

The Optimized Design on Spodumene Recovery from Iron Tailings in Kangding, Sichuan

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Abstract. In this paper, a flotation that implementing desliming in anticipation aimed at comprehensively recycling lithium from iron tailings in Kangding, Sichuan is put forward. Component of the iron tailings mainly with mica, limonite and chlorite which can result the serious sliming easily. Spodumene as the only desired mineral, the average grade of iron tailings is 0.76% Li_2O . After predesliming process, the combined modifying agent $\text{Na}_2\text{CO}_3\text{-NaOH}$ is used to inhibit mica, and the Laboratory-made collector QA-7 served as the collector to recycling spodumene. The results indicated that, via the Design-expert8.0 software to optimize the dosage of the regulators, the optimal dosage of $\text{Na}_2\text{CO}_3\text{-NaOH}$ and QA-7 are selected as 1700g/t and 800 g/t, respectively. Meanwhile, via the predesliming-one roughing-five cleaning-two scavenging and middlings back to the flowsheet in turn process, the Li_2O concentrate grade reached 5.71% with a recovery of 75.78% after a closed circuit experiment. In addition to realizing comprehensive utilization of iron tailings, the treatment is also a technical reference for handling the similar secondary resource.

1. Introduction

Lithium is a light metal, but also a rare metal, which has become an indispensable raw material for aerospace, nuclear energy, and battery manufacturing because of its many excellent properties. The raw materials for extracting lithium are mainly spodumene, lepidolite, amblygonite, petalite and zinnwaldite, of which spodumene is the main raw material for obtaining lithium [1-4].

China spodumene rich in resources, currently ranks second in the world, mainly in Sichuan, Xinjiang, Jiangxi, Hunan and other places. Spodumene, as a silicate mineral, is difficult to be chosen because of its complex composition and various structural types. In the actual classification of spodumene ore, spodumene is often associated with aluminosilicate minerals such as quartz, mica and feldspar, with similar surface properties and poor selectivity of the role of reagents. At present, flotation is the main separation method of spodumene. In the process of flotation operation, the stirring strength of the flotation machine, the temperature of the ore pulp and the ratio of the regulator have great influence on the flotation of spodumene. With the continuous exploitation of spodumene resources, high-grade spodumene deposits are gradually reduced. Therefore, it is of great significance



to strengthen the comprehensive recovery and utilization of complex refractory low-grade ores and secondary resources [5-6].

2. Test

2.1. Test samples

Spodumene ore is taken from a tailings of iron ore in Kangding, Sichuan province. The tailings contain low grade spodumene. The gangue minerals are mainly mica, quartz, magnetic (hematite), and chlorite and so on. The ore size is fine, and there is a certain degree of mud phenomenon. The tailings chemical multi-element analysis results in Table 1.

Tab. 1 Ore chemical multi - element analysis results

element	Li ₂ O	TFe	AL ₂ O ₃	MgO	CaO	K ₂ O	Na ₂ O	SiO ₂	S	P
content/%	0.76	8.57	12.54	0.55	0.62	2.63	3.11	65.93	0.01	0.01

The results of chemical multi-element analysis in Table 1 show that the content of Li₂O in the ore is relatively low, only 0.76%. The main impurities are some silicate minerals, which can be separated by flotation. The harmful impurities S and P in ores are low and need not be treated separately.

2.2. Equipment and Reagents

XMQ-240×90 sgrinding machine, Changsha shunze Mining Machinery Manufacturing Co.Ltd; -0.074 mm standard analysis sieves, Hangzhou Fuqiang Chemical Equipment Co.Ltd; XFG type hanging cell flotation machine, Ganzhou Fubang Metallurgical Machinery Co.Ltd; XTLZφ260/φ220 type vacuum filter, Jiangxi Industrial Machinery Manufacturing Co.Ltd; HZF-A300 type electronic balance Shanghai Tian precision instrument equipment Co. Ltd; elutriation device, laboratory self-made.

The collectors used in flotation test were the laboratory-made QA-7 (oxidized paraffin soap 733 and hydroxylamine hydrochloride mixed by mass ratio 4:1); sodium carbonate and sodium hydroxide were used as adjustment agents; the use of sodium carbonate added first plus sodium hydroxide, the amount of about 2:1. Oxidized paraffin soap 733, industrial grade, Kunming liangfan Technology Co.Ltd; hydroxylamine hydrochloride, analysis of pure, Hangzhou bangyi Chemical Co.Ltd; sodium carbonate, analysis of pure, Kaifeng Dongda Chemical Co.Ltd; sodium hydroxide, analysis of pure, Shanghai Avision chemical plant.

2.3. Test methods

In view of the fine granularity characteristics of the iron tailings, the principle of the first desliming and then flotation process, via the Miscellaneous module in design-expert 8.0 software to optimize the dosage of collector QA-7 and modifier that affecting froth flotation, get rough selection of the best reagent system, then the open circuit test, closed-circuit test inspection and improvement test plan. 500g ore each time for testing, open-circuit and closed-circuit test products were the superposition of the corresponding products of the five repeated tests, and the test samples were sent to Kunming Metallurgical Research Institute for analysis after drying and grinding.

3. Results and discussion

3.1. Raw ore screening test

Tab. 2 Fineness distribution of ore after grinding

Granularity level / μm	Yield/%	Li ₂ O	
		Grade/%	Distribution rate/%
150	8.49	0.97	10.88
-75	36.94	1.03	50.06
-37	29.57	0.82	31.9
-28	13.35	0.25	4.39
-10	11.65	0.18	2.76
Total	100	0.76	100

According to the distribution of ore fineness given in Table 2, it is known that the silicate minerals in the raw ore are mostly brittle and easy to grind, and these minerals are eventually enriched in the tailings, resulting in certain mudslides in the tailings. If the iron ore tailing is directly enriched by flotation of spodumene, the slime in the slurry forms a cover on the surface of the spodumene to deteriorate the flotation behavior. Therefore, it is considered that the ore is first subjected to elutriation treatment, can be used on industrial cyclone desliming.

3.2. Elutriation mud test

In order to reduce the adverse effects of slime on flotation, while reduce the dosage of ore flotation supply, raise the grade of Li₂O, desilter the -38 μm part after grinding, use the elutriation device in the laboratory desilter is shown in figure 1.

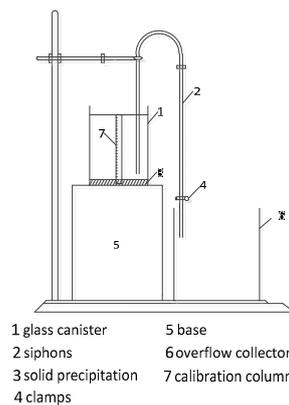


Fig. 1 Schematic diagram of elutriating apparatus

The settling time is calculated using the Stokes formula and the simplified Stokes formula is [7]:

$$d = \sqrt{\frac{h}{545(\rho_s - 1000)t}} \quad (m) \quad (1)$$

In the formula (1): d -spherical solid particles, $d = 3.8 \times 10^{-5} \text{ m}$; h -settlement distance, $h = 0.18 \text{ m}$; ρ_s -true proportion of solid materials, $\rho_s = 3100 \text{ kg/m}^3$.

The calculated washing time is 103.96s. Ultimately remove products smaller than 38 μm , to obtain flotation of 0.94% Li_2O grade ore, the metal distribution was 92.85%.

3.3. Roughing response surface optimization test

Flotation is a complex physico-chemical process. The dosage of the reagent has a great influence on the flotation results. At the same time, there must be interaction between different reagents. In order to achieve a holistic control of the flotation process, the Miscellaneous module in the design-expert software was used to analyze the interaction between the dosage of collector QA-7 and modifier in the flotation process to determine the best flotation pharmaceutical system. The collector used in flotation operations is a mixture of paraffin wax soap 733 and hydroxylamine hydrochloride in a mass ratio of 4:1, which utilizes the selective chelation of the oxirane group of hydroxylamine hydrochloride to the active site of lithium and aluminum on the mineral surface selective capture of spodumene with, oxidized paraffin wax soap. Test using sodium carbonate and sodium hydroxide, when used first added sodium carbonate plus sodium hydroxide to adjust the pulp pH, while inhibiting gangue minerals, the amount of about 2:1. Crude selection of the test process shown in Figure 2, according to experience choose the amount of roughing regulator dosage for the sum of 1000~2000g/t, collector QA-7 dosage range of 500~1000g / t, flotation test factors and levels of the table 3.

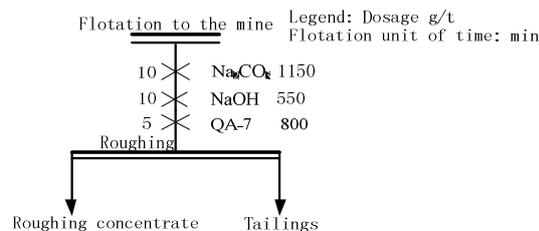


Fig. 2 Roughing test flow chart

Tab. 3 The influence factor and level of reverse flotation

Level	Adjuster dosage X_1 (g/t)	Collector dosage X_2 (g/t)
1	1000	500
2	1500	750
3	2000	1000

The flotation test in Table 3 factors and levels into the design-expert software program to get the test program, according to the test program design test, and then enter the test results into the system to get the optimal model and the best test parameters.

3.3.1. Analysis of variance

Tab. 4 Analysis of variance of each factor

Source	Grade					Rate of recovery				
	Sum of squares	Degree of freedom	Mean square	F-value	P-value	Sum of squares	Degree of freedom	Mean square	F-value	P-value
Model	0.514892	5	0.102978	93.38086	0.0017	145.7188	5	29.14376	613.218	0.0001
X ₁ -X ₁	0.260417	1	0.260417	236.1461	0.0006	17.34	1	17.34	364.8535	0.0003
X ₂ -X ₂	0.22815	1	0.22815	206.8866	0.0007	109.7393	1	109.7393	2309.04	<0.0001
X ₁ X ₂	0.000225	1	0.000225	0.68213	0.204	1.7424	1	1.7424	36.66209	0.0009
X ₁ ²	0.02205	1	0.02205	19.9496	0.00208	0.728022	1	0.728022	15.31842	0.0296
X ₂ ²	0.00405	1	0.00405	3.672544	0.1512	16.16909	1	16.16909	340.2162	0.0003
Residual	0.003308	3	0.001103	—	—	0.142578	3	0.047526	—	—
Cor total	0.5182	8	—	—	—	145.8614	8	—	—	—

As can be seen from Table 4, the F values of the model obtained from the design-expert software fitting the experimental data is both greater than the P value. The variance results show that the model is significant and can provide theoretical reference for the experiment.

3.3.2. Regression equation. The second-order linear regression equation of the model obtained from design expert software using ANOVA was:

$$Y_{\text{grade}} = 1.83 + 0.21 \times X_1 - 0.19 \times X_2 - 0.0075 \times X_1 \times X_2 - 0.11 X_1^2 - 0.045 X_2^2,$$

$$Y_{\text{Recovery rate}} = 89.42 - 1.70 \times X_1 + 4.28 \times X_2 - 0.66 \times X_1 \times X_2 - 0.60 X_1^2 - 2.84 X_2^2$$

3.3.3. Response surface contour map. The contour plots of the response surfaces for the amount of collectors and the amount of inhibitors obtained after fitting the experimental data are shown in Fig. 3

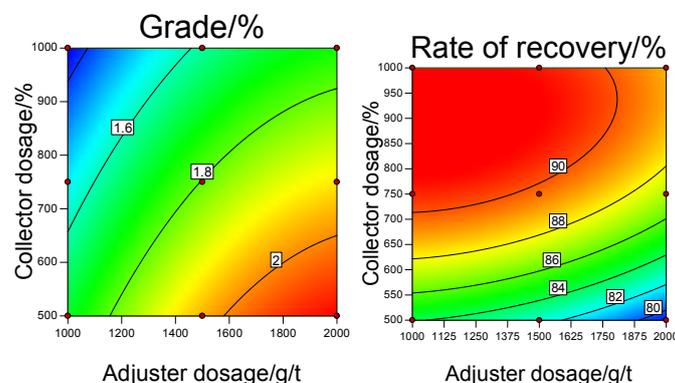


Fig. 3 Effects of flotation inhibitor dosage and collector dosage on Li₂O grade (A) and recovery (B)

Optimized by the software of Fig.3, taking into account the highest grade and recovery rate, the best choice is 791 g/t of collector and 1701 g/t of inhibitor. At this moment, the result of model fitting showed that the Li_2O grade was 1.86% and the recovery rate was 89.30%.

According to the optimum amount of collector and inhibitor dosage, the amount of collector 800 g/t and the amount of inhibitor 1700 g/t were determined for convenient production practice. Finally, a section of rough-grade phosphate concentrated Li_2O grade 1.94%, the recovery rate was 88.94%.

3.4. Open test

In order to increase the Li_2O content of flotation concentrates and increase the recovery rate of Li_2O , the open circuit test was conducted to determine the best selection and sweep times. The test flow is shown in Figure 4, the test results are shown in Table 5.

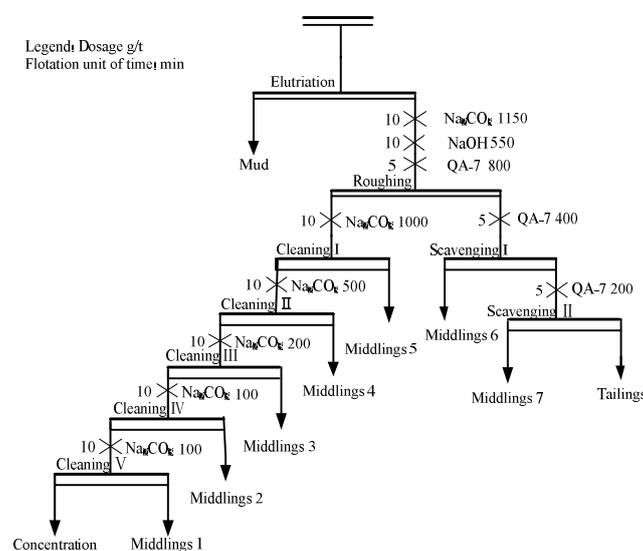


Fig. 4 Open test flow chart

Tab. 5 Open circuit test results

Product	Grade/%	Rate of recovery/%
Mud	0.22	7.15
Concentrate	6.07	60.96
Middlings 1	3.01	0.7
Middlings 2	2.72	1.79
Middlings 3	1.67	2.58
Middlings 4	1.23	3.97
Middlings 5	0.78	5.08
Middlings 6	0.63	3.93
Middlings 7	0.58	1.04
Tailings	0.19	12.8
Total	0.76	100

From the open test results in Table 5, it can be seen that after the ore of -0.038mm is removed from the ore, an open process of "one crude five-fine two-sweep" can be used to finally obtain the Li_2O grade of lithium concentrate of 6.07% and the recovery rate of 60.96%. To meet the needs of further processing of lithium, while tailings Li_2O grade dropped to 0.19%, plus sweep election spodumene has no need.

3.5. Closed circuit test

According to the results of the open-circuit test, the "closed circuit" process of "one rough five-fine two-sweep" and medium-sized mine circulation loop is determined. In order to verify the effect of the process, a closed-circuit test is conducted. Test process shown in Figure 5, multi-element flotation product analysis results in Table 6.

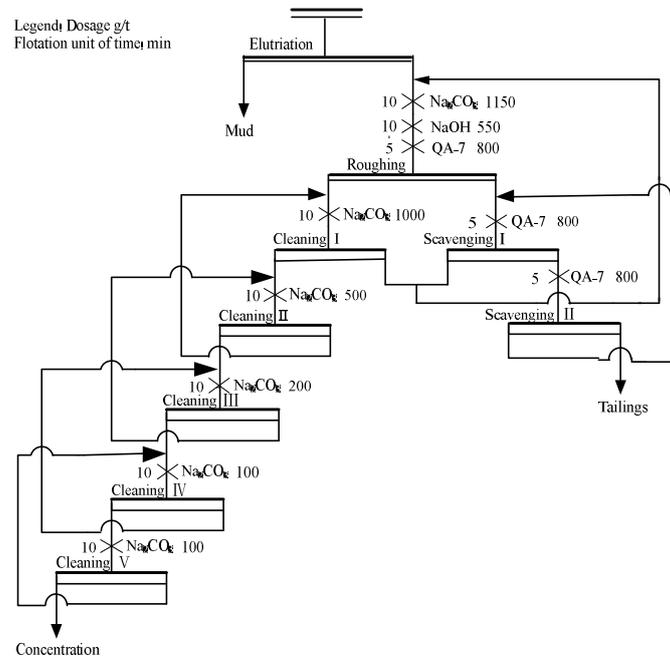


Fig. 5 Closed-circuit test flow chart

Tab. 6 Results of chemical multi - element analysis of flotation products

Product	Grade/%	Rate of recovery/%
Mud	0.22	7.15
Concentrate	5.71	75.78
Tailings	0.2	17.07
Raw ore	0.76	100

As can be seen from the results of the closed-circuit test in Table 6, the Li_2O grade of 5.71% and the recovery rate of 75.78% can be obtained by using the closed circuit flow of "one rough five-fine two-sweep" to achieve the comprehensive utilization of the tailings' resources.

4. Conclusion

(1) An iron ore tailing in Kangding, Sichuan Province contains 0.76% Li_2O . The experiment is to enrich the spodumene by beneficiation using deslagging and re-flotation, and then realize the comprehensive utilization of lithium resources.

(2) Sodium carbonate, sodium hydroxide as a regulator to inhibit mica, laboratory-made collector QA-7 as collector recovery spodumene, based on the previous single-factor flotation experience on the basis of the determination of the approximate range of pharmaceutical dosage, Flotation index of the main factors collected the amount of collectors does response surface optimization test, and ultimately determined the amount of flotation's coarse collector 800 g/t, the amount of inhibitor 1700 g/t.

(3) A closed-loop process with pre-elutriation, primary rougher selection, five-time selection, second-time sweep selection and mid-ore recycling was adopted to finally obtain to concentrate with

Li₂O grade of 5.71% and Li₂O recovery rate of 75.38%, which improved resource utilization level, for the comprehensive utilization of iron tailing's resources provide technical support.

Acknowledgments

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