

Lithium Carbonate Extraction from Zankan Spodumene Concentrate via Decrepitation and Hydrometallurgical Processes

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Abstract

It has been established that Nigeria is endowed with a variety of lithium ores, coupled with global interest in lithium production due to growing demand as an alternative source of energy. Therefore, thermal treatment of the obtained concentrate for lithium carbonate production cannot be overemphasised. Spodumene concentrate obtained from Zankan lithium ore was subjected to a decrepitation process and a sulfuric acid extraction process was used to obtain lithium carbonate from beta spodumene, with Na₂CO₃ as the leaching agent. In the process, temperature differences (200 °C, 225 °C, 250 °C, 275 °C, and 300 °C) of the acid roasting show a considerable significance grade of lithium carbonate; the highest grade of 78% was extracted at 250 °C. This suggests effective production of Li₂CO₃ as a key substance in lithium-ion batteries, even though further refining could lead to a higher yield for energy applications.

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I. Introduction

Following physical beneficiation that produced lithium concentrate, subsequent processing is required to produce lithium chemicals or metal. Calcination is carried out, where the alpha spodumene concentrate is transformed into more reactive beta spodumene by heating to between 950 - 1,100°C [1], to expand and recrystallizes into five-membered rings and form a β-spodumene tetragonal crystal structure, providing passages to liberate the lithium ions [2, 3]. The irreversible transformation of α-spodumene to β-spodumene is therefore a prerequisite treatment for the extraction [4] of lithium carbonate. Meanwhile, the third phase; γ-spodumene, is a metastable phase that occurs between the alpha and beta forms, which takes place between 700 and 900 °C [5]. Generally, it is not feasible to extract lithium carbonate from naturally occurring α-spodumene due to its monoclinic crystal structure. As the α-spodumene is chemically inert, it requires volume expansion to introduce cracks into the crystals and irreversibly transform it to β-spodumene. Since alpha-spodumene is virtually unattacked by hot sulphuric acid leaching. In addition to calcination production of Beta spodumene, mechanical activation and microwave heating produces phases with more reactivation rate, usually achieved faster with less energy than conventional process [1].

Table 1; Typical Pyrometallurgical and Hydrometallurgical treatment techniques [1]

Pyrometallurgical	Hydrometallurgical
Liming process	Acid leaching
Acid roasting	Alkali metal salt leaching
Sulfation roasting	Alkaline leaching
Chlorination roasting	Bioleaching
Roasting with sodium salts and caustic baking	
Metalthermic reduction process	

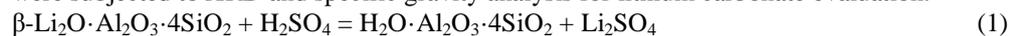
Concentrate transformed to β-spodumene is further treated (roasted) with acidic, alkaline as well chlorinated chemicals (Meshram et al., 2014) and can be handled pyrometallurgically, hydrometallurgically or the combination of the two processes [1]. Table 1 shows different typical treatment technique under the mentioned processes. A series of extraction experiments were conducted and established, indicating efficiency rate, reaction time and the temperature as details in table 2;

Table 2: Experimental methods and efficiency rate obtained by different authors on lithium extraction.

Feed	Additives	Phase conversion Temp.	Roasting Temp. °C	Rxn Time	Agitation speed	Lithium Extraction Efficiency	Source
β -spodumene	HF	1100	75	20 min	330rpm	90%	[6]
α -spodumene	H ₂ SO ₄ + HF	1100	100	3hr	-	96%	[7]
β -spodumene	NaCl ₂ + Ca(OH)	1000	-	3hr	-	98%	[8]
β -spodumene	Na ₂ CO ₃	1050	225	1hr	-	94%	[9]
β -spodumene	Na ₂ CO ₃	1100	250	3hr	-	90-93.3%	[10]
β -spodumene	Cl ₂	1100	-	2.5hr	-	-	[11]
β -spodumene	CaCl ₂	1100	900	2hr	-	90.2%	[12]
β -spodumene	Na ₂ CO ₃	1073oK	923	2hr	-	-	[13]
α -spodumene	CaO	1073oK	250	6hr	-	93.3%	[14]
β -spodumene	NaF	1000	600	1hr	-	90%	[15]

II. Material And Methods

The concentrate of 3.19% Lithium oxide made available from the flotation process of Zankan Spodumene was dried at room temperature, with a weight of 64 g, size range of -106+38 μ m was introduced into the Metallurgical furnace heated at 1100 °C [16] for 1 hour using a graphite crucible. Following the heat treatment (decrepitation), 55 g of the sample (concentrate) was weighed and H₂SO₄ solution was added. The roasting temperatures of 200 °C, 225 °C, 250 °C, 275 °C and 300 °C were considered at a roasting time of 30 minutes. The reaction shown in Eq., 1. The lithium sulphate was then leached with 30 mL of sodium carbonate solution [16, 17] at a mass of 50 g in 1 litre of water [18], reaction indicated in Eq., 2. The extracted products were subjected to XRD and specific gravity analysis for lithium carbonate evaluation.



III. Results And Discussion

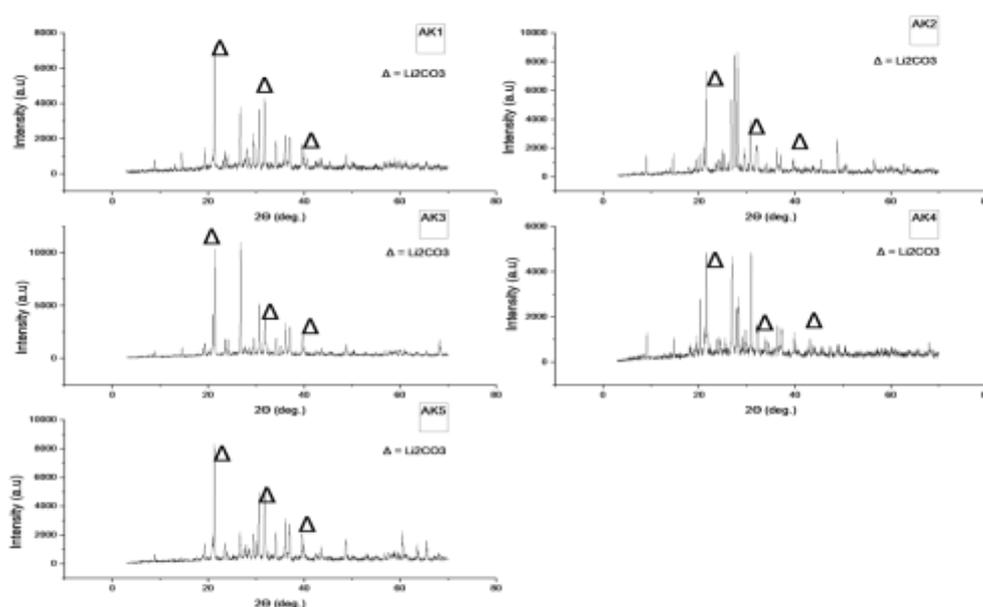


Figure 1: XRD diffractograms of Lithium carbonate

The XRD patterns, or diffractograms, for the precipitated samples after the decrepitation, acid roasting, leaching and crystallisation processes are shown in Figure 1 with AK1-5 indicated lithium carbonate obtained. Meanwhile, AK1 -5 corresponding weight-percent recoveries are 70, 60.9, 78, 69.2 and 72.6, respectively. Impurities present in the samples across are muscovite and graphite. The 78% optimal lithium carbonate obtained at 250 °C roasting temperature. These results demonstrate the technical viability of producing lithium carbonate from Zankan spodumene concentrate through a combined decrepitation and hydrometallurgical processing route, highlighting its relevance for energy-storage and battery-materials supply chains. The decrepitation treatment proved effective in inducing structural breakdown of the spodumene concentrate, thereby enhancing phase accessibility and significantly improving subsequent leaching efficiency, confirming the assertion of previous research [16, 19]. Figure 2 indicates processing route from run-of-mine (ROM) to hydrometallurgical stages for successfully converting lithium into a soluble form and enabling selective recovery as lithium carbonate.

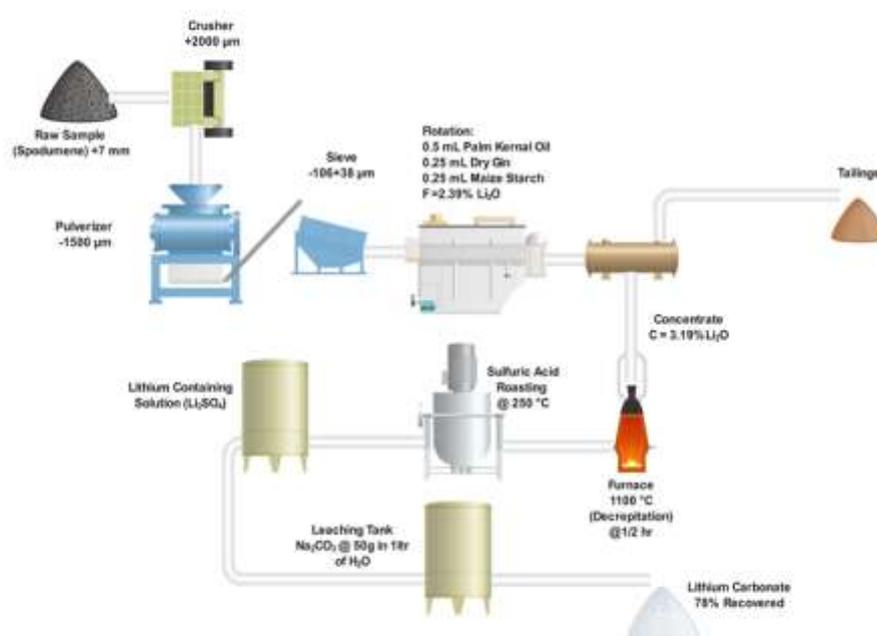


Figure 2: Graphical representation of the Beneficiation and Extraction Processes

IV. Conclusion

The sulfuric acid process was used to extract lithium carbonate from spodumene, with Na_2CO_3 as the leaching agent. In the process, temperature differences (200 °C, 225 °C, 250 °C, 275 °C, and 300 °C) during acid roasting show a considerable significance for lithium carbonate grade; the highest grade of 78% was extracted at 250 °C. In conclusion, Zankan spodumene concentrate represents a promising feedstock for lithium carbonate production when processed via an integrated decrepitation–hydrometallurgical approach. The outcomes of this research support the development of more efficient and scalable lithium extraction technologies, contributing to the establishment of resilient and sustainable lithium supply chains for next-generation battery and energy-storage applications.

References

- [1]. N. K. Salakjani, P. Singh, and A. N. Nikoloski, "Production of lithium—A literature review. Part 2. Extraction from spodumene," *Mineral Processing and Extractive Metallurgy Review*, vol. 42, no. 4, pp. 268-283, 2021.
- [2]. P. K. Choubey, M.-s. Kim, R. R. Srivastava, J.-c. Lee, and J.-Y. Lee, "Advance review on the exploitation of the prominent energy-storage element: Lithium. Part I: From mineral and brine resources," *Minerals Engineering*, vol. 89, pp. 119-137, 2016.
- [3]. N. K. Salakjani, P. Singh, and A. N. Nikoloski, "Production of lithium—A literature review part 1: Pretreatment of spodumene," *Mineral Processing and Extractive Metallurgy Review*, vol. 41, no. 5, pp. 335-348, 2020.
- [4]. C. Dessemond, F. Lajoie-Leroux, G. Soucy, N. Laroche, and J.-F. Magnan, "Spodumene: The lithium market, resources and processes," *Minerals*, vol. 9, no. 6, p. 334, 2019.

- [5]. N. K. Salakjani, P. Singh, and A. N. Nikoloski, "Mineralogical transformations of spodumene concentrate from Greenbushes, Western Australia. Part I: Conventional heating," *Minerals Engineering*, vol. 98, pp. 71-79, 2016.
- [6]. G. D. Rosales, M. del Carmen Ruiz, and M. H. Rodriguez, "Novel process for the extraction of lithium from β -spodumene by leaching with HF," *Hydrometallurgy*, vol. 147, pp. 1-6, 2014.
- [7]. G. Hui et al., "Kinetics of leaching lithium from α -spodumene in enhanced acid treatment using HF/H₂SO₄ as medium," *Transactions of Nonferrous Metals Society of China*, vol. 29, no. 2, pp. 407-415, 2019.
- [8]. G. Gabra, A. Torma, and C. Olivier, "Pressure leaching of beta-spodumene by sodium chloride," *Canadian Metallurgical Quarterly*, vol. 14, no. 4, pp. 355-359, 1975.
- [9]. Y. Chen, Q. Tian, B. Chen, X. Shi, and T. Liao, "Preparation of lithium carbonate from spodumene by a sodium carbonate autoclave process," *Hydrometallurgy*, vol. 109, no. 1-2, pp. 43-46, 2011.
- [10]. G. Kuang, Y. Liu, H. Li, S. Xing, F. Li, and H. Guo, "Extraction of lithium from β -spodumene using sodium sulfate solution," *Hydrometallurgy*, vol. 177, pp. 49-56, 2018.
- [11]. L. I. V. Barbosa, G.; Oorosco, R.P.; González, J.A., "Lithium extraction from β -spodumene through chlorination with chlorine gas," *Miner. Eng.*, vol. 56, pp. 29-34, 2014.
- [12]. L. I. Barbosa, J. A. González, and M. del Carmen Ruiz, "Extraction of lithium from β -spodumene using chlorination roasting with calcium chloride," *Thermochimica Acta*, vol. 605, pp. 63-67, 2015.
- [13]. L. L. dos Santos, R. M. do Nascimento, and S. B. C. Pergher, "Beta-spodumene: Na₂CO₃: NaCl system calcination: A kinetic study of the conversion to lithium salt," *Chemical Engineering Research and Design*, vol. 147, pp. 338-345, 2019.
- [14]. Y. Song, T. Zhao, L. He, Z. Zhao, and X. Liu, "A promising approach for directly extracting lithium from α -spodumene by alkaline digestion and precipitation as phosphate," *Hydrometallurgy*, vol. 189, p. 105141, 2019.
- [15]. G. D. Rosales, A. C. Resentera, J. A. Gonzalez, R. G. Wuilloud, and M. H. Rodriguez, "Efficient extraction of lithium from β -spodumene by direct roasting with NaF and leaching," *Chemical Engineering Research and Design*, vol. 150, pp. 320-326, 2019.
- [16]. N. Nandihalli, R. K. Chouhan, R. Kuchi, and I. Z. Hlova, "Aspects of spodumene lithium extraction techniques," *Sustainability*, vol. 16, no. 19, p. 8513, 2024.
- [17]. Q. Tian, B. Chen, Y. Chen, L. Ma, and X. Shi, "Roasting and leaching behavior of spodumene in sulphuric acid process," *Chin. J. Rare Met*, vol. 35, pp. 118-123, 2011.
- [18]. C. A. Young, *SME mineral processing and extractive metallurgy handbook*. Society for Mining, Metallurgy & Exploration, 2019.
- [19]. J. Rioyo, S. Tuset, and R. Grau, "Lithium extraction from spodumene by the traditional sulfuric acid process: A review," *Mineral Processing and Extractive Metallurgy Review*, vol. 43, no. 1, pp. 97-106, 2022.