Flotation beneficiation of lithium-bearing ore and determination of lithium with AAS

Purpose of the experiment

The purpose of this work is to become familiar with flotation as a phenomenon and with the equipment used in flotation processes. In this work, the flotation of the lithium-bearing ore spodumene pegamatite from Keliber's Syväjärvi mine is investigated, along with the effects of various parameters on the ore's floatability.
The objective is to perform the beneficiation of a finely ground lithium ore using laboratory-scale flotation equipment and determine the lithium content of the feed, concentrate, and tailings in accordance with a separate analytical procedure.

Theory

Flotation is the most widely used method for mineral beneficiation. In the flotation process, the desired minerals suspended in a mineral slurry are made to attach to air bubbles generated with the aid of reagents and are subsequently floated to the surface of the slurry as a concentrate. This process exploits the differences in the physicochemical surface properties of minerals. For a desired mineral to attach to an air bubble, its surface must be water-repellent, or hydrophobic.

If a mineral is not naturally hydrophobic, various collector reagents are used. These reagents selectively adsorb onto the surfaces of the desired minerals, forming a water-repellent layer. Collector reagents are typically organic compounds with polar molecules. One end of the molecule can attach to the mineral surface, while the other end consists of a hydrophobic hydrocarbon chain that repels water. Various modifying reagents are also used in flotation to activate the desired minerals and depress unwanted minerals.

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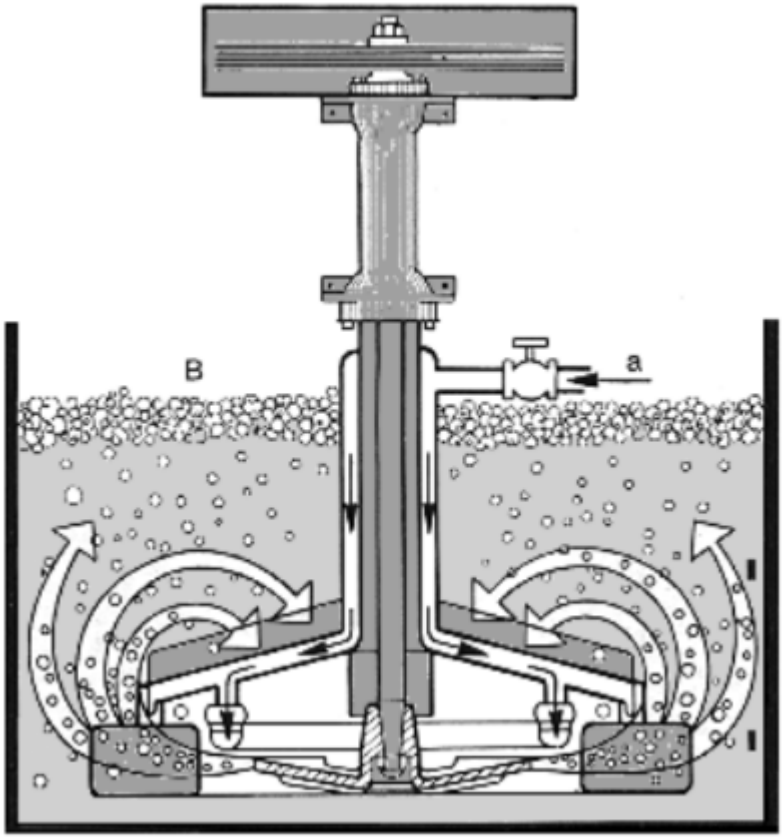
To produce a sufficient stable and load-bearing froth, frothing agents are added to promote the formation of forth bubbles. Prior to flotation, the ore must be ground to a sufficiently fine particle size so that each mineral occurs as individual particles and composite particles containing multiple minerals are minimized. This liberation of mineral grains enables selective flotation.



Picture 1. Flotation cell

Laboratory equipments:

- Crucible
- Graduated cylinder
- Beaker
- Bühner funnel
- Filtration paper
- pH meter

Reagents:

- Tap water
- NaOH solution
- Frother
- Collector
- Nitric acid
- Hydrochloride acid
- Sulphuric acid

Equipments and devices:

- Jaw crusher
- Ball mill
- Screen with 125 µm screen mesh
- Flotation cell
- pH meter
- Muffle furnace

Procedure

Before starting the work, familiarize yourself with the safety data sheets of the reagents. A separate laboratory report is to be prepared for the work.

Crushing, grinding and screening

Weigh slightly over 500 g of ore and crush the boulders using a jaw crusher to achieve the smallest possible particle size. Crushing is carried out in two stages, for example using settings of 10 mm and 0,1 mm. Grind the crushed material in a ball mill to a particle size below 125 µm. If necessary, sieve the crushed material in several stages using a 125 µm screen mesh. Collect the material passing through the screen mesh and return the material remaining on the screen mesh back to the ball mill for further grinding.

Continue processing the ore material until 500 g of ore has been ground to a particle size below 125 µm. Take a 1 g sample form the well-mixed ore powder. Perform the weighing using an analytical scale to ensure accurate results.

Steps of flotation

Flotation is carried out in a 2-liter flotation cell. Add two liters of water and 500 g of feed into the cell, start the flotation cell, and adjust the rotational speed to 1100 rpm.

Preflotation

To remove impurities in the ore, a preflotation is carried out. Add 5 mL portions of 5 % NaOH solution and adjust the pH to 10,5. Ass 1,5 mL of Berol 050 frother and 0,06 g (approximately 3 drops) of rapeseed fatty acid collector and allow to condition for 15 minutes. Open the air valve. When froth begins to form, skim it from the surface of the flotation cell. Continue until no more forth is produced. This froth contains impurities from the ore and is not collected.

Flotation

Add the H_2SO_4 solution in 5 mL portions, adjusting the pH to 7,5. Add 7,5 mL Berol 050 frother and 0,25 g (approximately 10 drops) pf rapeseed fatty acid collector and allow to condition for 15 minutes. Open the air valve. When froth begins to form, skim it from the surface of the flotation cell. Continue until no more forth is produced. Collect the froth and filter it through filter paper using a Büchner funnel. The precipitate is then dried in an oven.

Scavenging

The objective of the scavenging stages is to increase the recovery rate, i.e. to recover as much lithium as possible into the collected froths. The scavenging is carried out three times, following the same procedure as in the previous flotation stages. Collect the froth and filter the precipitates. Remember to weigh the filter papers before filtration and record the measurements in the laboratory notebook. The amounts of chemicals used in the scavenging stages are given in the table below.

Scavenging 1.	Scavenging 2.	Scavenging 3.
Berol 050 frother: 2,25 mL	Berol 050 frother: 2,25 mL	Berol 050 frother: 1,5 mL
Rapeseed fatty acid: 1,0 g	Rapeseed fatty acid: 0,15 g	Rapeseed fatty acid: 0,15 g

Filter the tailings remaining in the flotation cell. Place the samples in a drying oven to evaporate the water. Accurately weigh the dried tailings and concentrate to determine the recovery percentage.

Note: Retain the obtained concentrate and tailings until the experiment has been completed and the laboratory report has been approved.

Sample preparation for analysis

These steps must be carried out in a fume cupboard. Beware of acid splashes.

1.Weight approximately 5 g with an accuracy of 1 mg of both the concentrate and tailings samples into deep crucibles. Transfer the previously weighed feed sample into a crucible as well and ignite the samples in a muffle furnace at 950 °C for 2 hours. Place the samples in a cold furnace before switching it on. Use the following program:

- Slope: 500 °C/h
- T1: 950 °C
- T2: 950 °C
- Time: 2 h

2. After ignition and once the furnace has cooled, weight approximately 1 g of each sample into beakers for digestion, using an accuracy of 1 mg.

3. Add approximately 25 mL of concentrated hydrochloric acid (HCl) to each sample and heat on a hot plate in the fume cupboard until approximately 5 mL of liquid remains.

4. Carefully add approximately 20 mL of concentrated nitric acid (HNO_3) and continue heating until approximately 5 mL of liquid remains.

5. Carefully add approximately 50 mL of water.

6. Heat the solution until approximately 20-30 mL of liquid remains.

7. Filter the resulting mixture by gravity filtration using filter paper and a funnel into a 100 mL volumetric flask and dilute to the mark with water.

Determine the lithium concentration of the samples by atomic absorption spectroscopy (AAS) according to the separate analytical procedure.

Formulas used for calculations

Processing of results

1. Calculate the amount of lithium contained in the feed, concentrate, and tailings based on the analytical results.
2. Calculate the concentration ratio and lithium recovery of the flotation experiment.

Laboratory report

Prepare a written laboratory report describing the experimental procedure, calculations, results, and evaluation of the results.

Concentration ratio (K)

$$K = \frac{F}{C} = \frac{c-t}{f-t}$$

F = mass of ore feed

C = mass of the concentrate

c = lithium concent of the concentrate, %

f = lithium concent of the feed, %

t = lithium concent of the tailings, %

Recovery percentage

Recovery (**R**) is the fraction of the valuable mineral or metal that is recovered into the concentrate.

$$R = 100 \cdot \frac{c \cdot C}{f \cdot F} \%$$

Recovery (**R**) can be expressed based on analytical values as follows:

$$R = 100 \frac{c\,(f\text{-}t)}{f\,(c\text{-}t)} \%$$

Handling of waste

Pour the solutions down the drain and place the dried flotation tailings and concentrate into their designated containers. Dispose of the filter papers in energy waste.

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