

Development of an Optimal and Environmental Friendly Process for Extracting Lithium from Manono Using a Low-Temperature Digestion Method

Mukendi Papy Musampa¹, Bu Kweto¹, Tresor Lukusa², John Kabuba¹

Abstract—The metallurgical treatment of lithium ore generally leads to the production of lithium carbonate, which can be used in a wide range of industries, including batteries, ceramics, glass, cement, additives, and medicine. The lithium extraction process requires calcination at 1100 to 1200°C to change the phase from α to β , which is very energy-intensive and pollutes the environment. The aim of this study was to investigate the possibility of using ammonium bifluoride during the digestion of Manono spodumene ore without the conversion from α to β phase, to produce high-purity lithium carbonate. The processing of this ore begins with crushing, grinding, and digestion with ammonium bifluoride, followed by acid leaching, purification of the solution, and precipitation of lithium carbonate (Li_2CO_3). The lithium ore came from AVZ Manono in the Kitotolo mine. Chemical analysis revealed that the average Li_2O content was 7.6%, while mineralogical analysis indicated that the ore contained 25.5% spodumene and predominantly aluminosilicate gangue minerals. Digestion with ammonium bifluoride (NH_4HF_2) converted α -spodumene to lithium fluoride (LiF). The optimum conditions are 90 minutes, 150°C, and 1/22 molar ratio of spodumene, ore, and ABF, resulted in a conversion yield of α -spodumene to lithium fluoride (LiF) of 97%. The sulphuric acid leach applied extracted lithium under the following optimum conditions: 120 minutes, 30°C, and 33.3 g/L and 360 rpm. The leaching yield under these conditions was 98%. The Li_2SO_4 solution after leaching was purified by selective precipitation. The following products were used for precipitation: sodium carbonate and calcium hydroxide. Finally, lithium carbonate was precipitated by adding a sodium carbonate solution at a pH of 11.

Keywords—Ammonium bifluoride, Digestion, Leaching, Lithium, Spodumene

I. INTRODUCTION

The advent of mobile telephony and batteries for electric cars are major challenges for the 21st century. These technological developments aim to respond to the major climatic problems of our era and to reduce the consumption of

fossil fuels by developing sustainable energy sources. Today, around 80% of energy consumption comes from fossil fuels, which produce carbon dioxide (CO_2). Coal, oil and natural gas are the main sources of CO_2 emissions worldwide. A dangerous trend with harmful environmental impacts that global players are proposing to turn away from via the fascinating optics of the energy transition [1]. Polluting, carbon-based or risky energies (fossil and nuclear) no longer reassures the world's best future [2]. It has therefore become more than imperative to gradually replace them with clean, safe renewable energies (hydraulic, tidal, geothermal, wind and solar). This should ostensibly contribute to the goals of sustainable development [3]. Lithium has found applications in many industries. Its greatest contributions to the materials industry are in glassmaking, ceramics and lithium-ion batteries. However, the most important application of Lithium is focused on the development of Li-ion batteries. Lithium is also mainly used in electrode materials and as an electrolyte [4]. Due to its low density and high electrochemical potential, lithium has been found to be an effective strategic solution for the automotive sector. Reducing the carbon footprint responsible for global CO_2 emissions by 9% in 2022. Electric vehicles offer a sustainable alternative to environmental and industrial problems by using a natural resource that has become scarce, while promoting eco-economic thinking based on sustainable development in the production of vehicles with low environmental impact [5]. Lithium can be found in the form of lithium compounds such as lithium carbonate (Li_2CO_3), lithium chloride (LiCl) and lithium fluoride (LiF). Lithium carbonate is by far the most widely used precursor in the Li-ion battery and lithium metal production industries [6]. In the literature, numerous studies on the production of lithium carbonate-based precursors from spodumene propose lithium extraction using a process based on high-temperature (1100°C), chemical roasting with sulfuric acid, followed by leaching with water and hydrochloric acid or sulfuric acid. Once the pulp containing solubilized lithium has been obtained, it is purified using calcium sulfate and calcium hydroxide (limestone). After the purification process, the lithium contained in the enriched solution is precipitated as lithium carbonate using sodium carbonate as the precipitating

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agent [4]. These processes, the most widespread, are used to transform lithium minerals or concentrates into a high-purity, high-value-added soluble form such as lithium carbonate of 99.9% purity [7]. The most popular process, acid roasting, requires a prior phase transformation of α - spodumene into β - spodumene at high temperature (1100°C). This transformation, obeying Avrami's kinetic law, results in a mineral species (β - spodumene) that may be soluble in sulfuric acid, but at a relatively high temperature of 250°C. This is followed by a water leaching step at a temperature of 90°C [7]. Three criteria dictate the choice of acid to be used: its availability on the market, its cost and ease of use. For this purpose, the use of sulfuric acid is preferable to that of Hydrochloric acid, in the above-mentioned process, due to its wide availability on the market, its relatively low price and its easy handling. In addition to these reasons, the use of Hydrochloric acid leads to the dissolution of countless impurities that also dissolve during roasting, making the purification process much more complex [4]. Another process just as popular in the literature is the extraction of lithium from lime. The latter also requires roasting a mixture of spodumene pegmatite and lime at a temperature of 1040°C to obtain a soluble clinker, followed by water leaching of the clinker formed [8]. This process has been industrialized by Foote Mineral Company to produce lithium hydroxide. All the above lithium extraction methods adopt an approach involving very high-temperature calcination for the in-phase transformation of α -spodumene to β -spodumene, making them very energy-intensive and costly extraction routes, very often rendering them economically unviable on a large scale [9]. This study identified the high potential of using ammonium bifluoride (NH_4HF_2) in a low-temperature process to minimize the above disadvantages.

II. MATERIALS AND METHODS

A. Materials and Reagents

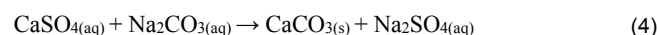
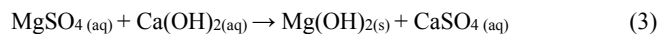
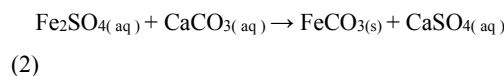
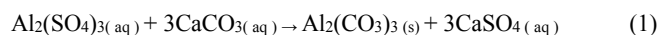
The spodumene ore was sourced from AVZ Minerals SA in the Democratic Republic of the Congo (DRC). The following analytical-grade chemicals were also used: NH_4HF_2 , H_2SO_4 , Na_2CO_3 , deionized water was used for all aqueous applications, and acetone was used for all aqueous applications with the purity of approximately around 99%. All chemical reagents were sourced from LabChem and Sigma Aldrich and were used without further purification.

B. Methods

Crystallographic analysis of the mineral was analyzed using X-ray diffraction (XRD). The X-ray pattern of the ore confirmed that the Manono ore is mainly composed of spodumene (26%) and contains some associated minerals. XRD X'Pert software helps Highscore more.

The Manono ore used contained on average 1.6% Li or 7.6% LiO_2 as an element. The chemical composition of the main ones present in the concentrate was determined using the XRF, ICP - OES method. The samples were heated to 1200°C

in an oven, then the resulting product was dissolved in sulfuric acid for analysis. Three types of metallurgical tests were carried out in this study: ABF digestion tests, solvolixivation and precipitation purification tests. Indeed, the digestion, solvolixivation and precipitation purification tests were carried out in the extractive metallurgy laboratory Faculty Polytechnic at University of Lubumbashi, Democratic Republic of the Congo (DRC). The digestion tests were carried out with the aim of transforming α -spodumene, by low temperature digestion using ammonium bifluoride, into lithium fluoride, much more soluble in some aqueous solvents, into occurrence of sulfuric acid. The leaching tests done by solubilizing lithium from lithium fluoride in an aqueous solvent. ABF digestion and sulfuric acid leaching of spodumene are not always selective because some impurities can be dissolved with lithium. To produce high purity lithium, the purification process must be effective in removing impurities by controlling the pH in the solution. When treating leaching liquors CaCO_3 is effective in removing Al and Fe by precipitation. The same involves haste. The same involves the precipitation of Mg and Ca with $\text{Ca}(\text{OH})_2$ and Na_2CO_3 , respectively to remove excess Ca. The precipitation equations are:



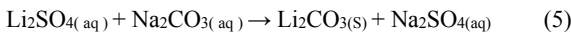
The purification solution experiments were done using beakers, stirring hot plates and a pH meter in order to determine the optimal purification conditions as shown in Figure 1.

After the precipitation of the metals, the precipitate is filtered, and the cake is washed with water. Calcium hydroxide ($\text{Ca}(\text{OH})_2$) is used to precipitate magnesium at a pH of 10.4. The amount of sodium carbonate (Na_2CO_3) equivalent to the amount of calcium in solution (Ca) was used to precipitate excess Ca in solution at room temperature with a pH varying between 10 and 11. At each stage of the purification process, a pH meter was used to determine the pH of the solutions and ICP-OES was used to determine the concentration of lithium and impurities in the solution. The purified leach liquor was adjusted to a pH of 7-8 and subjected to evaporation at 95 to 100°C to precipitate ϕ - Li_2CO_3 [10].



Fig. 1 Precipitation set up

The precipitation of Li_2CO_3 was carried out between 95 and 100°C using a hot saturated solution of Na_2CO_3 that was added dropwise to the filtrate because the solubilization of lithium carbonate decreases with increasing temperature [10]. Lithium carbonate is precipitated from an aqueous lithium solution using Na_2CO_3 and soluble contaminants (Na_2SO_4 and excess Na_2CO_3) are removed by washing with hot water [11] according to the following reaction:



Li_2CO_3 powder was precipitated, removed from the solution by filtration. The final dried product (Li_2CO_3) was characterized by ICP-OES.

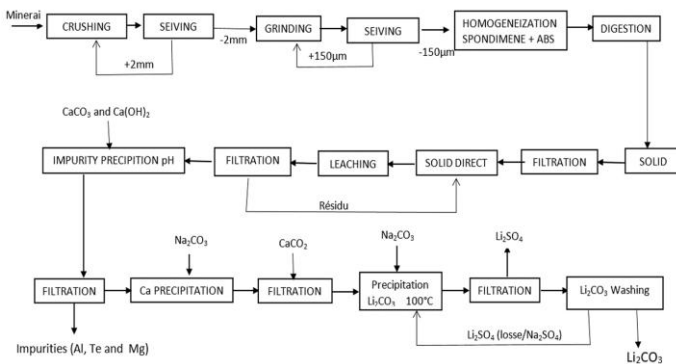


Fig. 2 Flow diagram for the treatment of Manono spodumene pegmatite

III. RESULTS AND DISCUSSION

Chemical characterization of our sample was carried out by inductively coupled plasma optical emission spectrometry (ICP-OES) and X-ray fluorescence spectrometry (XRF). The results obtained are presented in Table I.

TABLE I
CHEMICAL COMPOSITION OF MANONO SPODUMENE PEGMATITE

Compounds	SO ₂	Al ₂ O ₃	K ₂ O	Fe ₂ O ₃	Na ₂ O
XRF	65.5	15.1	2.1	1.1	0.6
Compounds	MnO	CaO	MgO	PAF	
XRF	0.2	0.1	0.1	7.8	
Compound	LiO				
ICP-OES	7.6				

The chemical characterization of the sample reveals a significant presence of silica and alumina, representing approximately 80% of its composition. This observation clearly indicates the presence of an aluminosilicate type gangue, the structure of which gives our sample a refractory physicochemical character. The gangue is mainly composed of these aluminosilicates, while the other compounds are practically minor components. In addition, our sample contains 1.5% Li or 7.6% Li_2O . As a result, this ore is economically valuable. The mineralogical characterization of our sample was carried out by X-ray diffraction. The results obtained are presented in Figure 3.

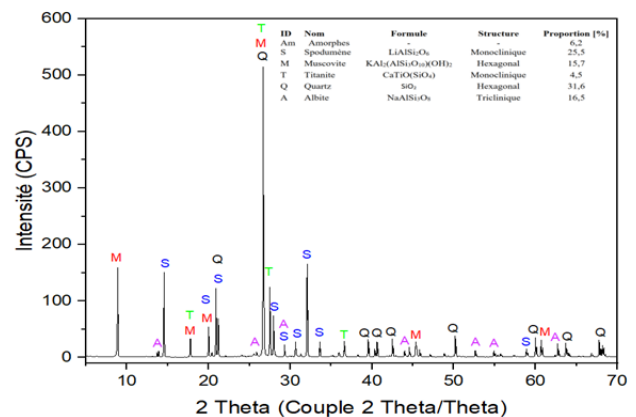


Fig. 3 Diffraction of spodumene pegmatite

The results of the mineralogical analysis show a considerably varied mineralogical composition of the studied sample. It was observed there the following: A high proportion of spodumene is consistent with the chemical composition of this pegmatite, i.e. 25.5% by mass. This interesting mineralogy gives this ore exceptional physicochemical potential for the envisaged extraction technology. And the gangue is essentially made up of quartz and aluminosilicate refractory to chemical attacks (muscovite, titanite and albite).

A. Digestion Test

The influence of the variation of time (30, 60, 90 and 120 mins) on the conversion efficiency of α -spodumene into LIF was studied: the results obtained are in Figure 4.

In Figure 4, it appears that the conversion efficiency of α -spodumene into LiF increases as a function of the evolution of digestion. From 75 minutes of digestion, an average conversion yield is reached; while beyond this time, the

increase is no longer very significant. The fact of the evolution of the conversion yield as a function of the digestion time would be explained by the kinetic theory of Avrami, which states that the kinetics of the transformation (concentration of the reactants at the level of the reaction medium), necessary for the process of germination and growth of the new phase.

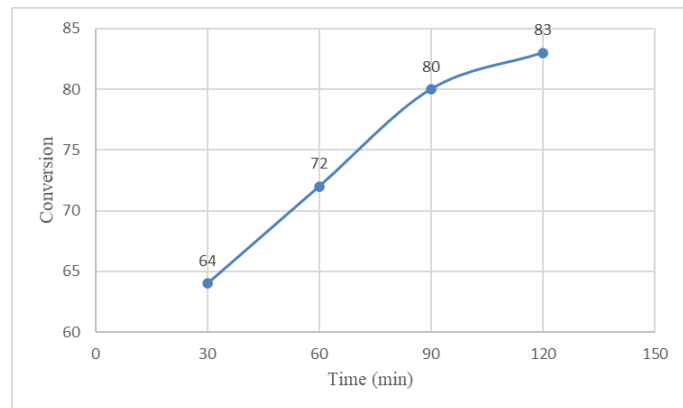


Fig. 4 Influence of time on conversion efficiency

Thus, the low yield observed after approximately 30 minutes of digestion would probably be due to a transformation of the unfinished phase due to a short maturation time. The low variation in yield beyond 75 minutes, after 90 minutes of digestion, would be due to the exhaustion of the reagents in the reaction medium, which leads to the phenomenon of long-distance diffusion.

The Influence of temperature variation (50, 100, 150 and 200°C) on the conversion efficiency of α -spodumene into LiF was studied. The results obtained are presented in Figure 5.

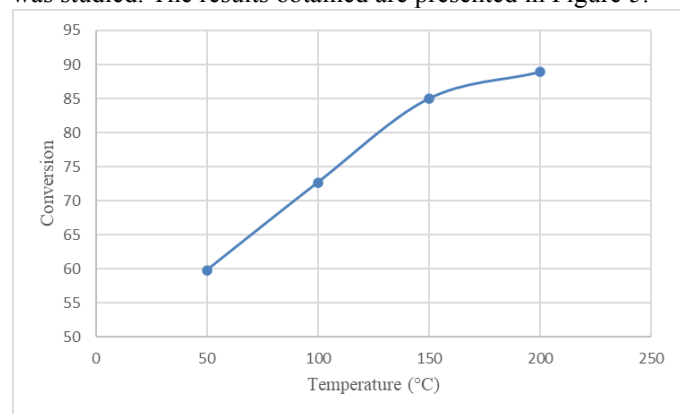


Fig. 5 Influence of temperature on conversion efficiency

In Figure 6, it appears that the conversion efficiency of α -spodumene into LiF increases as a function of the evolution of the digestion temperature. From 150°C of digestion, an average yield is obtained. While beyond this temperature, the increase is no longer significant. The shape of the evolution of the conversion yield as a function of the digestion temperature would be explained by the Arrhenius kinetic theory, which states that the kinetics of decomposition of the reactants and reaction increases with the increase in temperature. An

increase in the kinetics of decomposition of ABF into a powerful fluorinating agent found in the form of molten salt and favoring the search for digestion of α -spodumene by ABF.

Thus, the low yield observed at approximately digestion temperatures 50°C and 100 °C would probably be due to insufficient phase transformation due to the digestion temperature being lower than the ignition temperature (120 to 150°C) of the ABF decomposition reaction decomposes such that some of the ABF decomposes without reacting with α -spodumene. The small variation in yield beyond 150°C, around a digestion temperature of 200°C, would be because the ABF, which has not yet reacted, decomposes, so that digestion takes place less considerably given the exhaustion of reagents at the reaction level [12].

The influence of the variation in the α -spodumene/ABF molar ratio (1/11; 1/22; 1/33; 1/44) on the conversion yield of α -spodumene into LiF was studied. The results obtained are presented in Figure 6

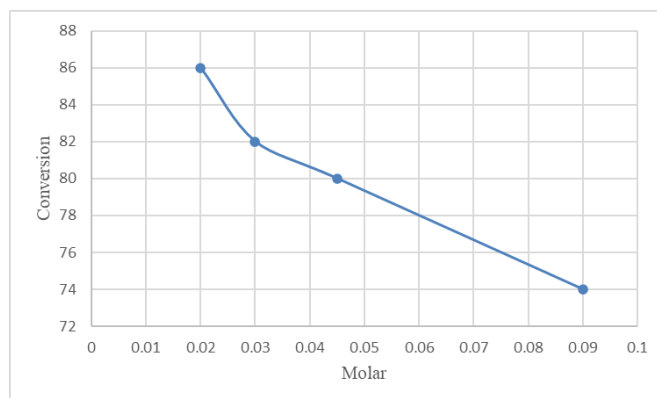


Fig. 6 Influence of the molar ratio on the conversion efficiency

In Figure 6, it appears that the conversion efficiency of α -spodumene into LiF increases as a function of the reduction in the α -spodumene/ABF molar ratio. From the molar ratio of 0.045, the average conversion yield. While beyond this molar ratio, we observe a slight increase in the conversion efficiency. The shape of the evolution of the conversion yield as a function of the molar ratio would be explained by the theory of diffusion of substances according to Fick's second law, which states that the kinetics of phase transformation would be controlled by variation in the concentration of the reactants in function of time and thus the decrease in molar ratio which corresponds to the increase in the quantity of ABF, contributes to the decrease in the variation of the concentration flow of ABF. This promotes the kinetics of digestion of α -spodumene. To do this, any increase in the molar ratio generates a reduction in the variation of the concentration flow and therefore an increase in the reaction kinetics of digestion of α -spodumene by ABF. The low yield observed for the ratio of 0.09 would probably be due to a strong variation in the concentration flow of the reagents due to the low quantity of ABF used (stoichiometric quantity).

The low variation in yield beyond 0.045 would be due to the decrease in the quantity of α -spodumene due to the conversion of a large part. After analyzing the influence of each parameter and their combined effects on the conversion efficiency, the optima retained for the digestion of α -spodumene by ABF, taking into account economic, technical and ecological constraints, are as follows: Time: 90 minutes, temperatures: 125°C, molar ratio: 1/22 (0.045)

A conformation test was carried out under these conditions and conversion yield obtained 97.10%.

B. Leaching study

The solvent used for leaching after digestion was sulfuric acid. The parameters tested included stirring speed, solid/liquid mass ratio, temperature and time to optimize the leaching conditions for LiF. The role of agitation is to keep the solids in suspension, homogenize the system and promote diffusion across the solid-liquid boundary (mass transfer) to influence the rate of reaction. Figure 7. Agitation has a significant influence on lithium extraction. Increasing the stirring speed increases the extraction yield. Above 360 rpm, the extraction yield remains constant based on the data in Figure 7 the optimum extraction is set at 360 rpm where 95% of the lithium extraction is obtained. Sitando & Crouse [4] reported similar results with a lithium extraction yield of 97.3% with a stirring speed of 320 rpm using Petalite.

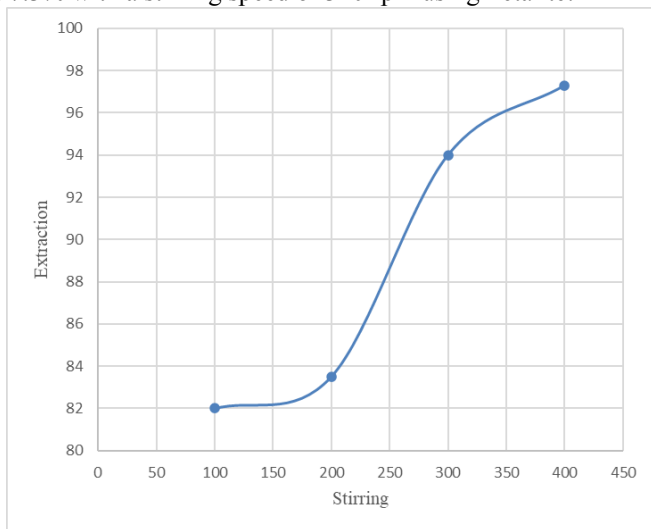


Fig. 7 Effect of stirring speed on lithium extraction rate

The influence of the variation in the S/L mass ratio on the lithium extraction yield was studied. Figure 8 shows that the lithium extraction yield decreases as the leaching S/L mass ratio increases. The shape of the curve implies considerable extraction already at the high mass ratio. This leads to a lithium extraction yield of over 94% from an S/L mass ratio of 150 g/L. Below this value, a slowdown in extraction kinetics is observed despite the increase in sulphuric acid concentration flow. This suggests the use of a large quantity of sulphuric acid due to the limited solubility of LiF in sulphuric acid.

The influence of temperature variation on lithium leaching

efficiency was studied. It can be seen in Figure 9 that the extraction yield of Li increases as a function of the leaching temperature. The shape of the curve implies a high leaching temperature at low temperatures, which also leads to an extraction yield of Li greater than 93% from a leaching temperature of 25°C. Above 50°C, there is a slowdown in the kinetics predicted by the Arrhenius theory due to the considerable evaporation of sulphuric acid and the existence of a probable synergy of sulphuric and fluoride acids leading to the solubilization of Aluminum in competition with Li [13] also reported that the leaching temperature has little effect on the dissolution of lithium.

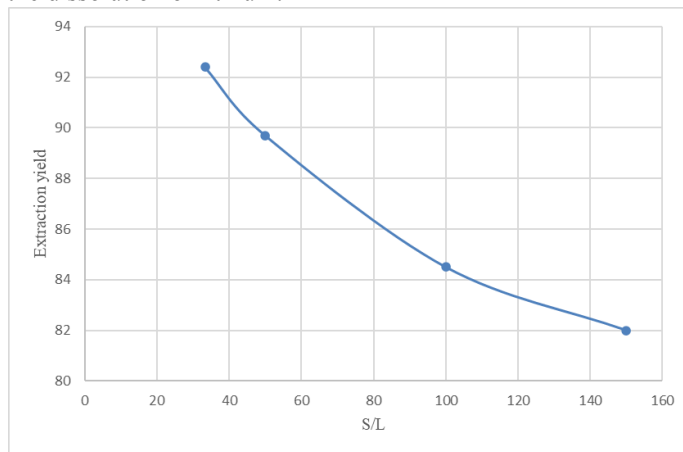


Fig. 8 Effect of S/L mass ratio on lithium extraction rate

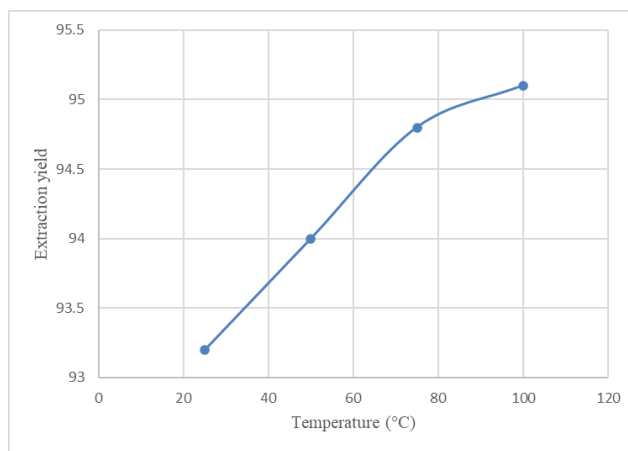


Fig. 9 Effect of temperature on lithium extraction rate

The influence of varying the teaching time (30, 60, 90, 120 minutes) on the lithium-leaching yield was studied. Figure 10 shows that the lithium extraction yield increases as a function of the leaching time. The shape of the curve implies high leaching kinetics at the start of the operation, leading to a Li extraction yield of over 90% after only 60 minutes of leaching. There is a slowdown in the kinetics predicted by Avrami's theory due to the exhaustion of the reagents, which leads to the phenomenon of long-range diffusion. Nevertheless, we can conclude that the teaching time of 120 minutes is not sufficient to extract Li adequately under these teaching conditions, hence the shape of the curve.

After analyzing the influence of each parameter on the extraction yield of Li. The optimums retained for the sulphuric acid leaching of LiF, considering economic, technical and ecological constraints, are as follows: Time: 120 minutes, temperature 30°C, S/L mass ratio: 33.33 g/L, stirring speed: 360 rpm

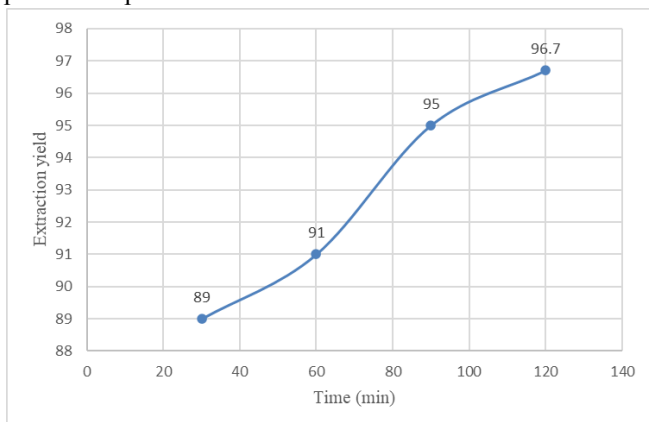


Fig. 10 Effect of time on lithium extraction yield

A confirmation test was carried out at these concentrations and the extraction yield of Li obtained was 98.23%. The results of the chemical analysis of the PLS obtained, carried out by ICP-OES, are presented in Table II.

TABLE II
SOLUTION ELEMENT RESULTS

Element	Li	Al	Fe	That
Content g/L	3.99	1.04	0.05	0.05
Element	P	N/A	Mg	If
Content g/L	0.24	0.07	0.03	1.5

C. Other Elimination of cations

After leaching the liquor which includes the alkali metals Li^+ , Na^+ and Ca^{2+} , Fe^{3+} , Mg^{2+} , Al^{3+} . Calcium carbonate (CaCO_3) is used to precipitate aluminum and iron at their respective pH and calcium hydroxide, $\text{Ca}(\text{OH})_2$ is used to precipitate magnesium. Sodium carbonate (Na_2CO_3) is used to remove excess Ca. CaCO_3 precipitates Fe^{3+} and Al^{3+} at < 1 ppm in solution [14]. A loss of lithium yield measured during the purification process may be due to the fact that Li is co-precipitated on CaSO_4 and $\text{Al}_2(\text{CO}_3)_3$ at pH 10. Lithium recovery decreases from 97% to 85% after precipitation of Li_2CO_3 .

IV. CONCLUSION

The study showed that it is possible to produce lithium carbonate from Manono spodumene without calcination at 1000 or 1100°C but by working at low temperature using ammonium bifluoride as the main reagent during digestion. Mineralogical analysis and particle size characterization revealed that the spodumene sample contained 7.6% Li_2O using ICP-OES and XRF and a significant presence of silica and aluminum, representing around 80%. The XRD results showed that the mineral phase was also spodumene. The

conversion rate of α -spodumene to lithium fluoride (LiF) is 97.10%, respecting parameters such as digestion temperature, digestion time, molar ratio between ore and ammonium bifluoride and reagent concentration. Acid leaching (H_2SO_4) gave a lithium extraction yield of 98% based on the following parameters. Leaching time 120 minutes, temperature 30°C, mass ratio 33.3 S/L, stirring speed 360 rpm.

DATA AVAILABILITY STATEMENT

The data presented in this study are available on request.

CONFLICTS OF INTEREST

The authors declare no conflict of interest.

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