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Metallurgy

Investigation of zinc leaching from clinker with pretreatment of raw materials by ultrahigh frequency radiation (microwave)

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Abstract

The processing of zinc-containing man-made raw materials is an important task from the point of view of rational use of natural resources and reducing the environmental burden. In recent years, there has been a growing interest in efficient methods of processing such materials, since zinc is an important metal for industry. The leaching of zinc from raw materials with a solution of sulfuric acid is a key step in the hydrometallurgical production of zinc. Optimizing this process is crucial to increase the efficiency of its extraction, reduce costs, and minimize negative environmental impacts. We have investigated the process of leaching zinc from man-made raw materials with a solution of sulfuric acid, with pretreatment of raw materials with microwave radiation. Optimal leaching parameters have been determined. Clinker, a residual product of zinc ore calcination, was used as the starting material. The zinc content in clinker is 1.25%. It is shown that high zinc recovery is achieved after preliminary exposure to microwave radiation at a temperature of 600 °C. The effect of solvent concentration, the ratio of solid and liquid phases, and temperature on the degree of zinc extraction has been studied.

Keywords: leaching, zinc, clinker, ultrahigh frequency radiation.

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Introduction

Zinc, being one of the most common metal elements in the world, is widely used in industries such as automotive, construction, shipbuilding, and aerospace. It is used in the form of electroplating, zinc alloys, zinc oxide, and other forms to protect steel surfaces from corrosion [1]. Key mining and processing facilities for zinc-containing resources are located in China, Peru, Australia, India, the United States, Mexico, and Bolivia. These seven countries together produce more than 76% of zinc in concentrates [2]. The main components of zinc ores are zinc sulfide, zinc oxide, or a combination of the two. Currently, 70% of the world's zinc production comes from primary zinc ores, while the remaining 30% comes from secondary zinc sources [[3], [4]].

Pyro- and hydrometallurgical methods are used for processing zinc-containing technogenic materials [5]. Pyrometallurgical processes are based on the reduction of zinc with carbon-containing materials at high temperatures. The most common pyrometallurgical method is rolling (reduction and ignition firing). As a result of rolling, zinc lotions and clinker are obtained. Zinc sublimations are directed to sulfuric acid leaching [6]. To date, 4.5 – 5.7 million tons of zinc industry waste have accumulated in Kazakhstan [7].

Traditionally, acids such as [[8], [9]], sulfuric acid and hydrochloric acid are used for zinc leaching, which can be environmentally hazardous. Modern research focuses on the use of less toxic and more stable reagents and methods, which contributes to improving the environmental safety of the process [[10], [11], [12]]. In recent years, the use of bacteria such as *Thiobacillus ferrooxidans* to leach zinc from man-made waste, such as ash or slag, has been actively investigated. It is an environmentally friendly alternative to chemical methods, which shows high results at low temperatures and pressures. Despite its advantages over traditional enrichment methods, bio-leaching technology has a significant disadvantage, which is the high duration of the technological process due to the weak kinetics of redox reactions [13]. Modern methods, such as the use of ultrasound and microwave radiation, are becoming increasingly popular to improve material recycling processes [14]. These methods affect the structure of the material, which can help improve leaching performance. In [15], ultrasonic technology using the ammonia-ammonium-chloride-water system was used to leach zinc from industrial waste. Some studies combine microwave treatment and ultrasound leaching with a microbiological process, where microwave and ultrasound treatment activates bacteria that promote additional dissolution of metals, including zinc [16]. This can become an important area for processing ores and waste.

One of the promising methods for increasing extraction efficiency is the pretreatment of raw materials by microwave firing, which makes it possible to intensify subsequent leaching processes. This review examines the mechanisms of the influence of microwave processing on the structure of raw materials, changes in its phase composition, and an increase in the degree of extraction of germanium from zinc oxide (ZnO) dust [17]. For the processing of lead oxides, their preliminary purification from chlorides and fluorides is necessary. For this purpose, technologies for washing welz oxides with aqueous and aqueous-alkaline solutions were used. Pyrometallurgical calcination is an alternative method of halogen removal. Microwave heating is a promising method of heat treatment of welz oxides, having a number of advantages, such as the transfer of electromagnetic energy instead of heat, high heating rate, selective heating of materials and the volumetric nature of heating [[18], [19]].

Clinker, as an intermediate product of ore processing, can contain various metals, including zinc, which is an important element for various industries. For efficient processing and extraction of zinc, it is necessary to increase the efficiency of leaching, which is usually carried out using acids. However, the use of traditional leaching methods may be limited by low productivity, as well as high energy consumption of the process. Hydrometallurgical processes remain the main ones for processing zinc-containing raw materials, as they allow obtaining high-purity metals with minimal energy costs. The leaching of zinc from raw materials with a solution of sulfuric acid is a key step in the hydrometallurgical production of zinc. Optimizing this process is crucial to increase the efficiency of its extraction, reduce costs, and minimize negative environmental impacts. In recent years, more and more attention has been paid to the use of non-traditional methods of intensifying leaching processes, such as microwave heating and ultrasonic treatment.

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In recent years, more and more attention has been paid to the use of non-traditional methods to intensify leaching processes, such as microwave heating. Therefore, the purpose of this study is to optimize the process of zinc leaching from man-made raw materials (clinker) with pretreatment by microwave radiation, by determining the effect of parameters on the efficiency of zinc extraction, which will increase efficiency and reduce the environmental burden during the processing of zinc-containing materials.

Methodology and Materials

Technogenic zinc-containing raw materials, clinker, were used as the starting material. The chemical composition of the sample is represented by the main elements: Fe (25.39%), Zn (1.25%) and Cu (0.81%), as well as gold (1.53 g/t) and silver (71.81 g/t), calcium (3.81%), silicon (4.58%), copper

(1.04%), zinc (more than 1.2%) and other substances.

The effect of microwave radiation on clinker occurred in a high-temperature microwave reactor "Energy K-50" (915 MHz, 25 kW). Figure 1 shows a single installation of the Energiya K family. The complex is designed to process the initial product by dielectric heating it in ultrahigh frequency electromagnetic fields in the mode of continuous movement of the material in a rotating dielectric retort (tube) installed in the waveguides of the drying chamber of the Complex.

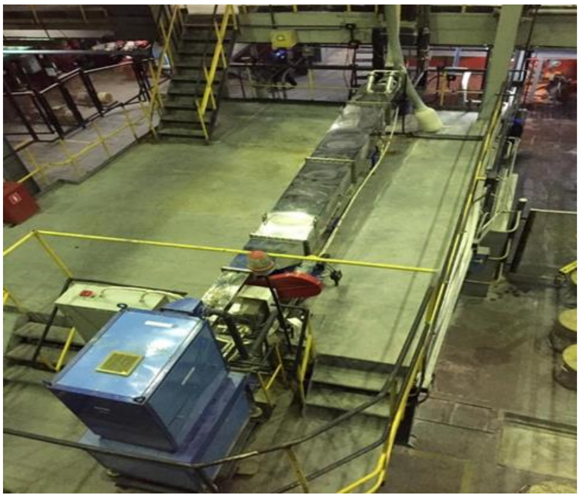


Figure 1 - A single installation of the «Energy K» family

Test experiments were conducted on leaching of the initial technogenic zinc-containing raw materials in order to determine the optimal conditions. An installation was used for leaching, including a reactor with a PE 8399 mechanical stirrer from Ekros.

The kinetics of the leaching process was studied by placing a solution of 0.1 dm3 sulfuric acid or alkali in conical flasks with a volume of 0.25 dm3 (acid concentration ranged from 60-140 g/dm3, sodium hydroxide from 25-150 g/dm3) and ore in the ratio S:L=1:5. The leaching was carried out with intensive stirring; the duration of the process was 6 hours, the mixing speed was 300 rpm, and the temperature ranged from 22-80 °C.

The leaching conditions are chosen to ensure an optimal balance between extraction efficiency, reaction rate, mass transfer, and process economy.

Methods of analysis

Modern analytical methods were used in the work. The phase composition of the samples was determined using a D8 Advance X–ray diffractometer (BRUKER), Cu-Ka radiation. The elemental analysis was performed using an X-ray fluorescence spectrometer with an Axios wavelength of 1 kW (PANalytical). The microstructure of the surface was monitored using a JXA-8230 electron probe microanalyzer from JEOL (Jeol, Tokyo, Japan). The quantitative zinc content in the solutions after leaching was determined using an Optima 8000DV inductively coupled plasma atomic emission spectrometer.

Results and discussion

In the course of the research, the following experimental scheme was used: crushing clinker to 90 % of the class size -0.071+0 mm, microwave processing and leaching (Fig.2) [20].

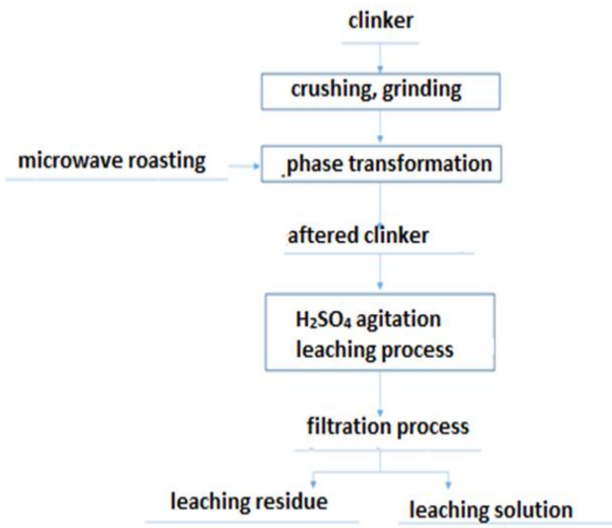


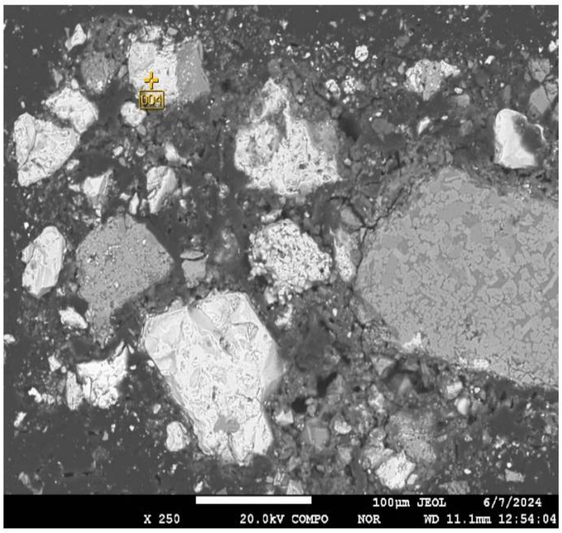
Figure 2 – Block diagram of the experiment

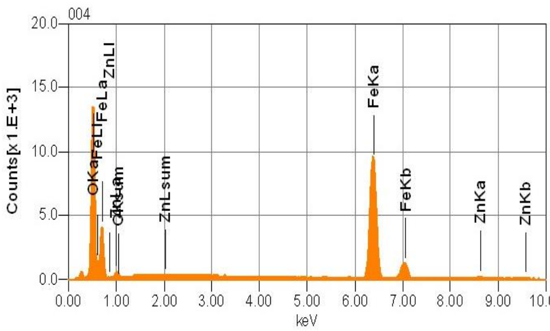
According to the above scheme, the crushed and crushed clinker has passed the stage of microwave firing. The parameters of the effect of microwave radiation on clinker are shown in Table 1.

Table 1 – Parameters of experiments on a microwave installation

Naming of indicators	Experience No. 1	Experience No. 2	Experience No. 3	Experience No. 4
Microwave power, kW	25	25	25	25
Frequency of waves, MHz	915	915	915	915
Duration of experience, min	5-7	5-7	5-7	5-7
Temperature, °C	250	460	600	700

When studying the microwave effect on clinker, the following parameters were taken: radiation power — 25 kW, frequency — 915 MHz, treatment duration — 5-7 minutes. These parameters remained unchanged in all experiments, while the heating temperature varied (from 250 to 700 °C), which made it possible to evaluate the influence of the temperature factor, all other things being equal. The frequency of 915 MHz is the industry standard for microwave processing, providing deep penetration of microwave energy into the processed material. The duration of 5-7 minutes was chosen experimentally as sufficient to achieve a stable thermal effect at a given power. Increasing the time does not give a significant increase in temperature or effect, but increases energy consumption.





Chemical formula	ms%	mol%	Sigma	Net	K ratio	Line
O	36.51	66.87	0.13	2926850	0.3257862	K
Fe	61.12	32.07	0.42	5578589	0.6352698	K
Zn	2.37	1.06	1.16	98005	0.0225208	K
Total	100.00	100.00				

Figure 3 - Microstructure and energy dispersion analysis of clinker after exposure to microwave radiation at 700 °C

The temperature range covers both the initial stages of dehydration and structural transformations (250-460 °C) and high-temperature effects, including phase transformations and activation of chemically inert compounds (600-700 °C).

As can be seen from the table, the microwave heating temperature range ranges from 250 – 700 °C. According to the results of energy dispersion analysis of clinker after exposure to microwave radiation, it was found that when the microwave heating temperature reaches 460 °C, cracks form in the sphalerite grain as a result of thermally induced mechanical stresses. When the temperature rises to 600 °C, the zinc-containing phase is mainly ZnO [20]. At the same time, zinc oxide is well soluble in dilute acids by the reaction:

$$ZnO + H_2SO_4 = ZnSO_4 + H_2O \quad (1)$$

$$ZnO + 2NaOH + H_2O = Na_2[Zn(OH)_4] \quad (2)$$

With a further increase in temperature (up to 700 °C), a complex ferritic compound (ZnO · Fe2O3) is synthesized, demonstrating a high degree of chemical resistance in weakly concentrated acidic media (Fig. 3). Thus, the temperature of the clinker phase transformation by microwave heating was chosen to be 600 °C.

In the course of the research, the process of leaching clinker pretreated with microwave radiation in different temperature conditions with a solution of sulfuric acid concentration of 100 g/dm^3 at a temperature of 22 ±2 ° C was studied (Fig. 4).

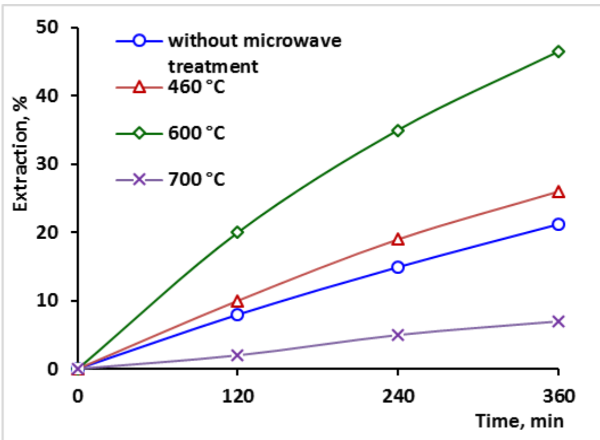


Figure 4 - Comparison of zinc extraction efficiency during clinker leaching with sulfuric acid before and after exposure to microwave radiation

The figure 4 shows that the degree of zinc recovery from clinker was: - from the feedstock – 21.12%; after preliminary exposure to microwave radiation at 460 ° C – 23.45%, at 600 ° C – 46.47%, at

700 ° C – 5.12%. Thus, the highest zinc recovery was achieved after preliminary exposure Microwave radiation at a temperature of 600 °C. Increasing the temperature to 700 ° C reduces the leaching process. This fact is probably related to the formation of a complex ferritic compound ($ZnO*Fe_2O_3$), which demonstrates a high degree of chemical resistance in weakly concentrated acids. In subsequent experiments, clinker was pretreated with microwave radiation at a temperature of 600 ° C.

Then, training using sulfuric acid of various concentrations from 25 to 125 was studied (Fig. 5). The kinetic curves shown in the figure show that with an increase in the concentration of sulfuric acid, zinc extraction increases from 15.0 to 46.47 %. At a higher concentration of H_2SO_4, the formation of insoluble iron sulfate ($Fe_2(SO_4)_3$ is possible, which can reduce the extraction of impurity elements.

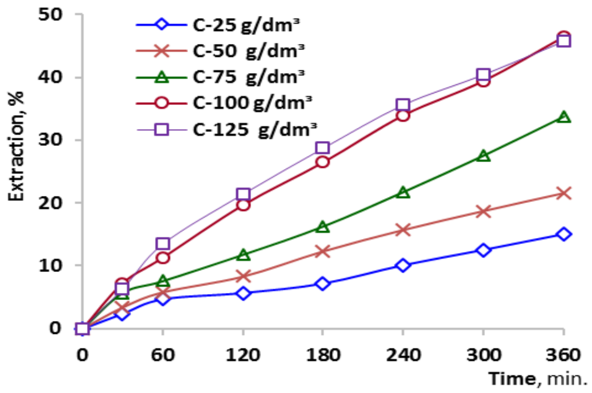


Figure 5 - Effect of sulfuric acid (H_2SO_4) concentration on zinc extraction from clinker after exposure to microwave radiation

When studying the effect of the concentration of the leaching reagent on the zinc dissolution process, sodium hydroxide (NaOH) was also used, the concentration of which varied between 25-150 g/dm^3 (Fig. 6).

Figure 5 shows the kinetic curves of the interaction of clinker pretreated with alkali depending on its concentration, which shows that with an increase in the concentration of sodium hydroxide from 25 to 50 g/dm^3, the degree of zinc leaching increases from 4.8 to 6.9 %. An increase in the alkali concentration leads to a sharp decrease in zinc extraction to 5.9 %. The reason for the decrease in zinc extraction at high NaOH concentrations may be the formation of insoluble compounds or the passivation of the clinker surface.

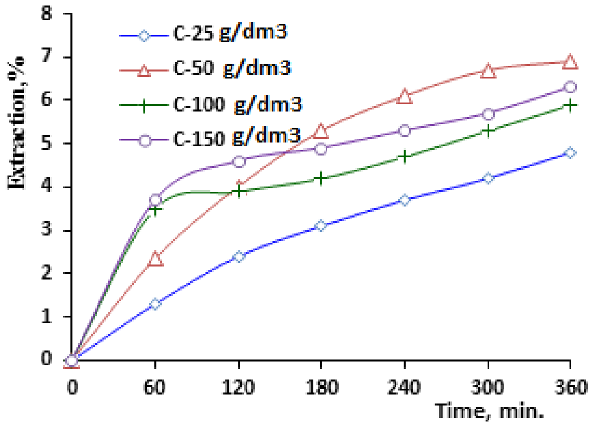


Figure 6 - Effect of sodium hydroxide (NaOH) concentration on zinc extraction from clinker after exposure to microwave radiation

The results shown in Figures 7 and 8 demonstrate the effect of the liquid–solid ratio, as well as the temperature of the leaching of clinker with sulfuric acid.

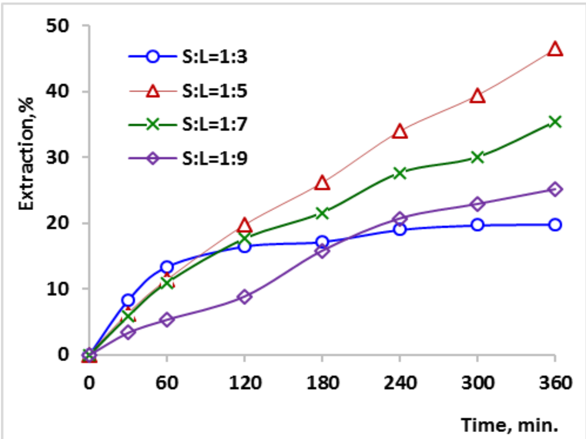


Figure 7 - The effect of the S:L ratio on zinc extraction from clinker after exposure to microwave radiation

It is shown that with an increase in the S:L ratio from 1:3 to 1:5, an increase in the degree of zinc extraction is observed from 32.0 to 46.0 %, and a further increase contributes to a decrease in extraction from 46.0 to 25.07 % (Fig. 7). The optimal solid-liquid ratio is 1:5.

As for the effect of temperature, the relationship is simpler: An increase in the clinker leaching temperature from 22 to 80 °C contributes to an increase in the degree of zinc extraction from 46.47 to 68.18 % (Fig. 8).

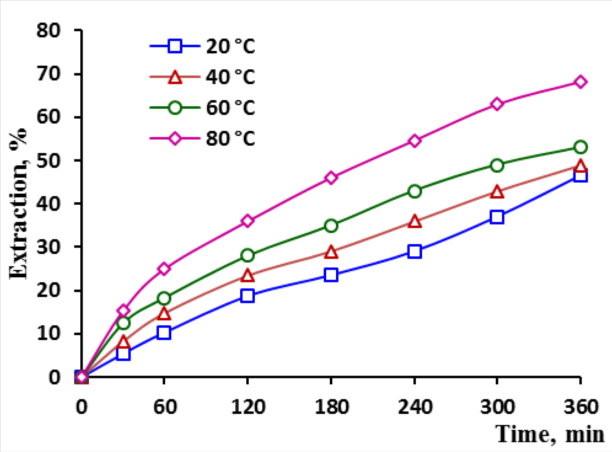


Figure 8 - Effect of temperature on zinc leaching from clinker after exposure to microwave radiation

Thus, the studies carried out and the results obtained have shown that the optimal conditions for exposure to microwave radiation are: a temperature of 600 ° C, and a leaching concentration of sulfuric acid of 100 g/l, a liquid–solid ratio of 5:1, and a leaching temperature of 80 °C.

A study of the effect of acid and alkali concentrations on the degree of zinc leaching from clinker pretreated with microwave radiation showed that the optimal concentration of sulfuric acid is 100 g/dm^3, and alkali is 50 g/dm^3.

Studies have shown that the optimal parameters for the leaching process are the temperature of 80 °C and the ratio of solid and liquid phases (S:L) 1:5. Under these conditions, a balanced ratio is achieved between the efficiency of chemical interaction and the processability, which allows for sufficient contact between the reagent (sulfuric acid) and the processed material. Under these conditions, the degree of extraction by sulfuric acid was 46.47%. Sulfuric acid leaching is the most common method, providing a high degree of recovery. In this study, sulfuric acid leaching showed a higher level of zinc recovery than alkaline leaching, which makes it the preferred method under the conditions studied.

Conclusion

Pretreatment of clinker using microwave and ultrasonic exposure is a promising method for increasing the efficiency of zinc leaching. The use of these methods can reduce energy consumption and increase the productivity of processing processes, which is important for the modern metallurgical and chemical industries.

Studies have shown that the highest zinc recovery is achieved after preliminary exposure to microwave radiation at a temperature of 600 °C. Increasing the temperature to 700 ° C reduces the leaching process. This fact is probably related to the formation of a complex ferritic compound ($ZnO*Fe_2O_3$), which demonstrates a high degree of chemical resistance in weakly concentrated acids.

Conflict of interest. There are no competing interests for all authors.

CRediT author statement: **A. Berkinbayeva:** Conceptualization, Methodology, and Software; **T. Surkova:** Data curation and Writing - Original draft preparation; **Z. Dossymbayeva**: Visualization and Investigation; **N. Umirbekova**: Visualization and Investigation; **A. Kebekbayeva**: Software and Validation; **N. Kyusubaeva**: Software and Validation.

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Ультра жоғары жиілікте сәулелендіру арқылы (микротолқынды пеш) алдын ала өңделген клинкерден мырыштың шаймалануын зерттеу

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Қабылданды: *27 мамыр 2025*

ТҮЙІНДЕМЕ
Құрамында мырыш бар техногендік шикізатты қайта өңдеу табиғи ресурстарды ұтымды пайдалану және экологиялық жүктемені азайту тұрғысынан маңызды міндет болып табылады. Соңғы жылдары мұндай материалдарды өңдеудің тиімді әдістеріне қызығушылық артып келеді, өйткені мырыш өнеркәсіп үшін маңызды металл болып табылады. Шикізаттан мырышты күкірт қышқылының ерітіндісімен шаймалау мырыштың гидрометаллургиялық өндірісіндегі негізгі қадам болып табылады. Бұл процесті оңтайландыру өндіру тиімділігін арттыру, шығындарды азайту және қоршаған ортаға теріс әсерді азайту үшін өте маңызды. Біз мырышты техногендік шикізаттан күкірт қышқылының ерітіндісімен микротолқынды сәулелендірумен алдын ала өңдеу арқылы шаймалау процесін зерттедік. Оңтайлы шаймалау параметрлері анықталды. Бастапқы шикізат ретінде мырыш кенінің вельцтеу өндірісінің қалдық өнімі клинкер пайдаланылды. Клинкердегі мырыш мөлшері - 1,25 %. Мырыштың жоғары экстракциясына 600 °C температурада микротолқынды сәулелендірудің алдын ала әсерінен кейін қол жеткізілгені көрсетілді. Шаймалаудың оңтайлы параметрлері анықталды.

Түйін сөздер: шаймалау, мырыш, клинкер, ультра жоғары жиілікті сәулелендіру.

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Исследование выщелачивания цинка из клинкера с предварительной обработкой сырья сверхвысокочастотным излучением

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АННОТАЦИЯ
Переработка цинкосодержащего техногенного сырья представляет собой важную задачу с точки зрения рационального использования природных ресурсов и снижения экологической нагрузки. В последние годы наблюдается рост интереса к эффективным методам переработки таких материалов, поскольку цинк является важным металлом для промышленности. Выщелачивание цинка из сырья раствором серной кислоты является ключевым этапом в гидрометаллургическом производстве цинка. Оптимизация этого процесса имеет решающее значение для повышения эффективности его извлечения, снижения затрат и минимизации негативного воздействия на окружающую среду. Нами исследован процесс выщелачивания цинка из техногенного сырья раствором серной кислоты с предварительной обработкой сырья СВЧ-излучением. Определены оптимальные параметры выщелачивания. В качестве исходного сырья использовали клинкер – остаточный продукт вельцевания цинковых руд. Содержание цинка в клинкере - 1,25 %. Показано, что высокое извлечение цинка достигнуто после предварительного воздействия СВЧ излучения при температуре 600 °C. Изучено влияние концентрации растворителей, соотношения твердой и жидкой фаз, температуры на степень извлечения цинка.

Ключевые слова: выщелачивание, цинк, клинкер, сверхвысокочастотное излучение.

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