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VARIABILITY IN FLOTATION PERFORMANCE OF LEPIDOLITE SAMPLES FROM THE EMILI DEPOSIT, FRANCE

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ABSTRACT – Lepidolite flotation testwork was performed on 21 variability samples from the EMILI deposit in Allier, France, with head assays ranging from 0.49 to 1.38% Li_2O and an average K80 of 236 µm. After desliming, flotation was performed with an amine collector which was able to produce lepidolite concentrates grading >4.2% Li_2O with over 74.6% lithium recovery from 13 samples – often without cleaner flotation. The lower grade lepidolite concentrates (<4.2% Li_2O) were a result of unwanted recovery of other mica-type gangue minerals, as well as low feed grades below the 0.8% Li_2O cut-off grade already excluded from the mine plan.

Keywords: Lepidolite, Flotation Optimization, Mica Contamination.

INTRODUCTION

The EMILI project (EMILI stands for Exploitation de Mica Lithinifère par Imerys) is a major lithium deposit under the Beauvoir Kaolin Quarry site in the Allier region, France. The drilling campaign revealed indicated resources with an average content of 1.07% Li_2O, 0.1% Sn, and 0.02% Ta, confirming the attractiveness of the deposit (Imerys, 2024). Lithium is hosted mainly in mica-type minerals like lepidolite, and to a lesser extent amblygonite. Given the projected rise in global lithium demand, lepidolite is becoming an increasingly important source of lithium.

Recent studies [2-6] have demonstrated that flotation with cationic amine collectors, such as dodecylamine (DA), is the primary method for lepidolite concentration. The initial stage of this flotation process, desliming, is critical to mitigate the adverse effects of fine particles on collector efficiency and selective lepidolite flotation [2-3,7]. Optimizing collector dosage is also important, as excessive dosages can lead to unwanted recovery of silicate gangue minerals and dilute the final concentrate [2]. Evaluations of various

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commercial amine collectors, including Aeromine 3000C, Armac C and T, Armeen 12D (distilled DA), and Flotigam EDA (ether monoamine), revealed significant variability in flotation performance. For example, poor solubility and reduced froth stability were reported with Armeen 12D, while Flotigam EDA displayed a strong performance at acidic conditions (pH ~3.5) and produced a concentrate grading 4.0% Li_2O with approximately 77% lithium recovery [3,7]. Gemini collectors, characterized by dual hydrophobic groups, have indicated superior flotation efficiency compared to conventional DA, primarily due to stronger collector adsorption onto lepidolite surfaces. This enhanced performance was confirmed by contact angle and zeta potential measurements conducted by Wei et al. (2021), showing significantly improved lepidolite selectivity and flotation recovery.

The selective flotation of lepidolite from quartz and feldspar remains challenging, primarily due to their similar iso-electric points (IEP ~2-3). Consequently, flotation operations typically occur below pH 3 to ensure higher selectivity [8,9]. Huang et al. (2022) further demonstrated a clear decrease in concentrate grade when flotation pH increased from 2 to 9, affirming acidic conditions were optimal. Similarly, Filippov et al. (2022) observed peak performance at pH levels between 3 and 5, achieving lithium recoveries between 87% and 95% at concentrate grades up to 4.5% Li_2O. At high pH, flotation of unwanted silicate gangue increased, decreasing the concentrate purity. Recent advancements have aimed at overcoming issues related to high acid consumption and corrosion by exploring mixed collector systems. Wei et al. (2021) reported promising results using a collector blend termed "SOL" (sodium dodecyl sulfonate, sodium oleate, and DA), demonstrating effective flotation at neutral pH (pH 7) with high lepidolite recovery and minimal quartz contamination. However, these findings need validation through more comprehensive industrial-scale testing.

Overall, recent research has significantly advanced the understanding of lepidolite flotation processes, focusing on collector selection, dosage optimization, and innovative collector mixtures. Nonetheless, challenges in selective gangue rejection remain, emphasizing the need for further research into collector chemistry, optimization of flotation conditions, and pre-treatment steps such as desliming. These improvements will significantly enhance lepidolite beneficiation efficiency and contribute positively to meeting the growing global lithium demand.

EXPERIMENTAL

Materials

Drill samples representing twenty-one variability were collected from the Emili deposit (France). Eighteen samples represented the variability of ore included in the mine plan, and the additional three variability samples represented the materials out of the mine plan. Each sample was homogenized before being split into 4 kg charges and a representative head sample for assays and particle size analysis. To prepare the flotation feed, each charge was wet ground to a target size of 100% passing 300 µm. The ground material was then deslimed by wet sieving at 20 µm. The deslimed product was filtered, weighed, and split into 1.2 kg charges for flotation testing, and a representative subsample for particle size analysis. The slimes material (-20 µm fraction) was dried, weighed, and subsampled for chemical analyses.

Sample Characterization

The chemical analysis used in this study included lithium assays by Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) and whole rock analysis (WRA) with X-ray Fluorescence Spectroscopy (XRF). WRA included a 12-element suite for SiO_2, Al_2O_3, Fe_2O_3, MgO, CaO, K_2O, TiO_2, MnO, Cr_2O_3, V_2O_5, Na_2O, and P_2O_5.

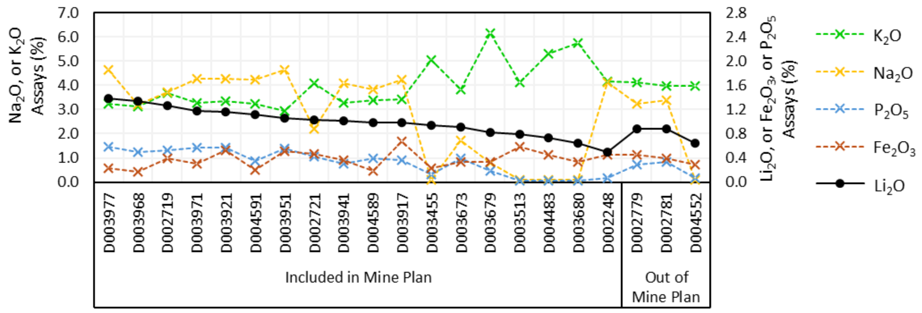

Figure 1 Key head assay results of the variability samples

The head assays of twenty-one variability samples (Figure 1) ranged from 0.49 to 1.38% Li_2O with low iron contents below 0.68% Fe_2O_3. The silica content varied from 66.5%-77.3% SiO_2 while aluminum contents varied from 13.8-20.5% Al_2O_3. The Na_2O and K_2O assays, which are mostly associated with mica and feldspar type minerals, ranged from 0.08-4.65% and 2.93-6.14%, respectively, while the P_2O_5 assays – mostly associated with phosphate minerals – ranged from 0.05-1.45%.

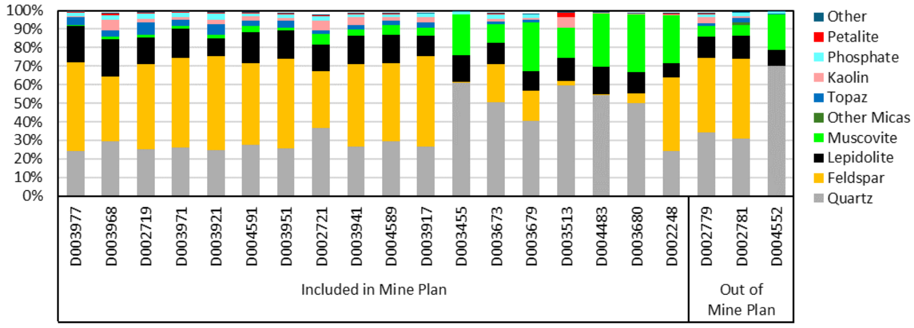

Figure 2 Modal mineralogy from MLA of the lepidolite flotation feed samples

The modal mineralogy of variability lepidolite flotation feed samples was determined with Mineral Liberation Analysis (MLA). The MLA results (Figure 2) confirmed that lepidolite was the main lithium-bearing mineral in all samples, at varying levels between 7.8 and 20.2%, which corresponded well with the Li_2O head assays. There was a good correlation between Li_2O and P_2O_5 content in the samples and phosphate minerals in the flotation feed (up to 4.0%), suggesting there is a potential association of phosphorus with lithium – perhaps in the form of the lithium-bearing phosphate mineral amblygonite

($LiAlFPO_4$). The major gangue minerals in the samples were quartz (24.1-70.0%), feldspars (0.3-50.6%), and muscovite (0.3-31.1%), with minor levels of topaz (<7.0%) and kaolin (<6.0%). In addition, the K_2O assays presented a stronger correlation with muscovite over lepidolite, where most variability samples containing over 3.8% K_2O contained over 10.2% muscovite.

The particle size analyses of the variability samples after stage grinding are presented in Figure 3. Prior to grinding, the particle size distribution was similar across the variability samples with the mass distribution in the -20 µm fraction ranging from a low level of 0.8% in sample D003679 to a high of 10.7% in sample D003513.

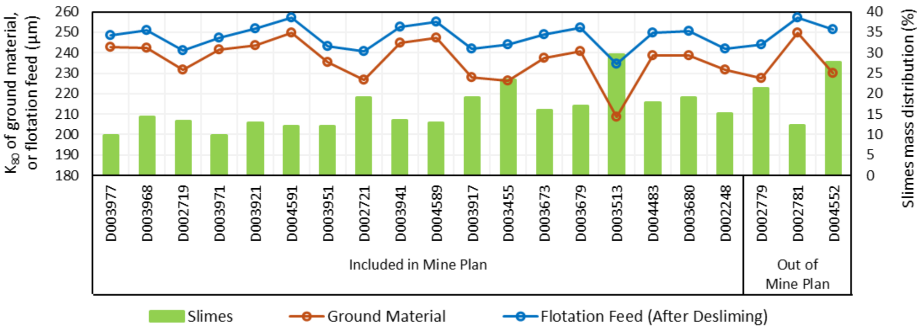

Figure 3 Particle size analyses of the variability samples after grinding

After grinding but before desliming, the K80 of the ground samples varied from 209 to 250 µm with an average of 236 µm, while after desliming the K80 of the flotation feed coarsened to between 234 and 257 µm with an average of 248 µm. The weight distribution of slimes after stage grinding ranged from 8.2 to 27.9% of the initial feed mass with an average of 10.9% for all samples. The range of fines production revealed that material across the deposit will exhibit different grinding behaviors and is an important consideration for plant design.

Flotation Testwork

The collector used for flotation was an amine collector with MIBC as a frother and H_2SO_4 as a pH modifier. Batch lepidolite rougher-cleaner flotation tests were performed on each of the twenty-one variability samples according to a flowsheet previously developed at SGS Lakefield with Imerys to produce 4.2% Li_2O lepidolite concentrate with maximum lithium recovery. Six tests included an additional scavenger flotation stage with an additional collector to maximize lithium recovery.

RESULTS AND DISCUSSION

Flotation Performance

The best lepidolite flotation performance for each variability sample was determined based on the highest lithium recovery to a combined concentrate grading over 4.2% Li_2O. For variability samples that did not meet the 4.2% Li_2O concentrate target the optimum performance was selected based on the final concentrate produced in the 2nd cleaner flotation stage. A summary of the performance evaluation is presented in Figure 4.

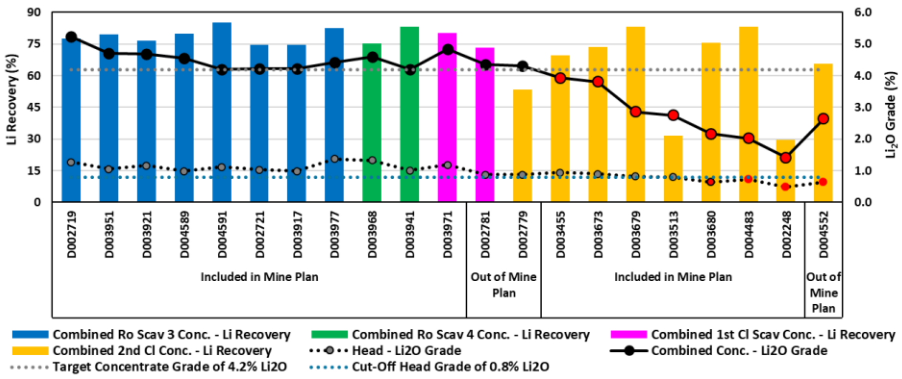

Figure 4 Particle size analyses of the variability samples after grinding

Twelve of the variability samples exhibited strong lepidolite flotation performance where concentrates grading over 4.2% Li_2O were produced with lithium recoveries over 73%. Eight of the twelve variability samples (shown in blue in Figure 4) met the concentrate target after 3 scavenger stages without any cleaning, to achieve over 74.6% lithium recovery. Two of the twelve variability samples (green) achieved included an additional fourth scavenging stage and additional collector to produce higher lithium recoveries of 75.4% and 83.2%, respectively, while also meeting the concentrate target without any cleaning stages. The two variability samples shown in pink in Figure 4 required one cleaner and cleaner scavenger stage to meet the target concentrate grade with lithium recoveries of 73.1 and 80.0%, respectively. The final sample to meet the 4.2% Li_2O was D002779, which required 2 cleaning stages and resulted in a lower lithium recovery of 53.3%.

The remaining Lepidolite concentrates were not able to meet the 4.2% Li_2O target and achieved varying performances. Samples D003455 and D003673 were able to approach the concentrate target at 3.93 and 3.81% Li_2O with strong global lithium recoveries of 69.6 and 73.5%, respectively. Meanwhile, the Li_2O content in the 2nd cleaner concentrates of the remaining six variability samples (shown in yellow in Figure 4) was below the 4.2% Li_2O target, varying from 1.42 to 2.86% Li_2O. In general, strong lithium recoveries were reported in most of these samples (>65.7%), except for variability samples D002248 and D003513 with 29.5 and 31.6% lithium recovery, respectively.

The increase in final concentrate Li_2O assay was strongly correlated (R^2 = 0.93) with a decrease in Al_2O_3 content and there was a moderate correlation between higher Li_2O assays and higher P_2O_5 and Na_2O content (R^2= 0.40-0.58), however, no correlation with K_2O content (R^2= 0.2) was observed (Figure 5). This indicates that variability samples producing concentrates below the target Li_2O grade also recovered mica gangue minerals during flotation, such as muscovite, since muscovite contains higher Al_2O_3 at a similar K_2O grade to lepidolite but contains little to no lithium. The strong relationship between muscovite in the flotation feed and Li_2O grade of the final concentrate shown in Figure 6 provides further support for this conclusion. Variability samples D003455, D003673, D002248, D003679, D003680, D003513, D004483, and D004552 all contained more than 10% muscovite in the flotation feed and did not meet the 4.2% Li_2O target, highlighting how selective rejection of non-lithium-bearing micas from lepidolite is challenging.

Meanwhile, P_2O_5 and Na_2O in the concentrates can possibly be attributed to amblygonite, which can also float amines to the lepidolite concentrate, but present no obvious impact on the concentrate Li_2O grade.

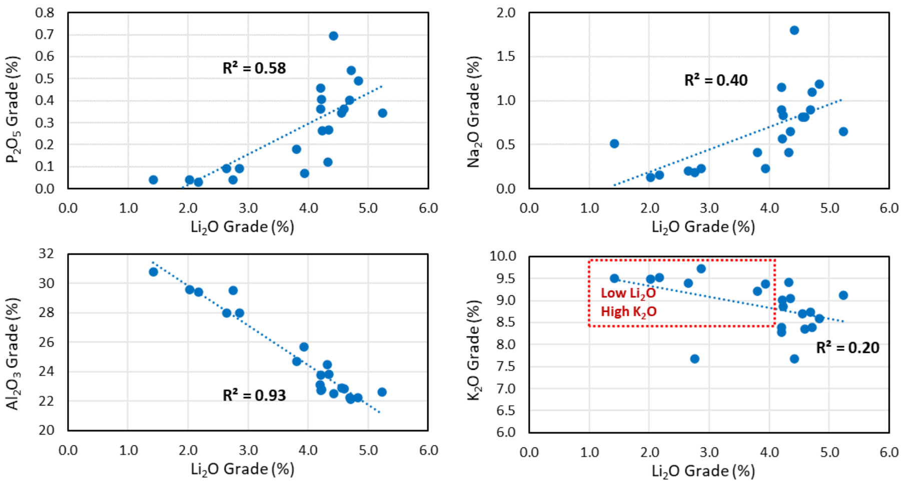

Figure 5 P_2O_5, Na_2O, Al_2O_3, and K_2O grades vs. Li_2O grade of the final concentrates

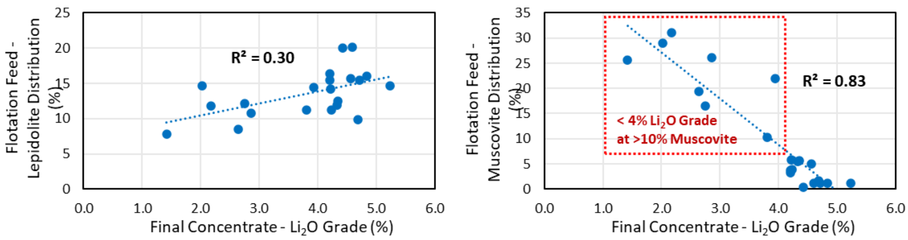

Figure 6 Li_2O concentrate grade vs. lepidolite and muscovite content in the feed

CONCLUSION

The results of this testwork confirmed that concentrates grading >4.2% Li_2O could be produced from thirteen of the twenty-one variability samples from the Emili deposit. For most of these samples, the target concentrate grade of 4.2% Li_2O was achieved without cleaner flotation stages at lithium recoveries greater than 74.6%.

The sample characterization revealed that the Li_2O and K_2O content in the 21 samples varied considerably, with the main lithium mineral being lepidolite and the main gangue minerals being quartz and muscovite. The slimes (-20 µm) distributions of the variability samples after wet grinding to 100% passing 300 µm were highly variable, suggesting the grindability characteristics vary across the Emili deposit.

There was a strong correlation between decreasing Li_2O grade in the final concentrates and increasing muscovite content in the flotation feed since muscovite is prone to float under the same conditions as lepidolite. This is most likely why the target grade of 4.2% Li_2O could not be met with the eight remaining variability samples. There was also some correlation between low concentrate quality and low lithium feed grades.

Overall, the variability flotation testwork confirmed that much of the Emili deposit was amenable to the production of a 4.2% Li_2O concentrate with high lithium recoveries. For samples where the concentrate targets were not met, poor flotation performance was attributed to higher gangue mica content and lower head grades, which will be an important consideration for project development and future operation.

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