Characterisation and Metallurgical Testwork of Lithium and Rubidium Ore

Purev-Ochir Togtokhbaatar[1*], Baasandorj Myagmarsuren[1], Battsengel Baatar[1]

[1] Faculty of Raw Materials and Environmental Engineering, German-Mongolian Institute for Resources and Technology (GMIT), Nalaikh District, Ulaanbaatar 12800, Mongolia
purevochir@gmit.edu.mn

Abstract. This study investigates the characterization and processing of lithium (Li) and rubidium (Rb) ores from a new deposit using multiple analytical techniques. For mineral characterization, Mineral Liberation Analysis-Scanning Electron Microscopy (MLA-SEM), X-ray Powder Diffraction (XRPD), and X-ray Fluorescence (XRF) were used. Metal quantification was done by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) and Li and Rb concentrations were determined 0.6% and 0.39%, respectively. The presence of fluorine in the ore was detected, hence a calcination process was used to enhance metal extraction and remove fluorine gas. Subsequently, leaching experiments were conducted at various temperatures with diluted sulfuric acid to compare their metal recovery. The study shows that calcination, particularly at 850°C, significantly improves minerals alteration, while the choice of leaching ligand influences extraction efficiency. The resulting high-concentration Li and Rb salts indicate potential for further refinement. To enhance the processing method, future work will explore electrodialysis to purify and concentrate the leaching solution, aiming to improve recovery rates and reduce environmental impact. This integrated approach, combining advanced characterization, calcination, and leaching, provides for the efficient extraction of Li and Rb from natural ores, with implications for sustainable resource recovery in the context of increasing global demand for critical metals.

Keywords: Li and Rb ore, Spodumene, Petalite, Calcination, Leaching.

1 Introduction

Lithium (Li) is one of the most in-demand and important elements of the 21st century due to its use in electric vehicles and battery technologies in various electronics [1]. The European Union has identified it as one of 34 strategically important elements [2]. In addition, developed countries are also prioritizing lithium (Li), with the Republic of Korea naming it one of 33 critically important elements.

In Mongolia, lithium (Li) occurs in three main deposit types: deposits associated with granitoid-pegmatites, lithium-rich clay deposits, and salt (lithium brine) deposits [3]. Furthermore, while rubidium is not as critical as lithium, it is an alkali metal with

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specific applications such as in atomic clocks, research into Bose-Einstein condensates, and deep violet-colored fireworks [5]

The importance of lithium is linked to its role in electric vehicle technology and renewable energy storage, with global demand projected to exceed 3 million tons of lithium carbonate by 2030[4]. Currently a net importer, Europe primarily sources its lithium from Chile, China, and the USA; however, projects in countries like Portugal and Austria are aimed at increasing domestic production. Rubidium (Rb) is also gaining interest as a byproduct.

Lithium and rubidium ore processing focuses on hard-rock ores such as spodumene, lepidolite, petalite, and zinnwaldite, which are found in Australia, Canada, and Europe. The process involves mining, crushing, and chemically extracting the lithium through methods like sulfuric acid roasting, alkaline roasting, or chlorination, from which rubidium (Rb) is often recovered as a byproduct [5].

In this study, we characterized ore samples containing lithium and rubidium. The analysis and processing were conducted as follows: Mineralogical analysis was performed using MLA-SEM (Freiberg University of Mining and Technology, Germany) and XRD (National University of Mongolia). Chemical composition was determined using ICP-AES (ALS) and XRF (Niton Gold II - GMIT) instruments. The ore was processed using traditional methods such as gravity, magnetic, and flotation separation but those technologies were not sufficient. However, roasting and diluted sulfuric acid leaching showed economically feasible results.

2 Materials and methods

2.1 Analysis of lithium and rubidium ore

Lithium and rubidium ore was provided by a local mining company. This ore extracted from the surface of the pegmatite ore deposit. The elemental composition was determined by the Inductively coupled plasma atomic emission spectroscopy (ICP-AES) according to the method of ME-ICP61a at the ALS Global standard Laboratory and KhanLab laboratory. The elemental compositions of two samples are given in Table 1.

Table 1. *Elemental composition of Li and Rb ore by ICP-AES*

Elements	ALS Global, ppm	Khanlab, ppm
F	>20000	—
Al %	8.39	—
Ba	450	188.6
Ca %	1.21	1.18
Cu	12	64.4
Fe, %	2.29	2.26
K %	5.9	6.3
Li	**6590**	**6481**

Mg %	0.26	0.22
Mn	577	516
Na %	2.5	2.5
Pb	97	56.6
Sr	125	94.9
Ti	1000	469
Zn	257	239.1
Rb	**3550**	**3136**

2.2 Mineral characterization of Li and Rb ore

The phase composition of the samples was determined by X-ray powder diffraction (XRD) using a Rigaku Miniflex 300/600 diffractometer equipped with a D/teX Ultra2 detector. Measurements were carried out in Bragg–Brentano geometry with Cu $K\alpha_1$ radiation (λ = 1.5406 Å). The samples were prepared as flat, plane specimens to ensure optimal diffraction conditions. Data were collected over a 2θ range of 5–70°, with a step size of 0.02° and a counting time of 8 s per step. The resulting diffractograms are presented in Figure 1. The ore sample was found to contain quartz (SiO_2), albite ($NaAlSi_3O_8$), muscovite [$KAl_2(F,OH)_2$], and K-feldspar ($KAlSi_3O_8$). Phase identification was performed using Match 5.0 (Crystal Impact) based on the chemical composition of the ore determined by ICP-AES.

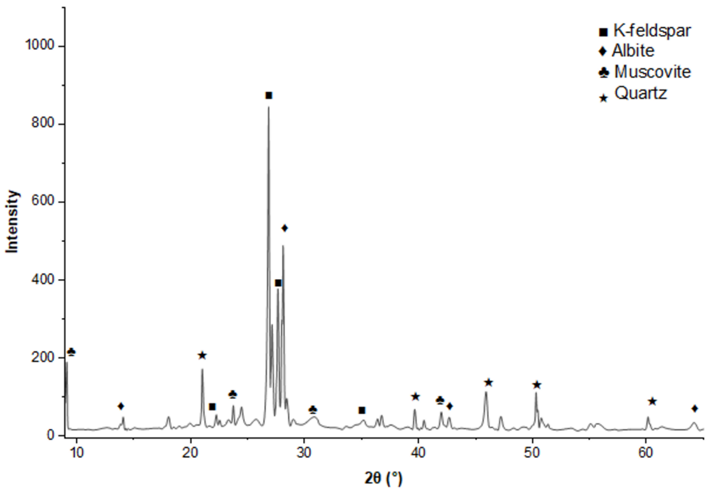


Fig. 1. Powder XRD patterns of the Li and Rb ore

To confirm and quantify the mineralogical composition of the concentrates, mineral liberation analysis (MLA) was carried out. The analyses were conducted using a Quanta FEG 600 scanning electron microscope equipped with a field emission gun as the electron source, two energy-dispersive X-ray (EDX) silicon drift detectors

(SDD) (Bruker Quantax 200), two Dual XFlash 5030 EDX detectors (Bruker), and a backscattered electron (BSE) detector. The acquired data were processed using Dataview 2.9.0.7 software. The identified mineral phases in the Li- and Rb-bearing ore are summarized in Table 2. Lithium was primarily associated with spodumene [$LiAl(SiO_3)_2$] and petalite ($LiAlSi_4O_{10}$), while MLA-SEM analysis revealed that rubidium occurs within muscovite [$KAl_2(AlSi_3O_{10})(OH)_2$] (see Table 3 for details).

Table 2. *Mineral composition of concentrates determined by MLA-SEM*

Minerals	Chemical formula	Weight, %	Area, %	Area, µm
Spodumene	$LiAl(SiO_3i_2$	0.34	0.28	182057.86
Petalite	$LiAlSi_4i_{10}00.27$	0.3	190346.31	
Quartz	SiO_2	5.8	5.85	3757456.49
Albite	$NaAlSi_3a_8$	25.68	25.96	16671190.5
K-feldspar	$KAlSi_3O_840.12$	41.52	26657005.8	
Muscovite	$KAl_2AAlSi_3A_{10}$ $0AlSi_2$	22.73	21.28	13662826.1
Andalusite	Al_2SiO_5	3.66	3.08	1978599.09
Other minerals	-	1.4	1.73	-
Total		100	100	64208568.2

Table 3. *Li and Rb containing minerals in the ore by MLA-SEM*

Mineral	Density	Formula	Li (%)
Spodumene	3.15	LiAl(Si2O6)	3.85
Petalite	2.39	LiAlSi4O10	2.27
Mineral	**Density**	**Formula**	**Rb (%)**
Muscovite	2.83	KAl3Si3O10(OH)2	1.7

Figure 2 the mineral associations within the ore. Most of target minerals- spodumene and petalite- are interlocked in the muscovite, albite and K-feldspar. In this figure spodumene and petalite mineral particles are highlighted by red rectangles. The quantitative analysis indicates that approximately 12% of petalite and 22% of spodumene are present as liberated particles.

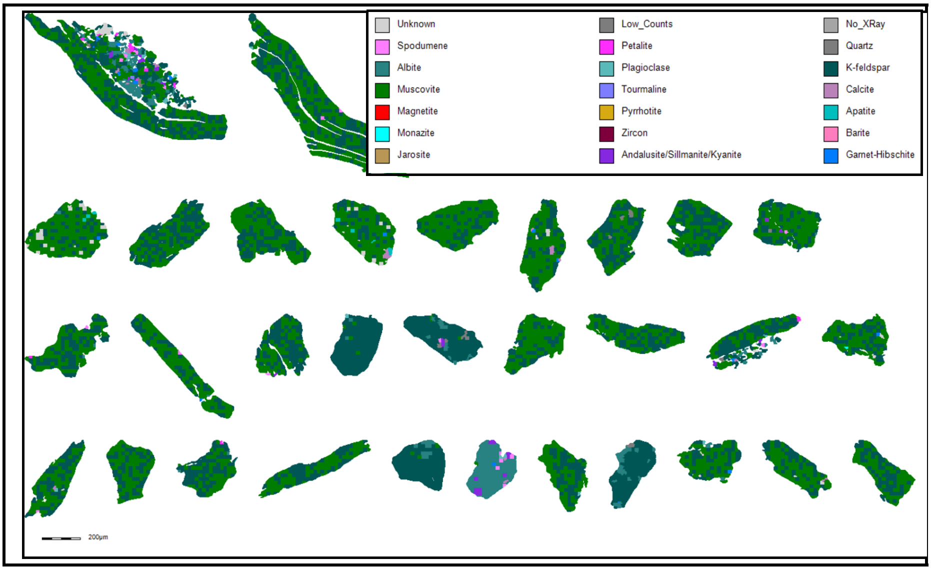
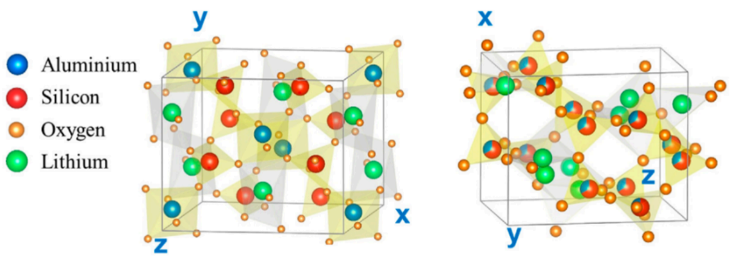


Fig. 2. *Mineral composition maps which present interlocked and free spodumene and gangue mineral grains in the different size fractions of the Li and Rb ore by MLA-SEM*

2.3 Experimental leaching procedure

To prepare the Li- and Rb-bearing ore for the leaching experiments, the material was first crushed using a laboratory jaw crusher (XJT jaw crusher) and cone crushers to achieve a particle size of < 2 mm. The crushed material was then ground in a laboratory ball mill for 15 min, yielding a product with P80 = 74 µm. Prior to leaching, fluorine was removed from the ore to prevent hydrofluoric acid (HF) formation during processing. Therefore, the samples were subjected to calcination for 1 h. During calcination, spodumene transforms from the α- (alpha) to the β- (beta) phase, as illustrated in Figure 3. Various calcination temperatures in the range of 800–1100 °C were investigated.



(a) α-spodumene **(b)** β-spodumene

Fig. 3. *Spodumene conversion (a) α-spodumene to (b) β-spodumene and its crystal structures [6]*

At temperatures above 900 °C, the milled ore melted, forming a glassy phase that was difficult to regrind. Therefore, a calcination temperature of 850 °C was selected. After cooling, the calcined Li- and Rb-bearing ore was subjected to leaching experiments (see details in Figure 4). The reagents and experimental conditions used in this study are summarized in Table 4.

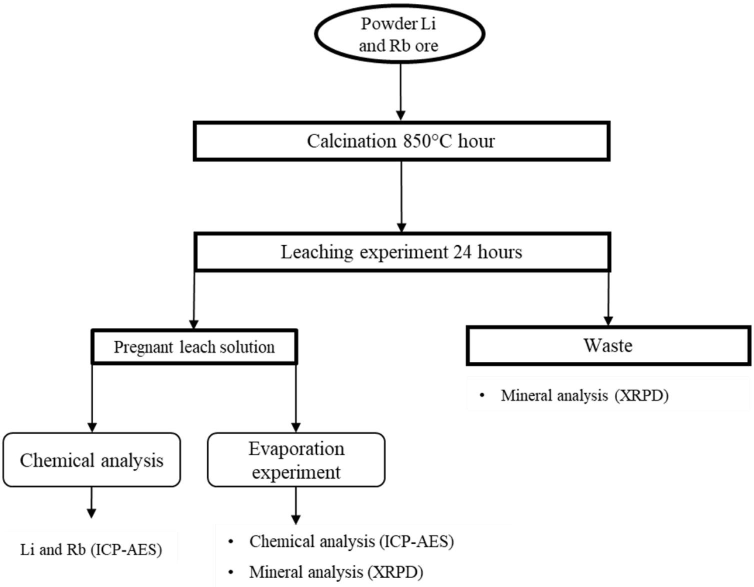


Fig. 4. *Schematic diagram of Li and Rb ore processing*

Table 4. *Sources and purities of chemicals and minerals used in the leaching experiments*

Chemicals	Purity	Source
Calcium chloride	≥ 98 %	Xilong Scientific Co., Ltd.
Sulfuric acid	98 %	Xilong Scientific Co., Ltd.
Sodium sulfate	≥ 99 %	Xilong Scientific Co., Ltd.

For the leaching experiment, 200 ml of 1M sulfuric acid was placed in sealed glass flask and stirred on a heated magnetic stirrer at 20°C, 50°C and 70 °C (see Figure 5 for details). Once the desired temperature was reached, approximately 10 g of the Li- and Rb-bearing ore was added to the flask. The leaching process was carried out for

24 h at a stirring speed of 400 rpm. During the leaching experiment, 2 ml samples were taken at 0, 2, 4, 6 and 24 hours to track Li and Rb dissolution. Samples were taken via 10 ml pipette and filtered through a 40 µm paper filter. For the quantification of the elements in leaching solution, samples were diluted by the factor of the laboratories' standard. The concentrations of Li and Rb in the leachate were determined using the ICP-AES.

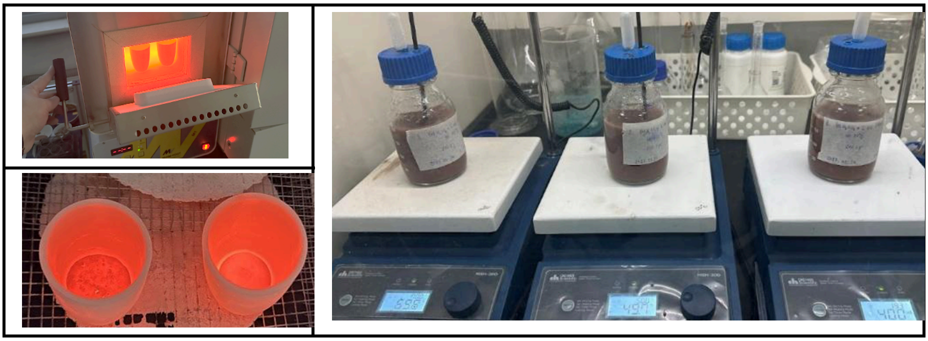


Fig. 5. *Calcination of Li and Rb ore at 850 °C and its leaching experiment with 1M sulfuric acid*

3 Result and discussion

After calcination of the Li and Rb bearing ore at 850 °C for an hour, XRD was carried out and results of original sample and calcinated samples are compared (see Figure 6). Overall, no significant changes were observed, however a new peak appeared at approximately 36°, which may indicate the formation of a desired mineral phase (α→β spodumene $LiAlSi_2O_6$) resulting from the calcination process.

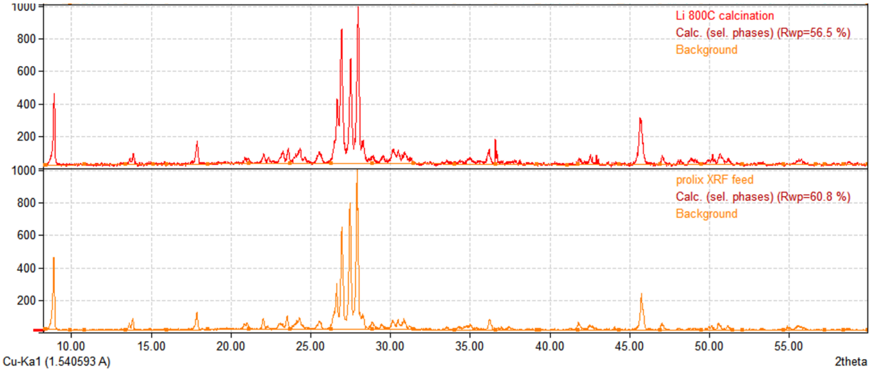


Fig. 6. *Comparison of XRD patterns between calcinated ore at 850°C and original ore*

The Leaching experiment described in the previous section was performed to investigate the feasibility of leaching of Li and Rb with diluted sulfuric acid. The cumulative experimental errors was estimated to be approximately 5%. A 10 g of calcinated ore sample was leached in 200 mL of 1M H_2SO_4 at 20 °C, 50 °C and 70 °C respectively. Temperature was found to play a critical role in the dissolution behavior of both Li and Rb. Figure 7 shows the leaching result of Li (left) and Rb (right) ore after calcination at 850 °C. As shown, Li dissolution strongly depended on temperature: Li recovery reached about 40% within the first 6 hours and exceeded 90% after 24 hours at elevated temperatures. In contrast, leaching at 20 °C and 50 °C yielded significantly lower recoveries and would be considered economically unfavorable. A similar trend was observed for Rb, with recovery exceeding 40% within 6 hours and reaching approximately 80% after 24 hours. These results collectively confirm that increasing the slight temperature changes significantly enhanced the leaching rate for both elements (Li and Rb), a kinetic dependency that is consistent with the Arrhenius equation. Again, the lower-temperature conditions proved less effective and uneconomical.

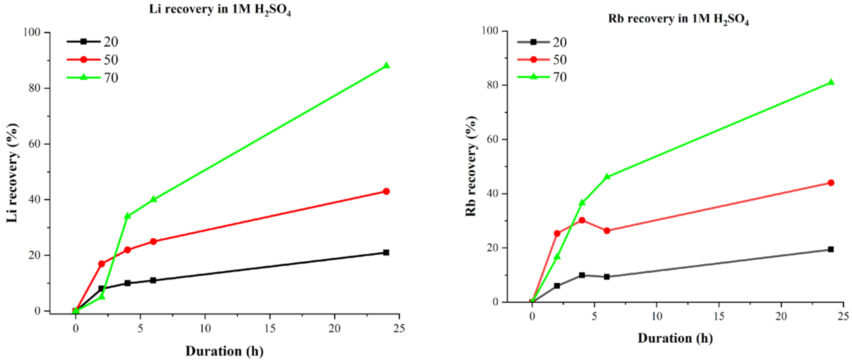


Fig. 7. *Comparison of metal recovery at various temperatures of Li (left) and Rb (right)*

The trench ore sample proved to be relatively easy to process using a combination of calcination and diluted sulfuric acid leaching. However, further investigations are required at a larger laboratory or pilot scale to confirm process feasibility and optimization. Once Li and Rb are dissolved into the leach solution, they can be separated through several sequential purification and precipitation steps. Specifically, Li can be recovered as Li_2CO_3 by precipitation with Na_2CO_3, while Rb can be extracted by various methods like solvent extraction, crystallization and precipitation as form Rb salts, as described in the works of [7][8][9].

4 Conclusion

In this research work, we introduce first part of ongoing research of Li and Rb ore processing at the German-Mongolian Institute for Resources and Technology. We determined Li (spodumene 0.34%, petalite 0.27 %) and Rb (muscovite 22.67 %) bearing minerals, its association and gangue minerals. Also, tested various temperatures for ore calcination and 850 °C for 1 hour was sufficient enough. Afterwards, various temperature tested (20 °C, 50 °C and 70 °C) with 1M sulfuric acid. Elevated temperatures enhanced the Li and Rb dissolution significantly. At 70 °C, Li recovery reached approximately 90 % and Rb recovery reached 80 % within 24 hours. Future work will therefore focus on exploring alternative processing media such as chloride and alkaline media, as well as salt-roasting and leaching combinations, to further improve recovery rates and reduce environmental impact. Additional studies on solution purification and selective precipitation potentially through electrodialysis or ion-exchange techniques will be conducted to obtain high-purity Li_2CO_3 and Rb products. Overall, this research provides a promising foundation for developing a sustainable and efficient process for the extraction of Li and Rb from Mongolian resources, contributing to the strategic supply of critical metals essential for the global energy transition.

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