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Optimisation of spodumene flotation

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ABSTRACT

Results of investigations into the optimum conditions for concentration of spodumene by anionic froth flotation are presented. Parameters investigated include: collector dosage; cell rotor velocity; feed pH, conditioning period, temperature, solids content, and reprocessing steps required to yield an optimum product. Results are based upon sequential testing of various values for each parameter while maintaining all others constant. These show that optimum conditions are attained with: the pulp at a pH of 7.5 to 9.8, 18% solids, 15°C and a 10min conditioning period; a cell rotor velocity of 1040rpm; and that an oleic acid concentration of 1.4kg/t as a collector is adequate. It was also found that under these operating conditions, no significant increase in grade could be achieved with more than two passes through cleaner cells. Analysis of these tests indicates that a product with an optimum lithia content and a calculated recovery of 96.24% may be achieved by the cleaning the rougher concentrate twice in conventional sub-aeration flotation cells.

Keywords: Froth flotation; Operating parameters; Spodumene

1. INTRODUCTION

Spodumene is an important mineral in the production of vitreous products. It is valued for reducing firing temperature and flux consumption, decreasing melt viscosity, increasing furnace throughput, improving forming characteristics, increasing product thermal shock resistance while serving as a bulk source of alumina and silica. The use of lithium compounds for remediation of premature concrete deterioration represents a new, potentially very large market. Spodumene is by far the most widely used of the lithium bearing minerals produced from pegmatite type deposits. Theoretically it contains 8.03% Li_2O but commercial products contain from 2.9 to 7.6% Li_2O.

The beneficiation of ores to recover spodumene is commonly accomplished by heavy media separation, the decrepitation process, and froth flotation followed by a reduction of iron bearing minerals in the final product using magnetic separators. Froth flotation is the most widely used procedure for beneficiation of lithium bearing silicates such as spodumene. Only a limited number of ores can be treated by heavy media separation and it is anticipated that the use of the decrepitation process will decline due to the health risk associated with carcinogenic cristobalite as a potential by-product of this process.

A high degree of recovery is key to the viability of mining operations producing spodumene to meet market specifications. As froth flotation is a process where a large number of variables can be adjusted to regulate the minerals recovered or rejected, it has become a widely established procedure in the mining industry. Despite a large number of investigations into the flotation of lithium minerals such as spodumene having been conducted, information on the specific conditions for froth flotation of lithium minerals such as spodumene is extremely limited. Publications of Norman and Gieseke (1940), Browning (1961), Arbiter *et al.* (1961), Thom (1962) and Rau (1985) provide indications of some but not all the principal operating parameters. Reports published of investigations by public institutions (eg. Redeker (1979), Andrews (1993) and Amarante *et al.* (1998)) tend to be oriented towards the development and promotion of specific deposits and document only a few of the major flotation parameters applied.

Optimisation of recovery while meeting the limitations of product specification has always been of primary importance to the mining industry. The recovery of lithium minerals by flotation can be accomplished by anionic and or cationic flotation. Anionic flotation tends to provide excellent recovery but

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has a major disadvantage in that it does not tend to produce a high purity concentrate. Cationic flotation tends to be the opposite in that recoveries tend to be much lower but with much purer concentrates. Anionic flotation as an initial rougher followed by cationic cleaner flotation offers the advantage of high recoveries and a pure concentrate but the removal of anionic collector requires a sulphuric acid wash which implies additional processing and costs. The objective of this report is to document the optimum conditions for the recovery of spodumene through standard direct (anionic) froth flotation to produce a high-grade concentrate without using cationic flotation as a cleaner. This investigation into the recovery of spodumene used low-grade ore with a significant variation in lithium content in the spodumene that could be considered as a typical to severe case for froth flotation.

2. EXPERIMENTAL

2.1 Material

Investigations were conducted using a sample collected from the Vilatuxe pegmatite district of Pontevedra, Spain. The material was selected for being representative of typical, partially altered, sub-surface material characteristic of somewhat low-grade ore (1.85% Li_2O) and accompanied by the gangue minerals typical of deposits of this type-quartz, albite and muscovite. Accessory minerals present include beryl, columbite-tantolite and apatite. Traces of cassiterite, zircon, arsenopyrite, garnet, tourmaline, topaz, pyrite and chalcopyrite are also present.

The spodumene grains present were found them to contain from 5.55% to 7.03% Li_2O. The relatively low grade of the ore and variation in lithium content of the spodumene, also being relatively low, would normally present significant difficulties in the recovery of the spodumene and in preparing a marketable concentrate grade. It was observed that at a crushing of the ore to <0.4mm, a liberation of 89% of the spodumene from the gangue minerals was obtained with only slight improvement obtained at finer grain sizes.

2.2 Apparatus

Tests were conducted in batch using a Denver D12 sub-aeration flotation cell, as its principal characteristics are identical to those used at an industrial scale. It was operated using the large (12.7cm) impeller/diffuser assembly with the 5-litre cell. The selection of the reagents used (caustic soda for pH control, oleic acid as a collector and pine oil as a frother) was based on them being widely available at a low cost and for being commonly used in industrial flotation processes.

The lithium content in the samples, concentrates and tailings were determined by atomic absorption analysis with a ±7% margin of error.

2.3. Methodology

A series of tests were conducted whereby the optimum conditions for separation and recovery were sequentially determined for the principal operating parameters:

- Dosage of oleic acid as a collector
- Pulp pH
- Flotation cell rotor velocity
- Pulp conditioning period with 0.08kg/t of pine oil
- Pulp temperature
- Feed stock concentration
- Reprocessing (cleaning) steps

Evaluation of the these parameters was determined by conducting a set of tests where the first parameter (dosage of oleic acid as a collector) was varied while maintaining constant all other factors of pulp pH (7.5), temperature (15°C), conditioning time (6min), density (18% solids) and cell rotor velocity during flotation (1040rpm). As this and the subsequent sets of tests were completed, the optimum parameter(s) determined were applied in the following tests while maintaining all other undetermined parameters as before. Values of each parameter applied are indicated in Table 1.

3. RESULTS AND DISCUSSION

The samples were prepared by grinding the ore to <0.4mm and then submitting it to attrition at 80% solids in a solution of caustic soda at a pH of 12 to liberate fines from the grains. The minus 50 micron fraction (8% of the raw ore) was rejected. The deslimed ore contained 2.01% Li_2O.

Table 1
Variations of parameters applied in froth flotation tests of spodumene

Dosage of oleic acid collector (kg/t)	0.35, 0.70, 1.40, 2.10, 2.80
Pulp pH	3.1, 4.7, 7.5, 9.8, 10.9
Flotation cell rotor velocity (rpm)	760, 1040, 1350, 1680
Pulp conditioning period (min)	1, 6, 10, 15, 20
Pulp temperature (°C)	15, 33, 50, 70
Feed stock concentration (%)	9.4, 17.8, 25.2, 32.0
Cleaning passes of concentrate	4 (excluding initial rougher)

The results of the tests with increasing concentrations in oleic acid as a collector (Figure 1) demonstrate the evolution in grade and recovery of the spodumene in the float. The highest grade recovered occurred at the lowest concentration (0.35kg/t) whereas the optimum recovery (96.9%) occurred at and above 1.40kg/t. Increases in collector concentration above this level attained an optimum concentrate grade of 4.7% Li_2O at 2.10kg/t. of oleic acid at an additional 2.0% loss in recovery. Higher collector concentrations resulted in a significant reduction in concentrate grade. This discrepancy is attributed to the more selective recovery of the less altered grains at lower concentrations while at intermediate levels, a more complete recovery was obtained but which also collected more gangue minerals as the oleic acid concentration was increased. With this series of tests being the first to be conducted and a high level of recovery being critical in this investigation, rather than accepting a lower recovery but higher grade, an oleic acid concentration of 1.4kg/t as a collector was considered the optimum level. In subsequent consideration, a 2.1kg/t might be preferable.

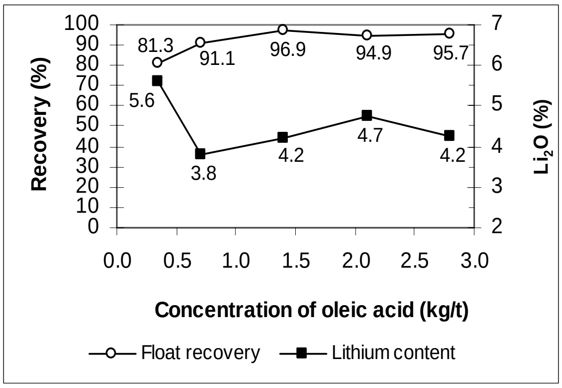

Figure 1. Recovery and grade of lithium oxide in float with variation in oleic acid collector

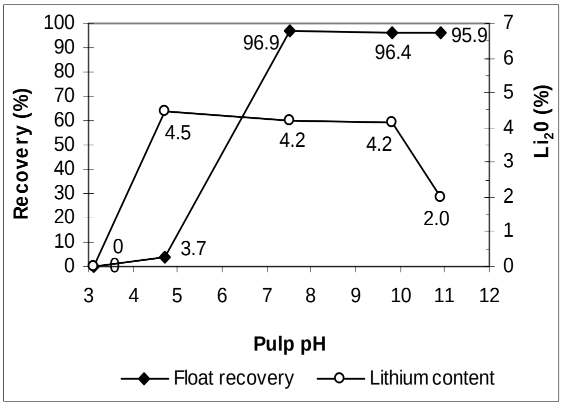

Figure 2. Recovery and grade of lithium oxide in float with variation in pulp pH

The effect of variation in pH of the pulp determined in the tests and as indicated in Figure 2 indicates a pronounced evolution in of the flotation of spodumene from pH 3.1 to 7.5. At a pH of 3.1 no spodumene is recovered whereas at and above 7.5 more than 95 to 96% is recovered. At a pH of 10.9 virtually all of the minerals present (96.4%) are recovered with the float, resulting in virtually no selective flotation.

Clearly from the Li_2O grade of the float, the optimum pH is from 7.5 to 9.8. The slightly higher level of recovery of 96.9% attained at a pH of 7.5 is indicative of being the optimum value, especially so as a minimum of reagents is required.

The results of tests of the effect in variation of the rotor velocity (Figure 3) on the recovery and grade of the produced concentrate shows a trend of progressive reduction in selectivity of the flotation process with increased rotor velocity. Recovery however increases significantly from 760rpm to 1040rpm. No significant change in recovery is noted at higher rotary velocities.

The results of tests to assess the effect of the conditioning period on the pulp prior to flotation as shown in Figure 4 also indicates a general trend of increased selectivity in the flotation process with period of pulp conditioning and a slight reduction in recovery with more than 10 minutes of conditioning of the pulp. A conditioning period of 10min is considered to be the best compromise between recovery and concentrate grade.

Temperature is a critical component in the effectiveness of the collector selectivity, as is indicated by the results of tests (Figure 5) with variations in pulp temperature. These results show a trend in the reduction of collector selectivity with increased temperature and that above 33°C there is a relatively insignificant increase in recovery at a considerable loss in selectivity. In selecting the optimum pulp temperature, the slightly lower recovery (95.9%) for a greater selectivity in concentration at 15°C is deemed preferable over the higher recoveries but lower concentrations obtained at higher temperatures. This also offers a valuable energy savings in the processing procedure.

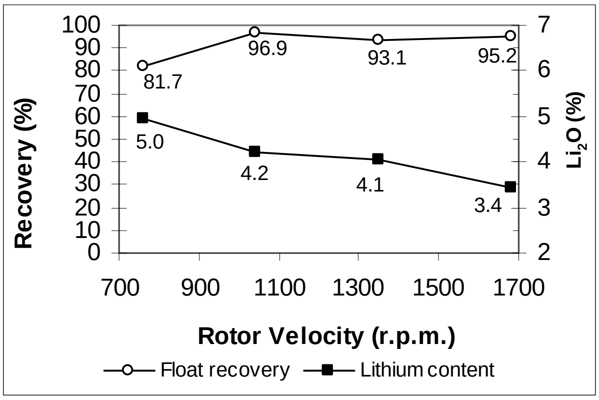

Figure 3. Recovery and grade of lithium oxide in float with variation in flotation cell rotor velocity

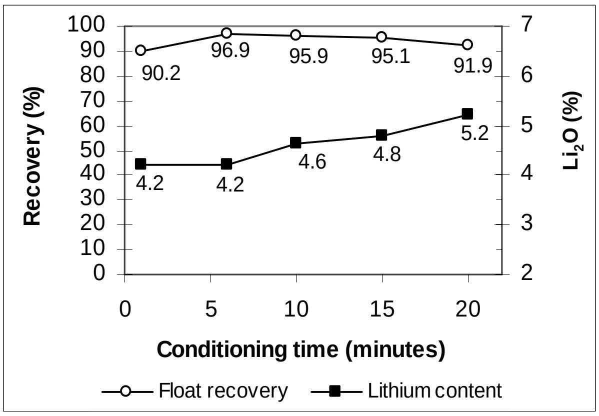

Figure 4. Recovery and grade of lithium oxide in float with variation in duration of pulp conditioning

Results of the tests of the variation in recovery and grade of concentrate produced by the flotation as shown in Figure 6 indicate an optimum recovery and a corresponding optimum concentration of spodumene at 17.8% solids under the operating conditions determined in the previous tests. Greater and lesser pulp concentrations all yielded slightly lower recoveries and notably weaker selection of the spodumene from the gangue minerals.

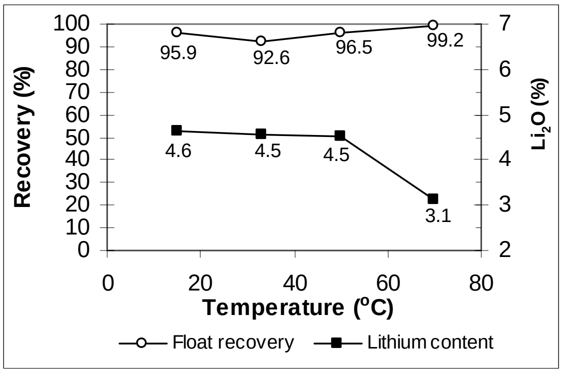

Figure 5. Recovery and grade of lithium oxide in float with variation in pulp temperature

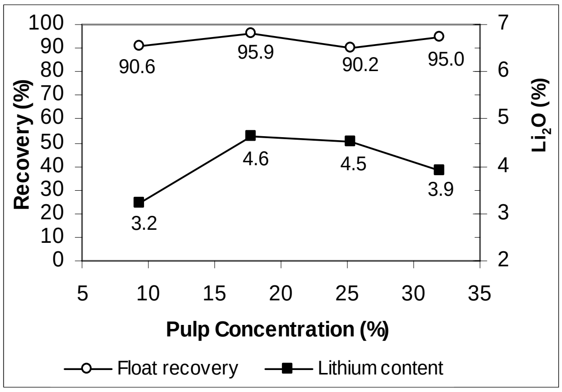

Figure 6. Recovery and grade of lithium oxide in float with variation in pulp concentration

The cleaning of the rougher concentrate (especially with anionic flotation) is necessary to produce a relatively pure product to meet market specifications. The results of the tests whereby the rougher concentrate was repeatedly cleaned (Table 2) indicate that the maximum attainable lithia content is with four passes but that after the second pass there is only marginal improvement gained and at a loss in recovery. In all probability any increase in grade by additional cleaning is due primarily to elimination of incompletely liberated grains or those having low lithia contents.

The grades and relatively low volumes of tailings from the cleaning cells are such that, those from the first cleaning cell could be recycled to the rougher cell while those from the second cleaning cell could be recycled to the first cleaning cell, without the results varying significantly from those indicated here. Based on this recycling of the cleaner cell tailings, the optimum attainable recovery is calculated to be 96.24%.

Table 2
Results from repeated cleaner cell flotation of rougher concentrate

Product	Rougher		Cleaner 1		Cleaner 2		Cleaner 3		Cleaner 4	
	Distrib. (%)	Grade (%)	Distrib. (%)	Grade (%)	Distrib. (%)	Grade (%)	Distrib. (%)	Grade (%)	Distrib. (%)	Grade (%)
Conc.	42.93	4.15	83.09	5.42	73.25	6.24	62.47	6.24	53.79	6.46
Tails	57.07	0.13	16.91	2.63	26.75	3.87	37.53	6.24	46.21	6.03
Calc. feed	100	2.01	100	4.15	100	5.42	100	6.24	100	6.24

4. CONCLUSIONS

Based on the tests conducted, optimum conditions for anionic froth flotation of spodumene include:

- Dosage of oleic acid at 1.4kg/t is adequate. A dosage of 2.1kg/t increases the concentrate grade by 20% at a 1.2% loss in recovery.
- Pulp pH should be from 7.5 to 9.8. Recovery is extremely poor to inexistent at a higher acidity and there are similar results in the selectivity of the spodumene at more alkaline values. A pH of 7.5 is considered preferable due to the associated lower costs of reagents and problems associated with corrosion.

- Flotation cell rotor velocity should be 1040rpm. Above this speed there is less selectivity in the process and below it recovery levels are considered unfavourable.
- Pulp conditioning period with 0.08kg/t of pine oil of 10 minutes yields an optimum compromise between recovery and float grade.
- Pulp temperature should be 15°C. At higher temperatures, especially above 50°C, results in significant reductions in selectivity of the collector.
- Feed stock concentration should be 18% solids. Selectivity of the process is reduced at higher and lower concentrations.
- The rougher concentrate should pass through two stages of cleaning cells steps to optimise the grade of spodumene produced. Additional cleaning does not provide any significant improvement in the concentrate grade.

A concentrate with a higher grade cannot be produced without rejecting incompletely liberated grains or those with a low lithium content due to the mineralogical characteristics of the spodumene from the deposit utilised in these tests. It is calculated as a first approximation, that, based on recycling the tails of the first cleaning cell to the rougher cell and the tails from the second cleaning cell to the first cleaning cell, the best attainable recovery to be 96.24%.

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