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Research article

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Application of microwave radiation for decrepitation of spodumene from the Kolmozerskoe deposit

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Abstract: The lithium-ion industry is experiencing a rapidly growing demand for compounds containing lithium. Spodumene is one of the primary industrial minerals used in the production of this metal. It exists in three polymorphic forms. In its natural state, it is known as α -spodumene, which possesses a high resistance to chemical attack due to its compact structure containing silicon and aluminum oxides. When subjected to microwave radiation, α -spodumene undergoes a transformation, first becoming the γ form and then transitioning to the β form. It is known that the β form can be chemically treated to extract lithium. In light of this, microwave exposure was applied to α -spodumene with the aim of decrepitation, followed by sulfuric acid decomposition of the mineral. The mineral was crushed into different sizes (1.0, 0.5, and 0.25 mm). Temperature changes, induced by both conventional and microwave heating, were analyzed. The heating process was continued for samples of various sizes until a temperature of 1200 °C was reached. Sulfation of calcined samples was carried out for 60 minutes at a temperature of 250 °C. After cooling to 22 °C, distilled water was added and mixed for 120 minutes in closed leaching vessels. To determine the recovery of valuable and associated components, leach cakes and the liquid phase were analyzed using inductively coupled plasma atomic emission spectrometry. Based on the analysis of experimental results, the feasibility of using microwave radiation for decrepitation of spodumene to extract lithium is confirmed. The influence of particle size on phase transformations and, consequently, the degree of lithium extraction from spodumene was investigated. It was found that the recovery of lithium during the microwave action and leaching process for particles smaller than 0.25 mm reached 96.82 %. Microwave heating resulted in lower recovery rates of "harmful" components, such as iron, sodium, and calcium, in the leaching process, leading to a higher purity of the resulting product.

Keywords: lithium, spodumene, microwave radiation, decrepitation, sulfation, leaching.

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Применение микроволнового излучения для декрипитации сподумена Колмозерского месторождения

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Аннотация: Литий-ионная промышленность демонстрирует быстрорастущий спрос на Li-содержащие соединения. Сподумен является одним из основных промышленных минералов для производства этого металла. Он имеет 3 полиморфные формы.

В природе — это α -сподумен, который обладает высокой устойчивостью к химическому воздействию благодаря своей компактной структуре, содержащей оксиды кремния и алюминия. Микроволновое излучение превращает α -сподумен сначала в γ -, а после в β -форму, и известно, что последняя может подвергаться химическому воздействию с целью извлечения лития. Основываясь на этом факте, была проведена микроволновая процедура воздействия на α -сподумен, направленная на декрипитацию с последующим серно-кислотным разложением минерала, измельченного до разной крупности (1,0, 0,5 и 0,25 мм). Также были проанализированы зависимости изменения температуры при использовании традиционного нагрева. Обычный и микроволновый нагревы образцов различной крупности проводили до достижения температуры 1200 °С. Сульфатизацию прокаленных образцов осуществляли в течение 60 мин при $t = 250$ °С. После охлаждения до 22 °С добавляли дистиллированную воду и перемешивали в течение 120 мин в закрытых сосудах для выщелачивания. Для определения извлечения ценных и попутных компонентов был проведен анализ кеков выщелачивания и жидкой фазы методом атомно-эмиссионной спектроскопии с индуктивно связанной плазмой. На основе анализа результатов экспериментов обоснована рациональность применения микроволнового излучения для декрипитации сподумена с целью извлечения лития. Изучено влияние крупности на фазовые превращения и, соответственно, степень извлечения лития из сподумена. Показано, что извлечение лития в процессе микроволнового воздействия и выщелачивания класса менее 0,25 мм достигло 96,82 %. Микроволновый нагрев привел к более низким показателям извлечения «вредных» компонентов, таких как железо, натрий и кальций, в процессе выщелачивания, что дает преимущество в чистоте получаемого продукта.

Ключевые слова: литий, сподумен, микроволновое излучение, декрипитация, сульфатизация, выщелачивание.

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Introduction

Lithium, a metal with unique physical and chemical properties such as a high specific heat capacity, good conductivity, and strong chemical activity, is finding increasing applications across various industries and technologies. Its utility has expanded from traditional sectors like metallurgy, energy, aerospace, and medicine to the forefront of green energy technologies [1–3]. The current surge in the lithium market is predominantly fueled by the growing demand for energy storage devices [4]. In order to meet the projected lithium demand by 2030, there are plans to achieve the designed capacity of the Russian joint venture between MMC Norilsk Nickel and Rosatom for processing rare metal pegmatites from the Kolmozerskoe deposit (Murmansk Oblast, Russia) [5].

Sulfuric acid leaching is one of the methods employed to extract lithium carbonate from spodumene concentrates. Kola MMC is set to become the largest sulfuric acid producer in the Murmansk Oblast by 2027. They have plans to initiate copper production using the roasting–leaching–electroextraction process [6]. As a byproduct of this process, sulfuric acid will be generated, making it practical to utilize in the processing of spodumene from the Kolmozerskoe deposit.

Currently, lithium production in the industry relies on three primary sources [7–9]:

- solid mineral resources, including spodumene, complex Li–Be-pegmatite ores — 50 %, and lithium mica — 20 %;

- liquid mineral resources, encompassing brines, and lake brines, oil formation waters, and thermal waters — 20 %;

- secondary raw materials, which consist of batteries, accumulators, and chemical current sources — 10 %).

Among these industrial minerals used for lithium extraction, spodumene holds significant importance [10]. Its chemical composition includes lithium oxide (8.1 %), aluminum oxide (27.4 %), and silicon dioxide (64.5 %) [11]. The pegmatites from the Kolmozerskoe deposit are not only a valuable source of lithium but also beryllium, tantalum, niobium, and other metals [12].

Spodumene can exist in three distinct modifications: α , β , γ [1]. Natural spodumene is naturally found in the crystalline α phase. The β form results from recrystallization when α spodumene is heated at temperatures within the range of 900 to 1100 °C [13; 14], with complete transformation occurring up to 1100 °C. The γ modification of spodumene is a metastable phase that occurs when α spodumene is heated to temperatures ranging from 700 to 900 °C [2; 15]. The γ phase is characterized by low reactivity and a limited degree of lithium extraction, underscoring the need for decrepitation [16; 17].

Traditional methods of thermal decrepitation have several disadvantages, including high energy consumption and adverse environmental impacts [18]. Addition-

ally, the transportation of hydrocarbon energy resources to the production region in the Murmansk Oblast leads to added costs in the production of finished products. Therefore, it is crucial to assess the feasibility of using microwave radiation in comparison with conventional decrepitation methods.

When compared to conventional heating, microwave heating offers several advantages, including resource savings, shorter processing times, more controlled heating processes, and direct, non-contact selective and bulk heating [11]. Achieving uniform heating throughout the entire material bulk can increase the porosity of the carrier mineral, thereby reducing the required sintering time and the temperature of chemical reactions, while also enhancing the diffusion of the leaching agent within the mineral and improving recovery rates [12; 19]. Consequently, the use of ultrahigh-frequency (microwave) heating for the decrepitation of spodumene can be an efficient and cost-effective method. However, further research is necessary to assess its feasibility.

In terms of their interaction with microwaves, materials can be categorized into three groups: transparent, conductive/opaque, and absorbing [6]. Low dielectric loss materials, such as spodumene, are challenging to heat. However, they can absorb microwaves at elevated temperatures due to increased dielectric losses. Therefore, for microwave heating of such materials, a combined method is typically employed [20]. In this approach, materials are preheated by another heat source to a specific temperature at which they become more efficient in absorbing microwave radiation before being exposed to microwaves directly [13].

The mechanism and the impact of hybrid microwave on the structural changes of spodumene have not been comprehensively studied. Discrepancies in the results of phase transformations observed in spodumene polymorphs at high temperatures can be attributed to differences in grain sizes, impurity concentrations, amorphous materials formed during grinding, as well as variations in experimental setups, heating methods, and temperature measurements [21; 22]. However, the influence of process parameters on the phase transformations of spodumene from the Kolmozerskoe deposit and its leachability has not been thoroughly investigated.

In order to explore the potential advantages of both microwave and direct decrepitation methods, this study examines the calcination of spodumene using microwave radiation, investigates the impact of microwave and direct methods at different material sizes on phase transformations, and consequently assesses the leach-

bility of this mineral. Furthermore, it compares microwave heating with standard methods.

Materials and methods

Mineralogical studies have revealed that the ore from the Kolmozerskoe deposit comprises approximately 20 % spodumene ($\sim 1.5 \pm 1.6$ % Li_2O), about 30 % quartz, 30 % albite, 15 % microcline, 5 % muscovite, along with traces of zircon and tourmaline [23].

Traditionally, spodumene ores are enriched to yield a concentrate containing roughly 5.0–6.0 % Li_2O and less than 1 % Fe_2O_3 as the raw material for subsequent calcination processes and lithium extraction [24]. For the experiments, spodumene was manually sampled from the ore mass and subjected to analysis. The sample was initially crushed to a particle size of -1 mm, after which quartered samples were further crushed to ensure 100 % content of classes smaller than 0.5 and 0.25 mm. The chemical composition of the quartered samples for research was as follows, wt.%:

Li_2O	5.6
Al_2O_3	25.1
SiO_2	65.7
Fe_2O_3	0.7
Na_2O	1.0

To investigate the efficiency of microwave calcination of the spodumene concentrate and the impact of feedstock size, experiments were conducted to calcine size classes less than 1.0, 0.5, and 0.25 mm. The temperature profiles of the samples over time during direct heating are depicted in Fig. 1.

Samples with particle sizes less than 1.0, 0.5, and 0.25 mm were exposed to direct heating in a muffle furnace, reaching a temperature of 1200°C . During the initial 10 min of the process, they exhibited varying heating rates, but after this time, the rates equalized and stabilized at approximately $30^\circ\text{C}/\text{min}$.

The microwave heating of the spodumene concentrate was conducted in a specialized microwave chamber equipped with independent magnetrons and a control system, enabling the adjustment of a 1 kW microwave power at a frequency of 2.5 GHz, along with temperature monitoring. In each test, a 100 g sample of spodumene concentrate was placed in glassware, chosen for its transparency to microwave radiation. The thickness of the material layer remained consistent at 10 mm. Temperature profiles illustrating the relationship between

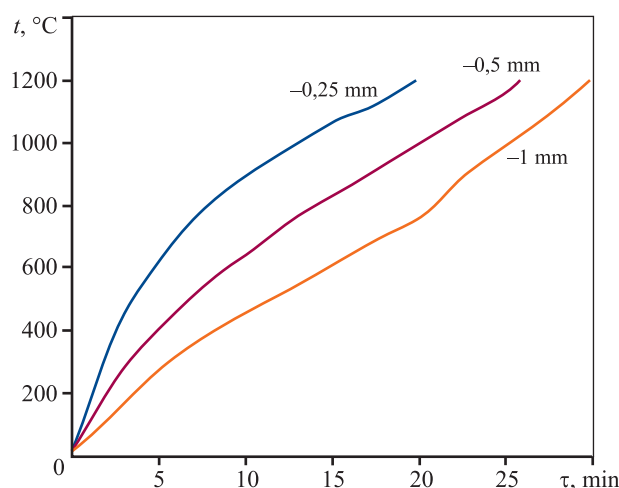


Fig. 1. Temperature as a function of heating duration in a muffle furnace

Рис. 1. Зависимость температуры образцов разной крупности от продолжительности нагрева в муфельной печи

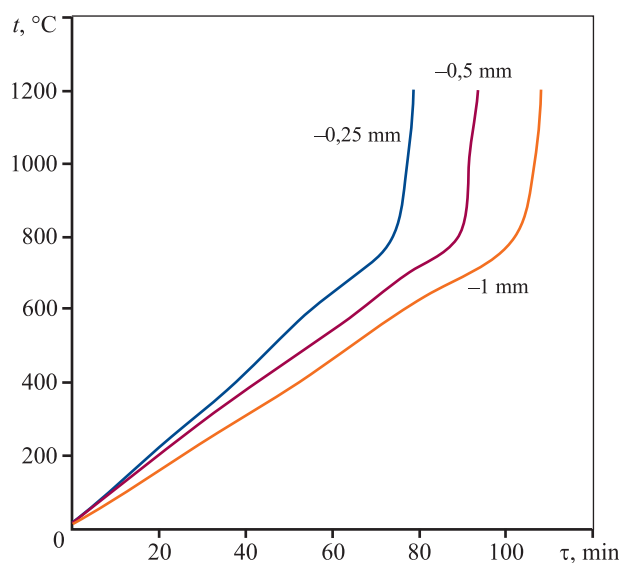


Fig. 2. Temperature as a function of microwave heating duration

Рис. 2. Зависимость температуры образцов разной крупности от продолжительности микроволнового нагрева

temperature and microwave exposure time are presented in Fig. 2.

The efficiency of microwave heating is contingent on the thermophysical properties of the sample [25]. Spodumene exhibits weak absorption of microwave radiation at low temperatures. The heating rate follows a linear pattern up to approximately 700 °C, at which point it reaches around 20 °C/min. This rate is lower compared to direct heating in a muffle furnace. Beyond

800 °C, there is a sharp increase in the heating rate, reaching about 70 °C/min. This significant change is attributed to alterations in the dielectric properties of spodumene as it crosses the critical temperature of approximately 634 °C [13], which explains the rapid acceleration in its heating rate. As the temperature rises to around 800 °C, the heating profiles rapidly become nearly exponential, enabling the temperature to reach 1200 °C in approximately 6 min.

The results indicate that the time required to raise the temperature from 800 to 1200 °C is considerably shorter when microwave radiation is employed. The direct heating method yields the best results for $t < 800$ °C. The temperature profiles depicted in Figs. 1 and 2 highlight the efficiency of microwave hybrid heating of spodumene.

The temperature profiles for different heating methods reveal that smaller spodumene particles undergo a faster transformation into β spodumene with shorter heat exposure times. This implies that particle size has an impact on both microwave and direct heating of spodumene.

The leaching properties of samples subjected to microwave and conventional calcination methods were compared. To extract lithium from the calcined samples, specifically β -spodumene, a process involving sulfatizing roasting followed by water leaching was carried out. In this process, concentrated sulfuric acid was introduced to the calcined sample. The mixture was then heated in an oven at a temperature of 250 °C for 60 min. Following sulfatizing roasting and subsequent cooling to room temperature, distilled water was added, and the resulting mixture was stirred for 120 min at ambient temperature with agitation at 200 rpm. This step was performed to leach the soluble lithium sulfate that was produced during the roasting process. The filtrate, containing the dissolved components, was then separated from the solid residue using a Büchner funnel, Bunsen flask, and filter paper. The leaching cakes and the resulting solutions were analyzed for their Li, Fe, Na, and Ca content. It's important to note that only samples with a particle size smaller than 0.25 mm were investigated in this study.

Results and discussion

Table presents a summary of the metal balance following conventional and microwave radiation treatments.

For direct heating, the lithium extraction rate was 97.37 %, while the microwave procedure yielded a slightly lower rate of 96.82 %. These results indicate that α -spodumene exhibits very low reactivity, suggest-

Metal balance

Баланс металла

Product	Weight (volume), g (ml)	Content, % (g/l)				Extraction, %			
		Li	Fe	Na	Ca	Li	Fe	Na	Ca
Heating in a muffle furnace									
Product solution	(100.02)	(1.27)	(0.02)	(0.30)	(0.57)	97.37	9.81	72.25	94.32
Leaching cake	98.35	0.03	0.22	0.12	0.03	2.63	90.19	27.75	5.68
Initial material	100.00	1.30	0.24	0.42	0.60	100.00	100.00	100.00	100.00
Microwave heating									
Product solution	(100.04)	(1.26)	(0.01)	(0.16)	(0.23)	96.82	5.06	37.36	38.04
Leaching cake	98.23	0.04	0.23	0.27	0.38	3.18	94.94	62.64	61.96
Initial material	100.00	1.30	0.24	0.42	0.60	100.00	100.00	100.00	100.00

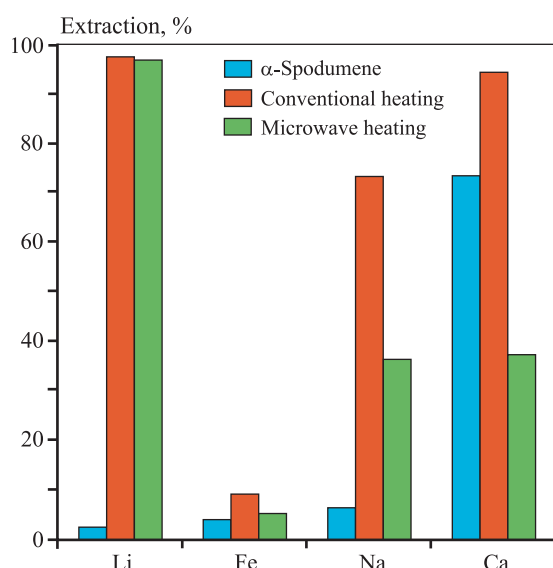


Fig. 3. Extraction of major elements by leaching from initial spodumene concentrates and calcined by microwave and conventional methods

Рис. 3. Извлечение основных элементов при выщелачивании из необработанного и прокаленного с помощью микроволнового и обычного методов нагрева образцов концентрата сподумена

ing limited efficiency in lithium leaching. Fig. 3 illustrates the recovery of iron, sodium, and calcium through leaching from initial spodumene concentrates and those subjected to calcination by both microwave and conventional methods. The obtained results indicate that the extraction of these elements was lower under microwave exposure, providing an advantage over conventional heating in subsequent stages of lithium extraction from the resulting solutions.

Conclusions

In this study, the impact of microwave radiation power on lithium leaching was thoroughly investigated. As the temperature increases to approximately 700 °C, the microwave heating rate experiences a sharp rise, allowing it to reach 1200 °C in approximately 6 minutes. The research has confirmed that microwave power plays a significant role in influencing phase transformations and, consequently, the leaching of calcined samples of spodumene concentrate.

The study has also revealed that the most efficient heating method involves a combination of conventional and microwave treatments on the sample. Based on the research findings, it is suggested that traditional heating should be applied up to a temperature of 800 °C, after which microwave exposure is initiated. This approach allows for achieving the maximum heating rate.

With the combined method, it becomes possible to reach a temperature of 1200 °C in approximately 14 minutes, which is significantly quicker compared to 20 and 80 min required for conventional and microwave exposure, respectively.

By using microwave radiation, a lithium recovery rate of 96.82 % was achieved, which is comparable to the recovery rate of the calcined sample under conventional heating. Moreover, microwave calcination resulted in lower impurity levels in the leachate, presenting an advantage in downstream processing stages.

An establishment of a pilot plant for implementing the combined heating procedure and conducting additional research will enable further validation and refinement of the results obtained.

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O.N. Krivolapova — determined the purpose of the work, supervised the experiments, wrote the article.

I.L. Fureev — prepared mixtures and initial samples, conducted laboratory experiments, participated in the discussion of the results.

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