ₂	6.2	Zimbabwe
Amblygonite	LiAlPO ₄ (F,OH)	10.3	Zimbabwe
Bikitaite	LiAlSi ₂ O ₆ H ₂ O	11.8	Zimbabwe
Eucryptite	LiAlSiO ₄	11.9	Zimbabwe
Montebrasite	Li ₂ O·Al ₂ O ₃ ·2SiO ₂	7	Canada
Jadarite	LiNaSiB ₃ O ₇ (OH)	3.16	Serbia – at feasibility stage
Zinnwaldite	KLiFeAl(AlSi ₃)O ₁₀ (F·OH) ₂	2.5–5	No current exploitation

limestone and gypsum to produce a water-soluble lithium salt. Roasting with gypsum/Ca(OH)₂ produced a lithium extraction of over 90% between 900 and 980 °C. With limestone a peak leach extraction of 90% occurred at 825 °C, with a significant drop-off above that temperature possibly caused by increased crystallinity of eucryptite (Jandova and Vu, 2008). Eucryptite normally requires leaching with strong acid for lithium leach extraction. The mineral is known to exist as different polymorphs that have varying stability ranges depending on temperature. Co-extraction of rubidium was around 25% for gypsum/Ca(OH)₂ and 90% for the limestone at the temperature that gave optimum lithium extraction. Alex and Suri (1996) used pugging of zinnwaldite at 700 °C with sulphuric acid to give lithium extraction of 90%. Unfortunately there was significant co-extraction of both iron and aluminium under these conditions.

The lithium potential of the St. Austell granite, situated in Cornwall (UK) was investigated by the British Geological Survey (Hawkes et al., 1987). An area of approximately 8 km² × 100 m depth was identified as containing a resource of 3.3 million tonnes of lithium. The china clay operations at Rostowrack and Treleavour Downs, run by Goonvean Ltd., fall within this resource area. Using the china clay production figures for these operations, it has been estimated that approximately 100,000 tonnes per year of mica-ceous residues (the hydrocyclone underflow product), assaying around 0.84% Li₂O, are available for treatment. A sample of mica-ceous residues from these areas was used in this study. Additional resource would be available by reprocessing material held in nearby tailings dams, although a lower Li₂O content would be expected as a result of mixing with residues from operations in lower Li areas. The advantage of using the hydrocyclone residue from an operating plant is that no additional mining costs are accrued. In addition the minerals within the residue are typically fine and well liberated therefore saving on the grinding costs that would result from the processing of other pegmatite deposits.

Previous studies have mentioned the physical separation of waste materials to produce a mica concentrate. Jandova et al. (2010) used dry magnetic separation whereas Hawkes et al. (1987) considered both froth flotation, for recovery of a mica concentrate, and dry magnetic separation for the separation of the various mica minerals. In these investigations a limited size range was used and no quantitative data on lithium and rubidium recovery was presented.

In this study we have investigated the efficiency of froth flotation and magnetic separation for separation of both lithium and rubidium from the hydrocyclone underflow. Where possible we have linked mineralogy with separator performance. Following production of a lithium–mica concentrate the effectiveness of the roast/water leach procedure has been investigated using the reagent systems considered by Jandova and Vu (2008) and Jandova et al. (2009, 2010) with the addition of sodium sulphate (Na₂SO₄). The mineral phase changes that occur during roasting have been followed using X-ray diffraction (XRD) and differential thermal analysis (DTA).

2. Experimental

2.1. Materials

The material used in this test work was obtained from Goonvean Ltd., St. Austell, UK. The sample was collected from the underflow of a group of 250 mm diameter hydrocyclones, which were processing material for china clay production, mined from the Trelavour Downs and Rostowrack pits. The overflow is further processed into china clay products ready for sale. The underflow, which consists of fine mica-rich sand, is a waste product that is discharged to a nearby tailing dam (see Fig. 1).

The hydrocyclone underflow was further classified using a 50 mm laboratory hydrocyclone operated at a pressure of 276 kPa in order to remove the majority of the –10 μm fraction which is known to be rich in kaolinite. Fig. 2 shows the particle size distribution of this fraction obtained using a Malvern laser-sizer (Mastersizer MAF 5000). The de-slimed hydrocyclone underflow was then homogenised before being riffled into 1.2 kg lots. These lots were the feed samples for the froth flotation experiments.

2.2. Analytical procedures

Semi-quantitative information on the mineralogy of the feed, separation products and new materials formed on roasting were produced by X-ray diffraction (XRD) using a Siemens Diffractometer D5000. X-ray fluorescence (XRF), using a Bruker S4 Pioneer, with the boric jacket preparation method was used for elemental analysis of solid samples. Quantitative elemental analysis of specific mineral grains was undertaken using a JEOL JAX-8200 electron microprobe. Atomic absorption spectrometry (AAS), using a Unicam SP 9 spectrometer, was used for the determination of lithium

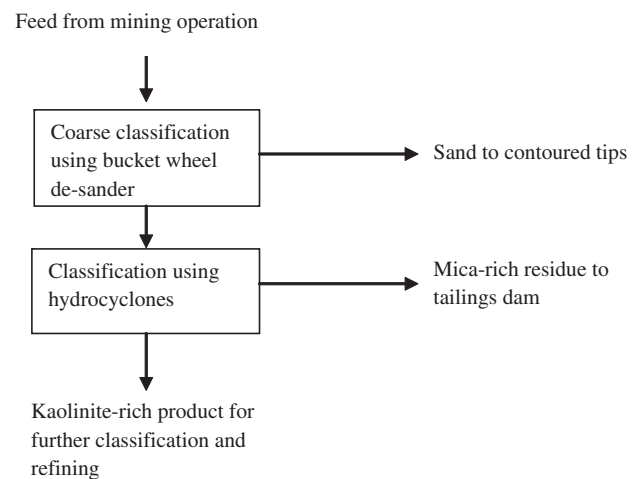


Fig. 1. Simplified flow diagram of china clay production by Goonvean Ltd.

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