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Extraction of cerium from Iron Ore Mine Tailings by Leaching and Solvent Extraction Method

Mohammad Karimi and Mohammad Karamoozian*

Faculty of Mining, Petroleum & Geophysics Eng., Shahrood University of Technology, Shahrood, Iran

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Abstract

This research investigates the process of cerium extraction from tailings that have been separated from iron using a magnetic drum separator, through both acid leaching and solvent extraction methods. Initially, the mineralogical characteristics of the samples were analysed using microscopic studies. The main minerals identified were feldspar, garnet, calcite, gypsum, amphibole, and secondary minerals such as chlorite, quartz, and apatite. The metallic minerals were included pyrite, chalcopyrite, magnetite, and hematite. The sample was taken from the tailing's damps, then it was crushed to a particle size of less than 800 microns. The sample was then placed in a stirred tank along with water, fed into a spiral separator, and subsequently into a shaking table. The analysis results showed that the cerium grade increased from 320 ppm in the feed to 1364 ppm. In the leaching experiments, the influence of various parameters including temperature, acid concentration, type of acid, leaching time, and particle size on cerium leaching recovery rate was evaluated. The results indicated that temperature and acid concentration had the greatest impact on the leaching rate of cerium. In this stage, 95% of cerium was dissolved. Optimization tests for leaching conditions showed that the best conditions for cerium leaching were using hydrochloric acid at a 1:3 concentration with water (concentration of hydrochloric acid became 9 Molar), at a temperature of 90°C and a leaching time of 4 hours. In the subsequent phase, an optimization experiment was conducted with the same parameters. Under these conditions, 96.5% of cerium was dissolved. Then, the solvent extraction process was examined using organic solvents, di(2-ethylhexyl) phosphoric acid and tributyl phosphate. The results showed that the highest cerium extraction rate (81%) was achieved when di(2-ethylhexyl) phosphoric acid was used, considering parameters such as pH 3, organic-to-aqueous phase ratio of 1:1, 20-minute extraction time, 25°C temperature, and stirring speed of 300 rpm. Finally, the results of this research contribute to the optimization of the cerium extraction process and provide suggestions for improving the efficiency of this process.

1. Introduction

Rare Earth Elements (REEs) are a group of 17 chemical elements in the periodic table, consisting of 15 lanthanides (from lanthanum to lutetium) along with scandium and yttrium. Due to their similar chemical and physical properties, these elements are commonly found together in various minerals and ores. Based on their geochemical behaviour, rare earth elements are categorized into two groups: light rare earth elements (LREEs) and heavy rare earth elements (HREEs). The LREE group includes lanthanum (La), cerium (Ce), praseodymium (Pr), neodymium (Nd), and samarium (Sm). The HREE group includes europium (Eu), gadolinium (Gd), terbium (Tb), dysprosium (Dy), holmium (Ho), erbium (Er), thulium (Tm), ytterbium (Yb), lutetium (Lu), and yttrium (Y) [1].

Cerium, one of the rare earth elements and a member of the lanthanide series, is represented by the symbol "Ce" and atomic number 58 in the periodic table [2]. It is classified as a soft, lustrous metal and is highly stable in combination with

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Corresponding author: m.karamoozian@shahroodut.ac.ir (M. Karamoozian)

oxygen in the form of cerium oxide (CeO_2). Cerium is considered one of the most important metals in the rare earth industry and is widely used in various sectors, including alloy production, catalysts, pigments, solar panels, light-emitting diodes (LEDs), and glass polishing [3]. It is also used in the manufacture of permanent magnets and phosphorescent powders. Due to its unique chemical and physical properties, cerium plays a vital role in many industrial and technological processes, particularly in the automotive and electronics industries [4].

Cerium is primarily extracted from rare earth-containing minerals such as bastnäsite, monazite, and loparite. Major reserves of this element are found in China, the United States, and Australia [5]. Given the growing demand for cerium and its compounds, particularly in advanced technology industries, its separation and purification have become a major challenge in materials science and chemistry. Various techniques such as solvent extraction, ion exchange, adsorption, and precipitation are employed to extract and purify cerium from other rare earth elements and impurities [6].

During mining activities and the operation of crushing and magnetic beneficiation plants, waste dumps with different mineralogical compositions are formed, which certainly exhibit different behaviours during beneficiation processes. Therefore, the first step is to identify the samples and determine their mineralogical characteristics and processing behaviour.

The aim of this study is the separation of cerium from the loaded leach solution using solvent extraction. In this regard, two extractants di-(2-ethylhexyl) phosphoric acid (D2EHPA) and tributyl phosphate (TBP) in kerosene as a diluent were used, and the effects of various parameters were investigated. Also, in order to increase the cerium grade prior to the leaching process, a pre-concentration stage is carried out using gravity separation techniques, including spiral separators and shaking tables.

2. Material and Methods

A total of 500 kg of samples were collected from various parts of the tailings dump for experimental studies. Since the particle size was 10 mm, the sample was ground in a ball mill to below 800 microns in a controlled manner. After homogenization, riffle splitting, and dividing the sample to obtain a representative portion, it was sent to the laboratory for ICP-MS, XRD, and thin polished section analysis. The results showed that the cerium grade in the tailings was 320 ppm.

Subsequently, 10 kg of sample was prepared for gravity separation tests using spiral and shaking table methods. Gravity separation is a technique that utilizes the difference in mineral densities to separate and extract cerium and other rare earth elements from mineral matrices. This method is particularly effective for ores containing both heavy and light mineral phases.

After conducting the spiral and shaking table tests to increase the cerium grade, it was observed that iron grade also increased along with cerium. To reduce the iron content, a dry magnetic separator was employed, and the magnetic fraction (magnetite-rich) was separated from the tailings.

3. Results

3.1. XRD analysis

X-ray diffraction (XRD) analysis was performed on the representative tailings sample from the iron ore processing plant. The analyses conducted using the XRD method on the submitted sample are presented in Table 1, and the corresponding XRD graphs can be seen in Figure 1.

Table 1 The results of the X-ray diffraction (XRD) analysis

Mineral	Formula
Albite	(Na0.98Ca0.02) (Al1.02Si2.98O8)
Andradite	Ca3Fe2(SiO4)3
Gypsum	CaSO4·2H2O
Riebeckite	Na1.38K0.13Ca0.17Mg0.25Mg2.81Fe1.66Fe0.48Al0.04Si7.94O22(OH)2
Clinochlore	(Mg0.99Al0.01)5(Al0.67Fe0.33) (Si3.02Al0.98) O10(OH)8
Calcite	CaCO3
Quartz	SiO2
Amorphous phase	-

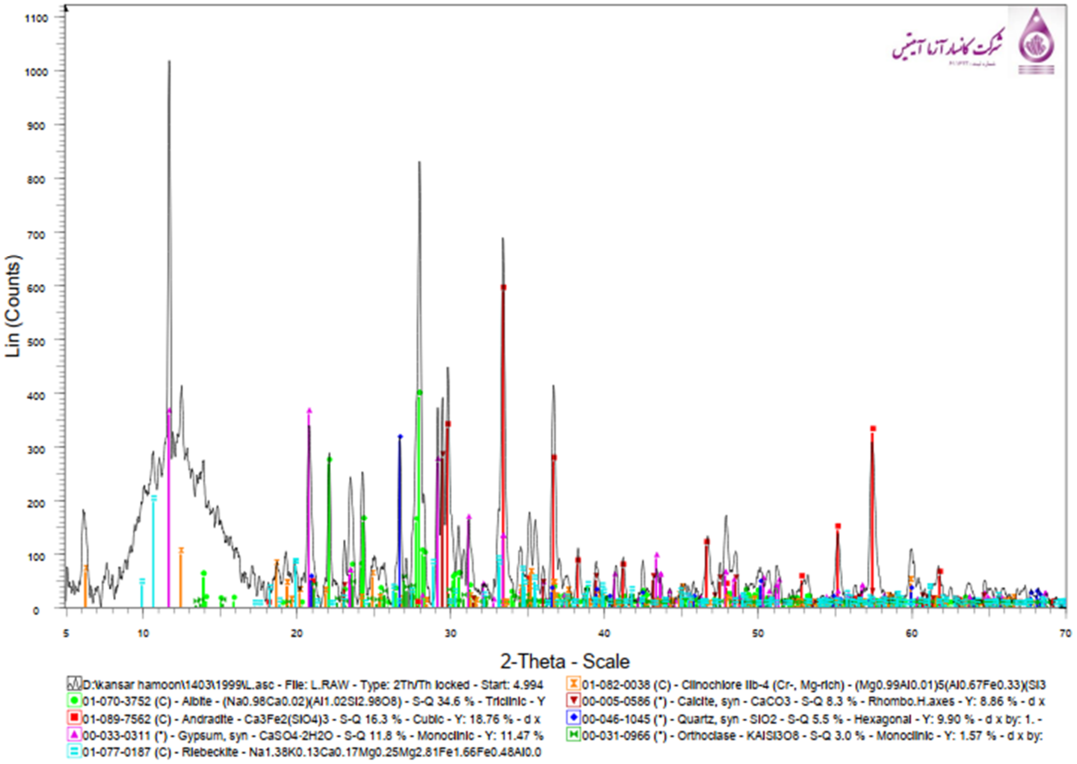

Figure 1. XRD graph

3.2. Thin polished section analysis

Macroscopic description: The sample was in powder form, which was molded and then prepared as a thin polished section.

- Main minerals: feldspar, garnet, calcite, gypsum, amphibole
- Accessory minerals: chlorite, quartz, opaque minerals
- Metallic minerals: pyrite, chalcopyrite, magnetite, hematite

✓ Microscopic description

As shown in Figure 2, The examined sample predominantly consists of feldspar, garnet, calcite, and amphibole, with lesser amounts of chlorite, gypsum, quartz, and opaque phases. These minerals appear as fine-grained and highly fractured. Feldspar is observed as fragmented crystals, probably albite, sometimes showing polysynthetic twinning. Garnet occurs as fine grains and occasionally subhedral fragmented crystals, approximately 600 microns in size.

Fragments of gypsum and calcite are seen as independent particles and occasionally in association. Carbonate-rich zones are also present. Amphibole appears as fibrous, green-colored crystals up to 200 microns, often associated with garnet and albite. opaque minerals appear as discrete grains up to 1 mm and are also embedded in rock fragments. Under reflected light, they were identified as pyrite, chalcopyrite, magnetite, and hematite.

- Pyrite appears as isolated grains with a spongy texture, up to 1 mm, and also as amorphous infillings in magnetite.
- Chalcopyrite, with lower abundance, fills spaces between garnet crystals.
- Magnetite appears subhedral, often undergoing replacement by hematite.

A cluster of fine particles with high relief and strong birefringence, likely monazite, was observed at the edge of a carbonate fragment, although it was lost during section preparation. Accurate identification of this phase requires complementary analyses such as SEM or EPMA.

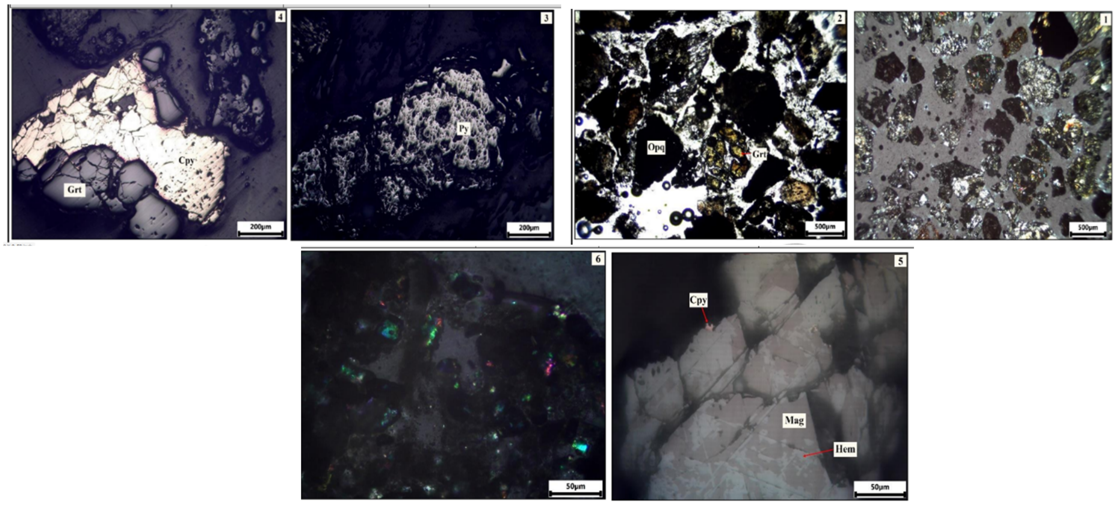

Figure 2. Microscopic pictures of minerals

3.3. Leaching test procedure

Following the spiral and shaking table gravity separation tests, the concentrate sample was ground using a ball mill in a controlled manner to two particle sizes: -50 microns and -100 microns, in preparation for leaching experiments.

The leaching experiments were conducted on the representative samples using 100 mL beakers placed on hot plates equipped with magnetic stirrers capable of controlling stirring speed. In the first stage, a total of 16 leaching tests were designed and performed.

Procedure for each leaching test was as follows:

Initially, acid solutions of sulfuric acid, nitric acid, and hydrochloric acid were prepared based on predefined concentrations. Then, 5 grams of the representative mineral sample were mixed with a specific volume of the prepared acid solution according to a set solid percentage. This mixture was transferred into the beaker.

After adjusting the stirring speed, the solution was stirred for a predetermined period of time. Upon completion of the leaching process, the resulting pulp was filtered, and the liquid phase (leachate) was sent to the laboratory for ICP-MS analysis to determine the cerium content.

This experiment was conducted at temperatures of 25, 45, 60, and 90°C, using three types of acids: hydrochloric acid, nitric acid, and sulfuric acid. It was carried out at different durations (1, 2, 3, and 4 hours), with various water-to-acid ratios (1:1, 1:2, 1:3, 1:4, 1:5), and using two particle sizes (-50 and -100 microns), across a total of 16 experiments.

3.3.1. The influence of important parameters on the leaching process

The following results were obtained using Design Expert software based on the Taguchi method.

✓ **Temperature**

In leaching processes, temperature is one of the most critical parameters that significantly affects metal recovery efficiency. For cerium, increasing the temperature leads to an enhancement in the rate of chemical reactions, resulting in higher recovery. As temperature rises, reaction rates increase. Chemical reactions generally proceed faster at higher temperatures due to increased molecular kinetic energy, which raises the probability of collisions between molecules and ions.

Furthermore, solubility increases at elevated temperatures. Many minerals and compounds—particularly metallic compounds like cerium—dissolve better in solvents under such conditions. As a result, more metals are transferred into the solution, improving overall recovery [7].

As shown in Figure 3, the leaching rate of cerium increases with temperature.

✓ **Acid type**

The type of acid used in the cerium leaching process is one of the key factors in determining the extraction efficiency of this element. Different acids, due to their distinct chemical properties, have varying effects on cerium extraction. In this study, sulfuric acid, nitric acid, and hydrochloric acid were used. Based on the recovery rates, hydrochloric acid showed the highest efficiency in cerium leaching.

Figure 4 illustrates the effect of acid type on the leaching process.

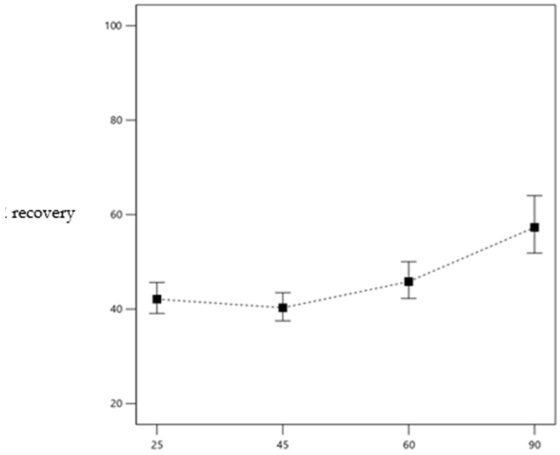

Figure 3. Effect of temperature

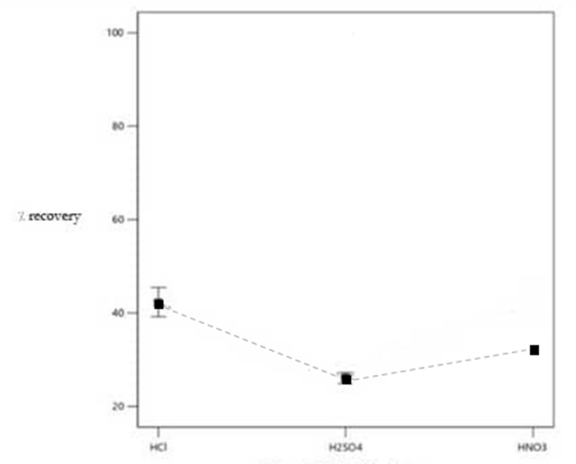

Figure 4. Effect of acid type

✓ **Particle size**

The particle size in the cerium leaching process is an important factor that can significantly influence extraction efficiency and reaction kinetics. The size of the mineral particles directly affects the contact surface between the material and the leaching solution, which can either enhance or reduce the performance of the leaching process. As particle size decreases, the surface area available for reaction increases, resulting in shorter extraction times for cerium.

This is particularly important in slow chemical reactions. Finer particles lead to a reduction in leaching time and an increase in extraction rate. These smaller particles, having a larger surface area, can react more rapidly and extensively with acid or other leaching agents, thereby increasing the dissolution rate and overall recovery of cerium.

In this study, two particle sizes were used: under 50 microns and under 100 microns. As shown in Figure 5, the leaching recovery rate was higher for the -50-micron particle size.

✓ **Time**

Leaching time has a direct impact on the rate of chemical reactions. These reactions typically occur more rapidly during the initial stages of the process and then slow down over time as the dissolution of cerium progresses. This phenomenon is due to changes in the contact surface area and the concentration of the solution.

As shown in Figure 6, cerium recovery increases with increasing leaching time.

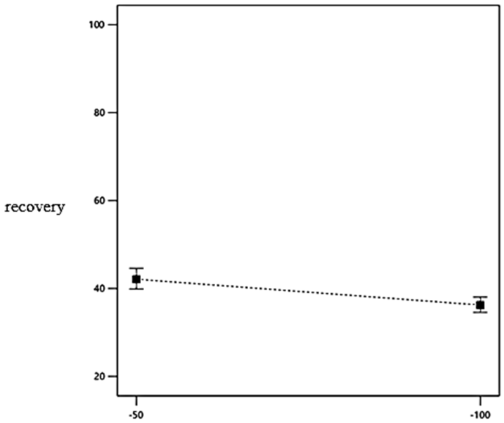

Figure 5. Effect of particle size

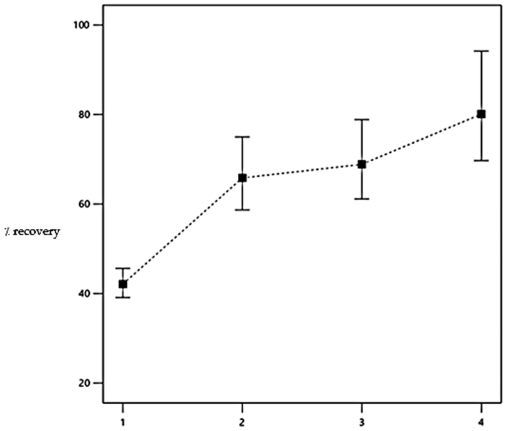

Figure 6. Effect of time (hour)

✓ Acid Concentration

The acid concentration plays a key role in optimizing cerium extraction processes. It significantly influences the rate of chemical reactions, solvent selectivity, process efficiency, and the overall recovery of cerium. This section discusses in detail the effect of acid concentration on the leaching rate of cerium. In cerium leaching processes, acid acts as the leaching agent, dissolving cerium compounds from the mineral matrix. As acid concentration increases, the rate of chemical reactions typically rises. This is because a higher acid concentration enhances the acid's ability to dissolve mineral compounds, particularly cerium-bearing phases.

An increase in acid concentration leads to a higher dissolution rate of cerium due to a greater number of acid molecules available in the solution, which in turn increases the frequency of chemical interactions. As shown in Figure 7, the highest cerium recovery was achieved at a 1:3 water-to-hydrochloric acid ratio, at which point the concentration of hydrochloric acid reached approximately 9 Molar.

Based on the results of the optimization experiments, the optimal conditions for cerium leaching are generally as follows:

- Type of acid: hydrochloric acid
- Acid concentration: 1:3 (acid to water ratio),
- Operating temperature: 90 °C.
- Leaching time: 4 hours.
- Particle size: 50 microns.

Based on the analysis of the results and the optimization of factors affecting the acidic leaching of cerium, an experiment was conducted under the following conditions: hydrochloric acid, a water-to-acid ratio of 1:3, leaching time of 4 hours, particle size of 50 microns, and a temperature of 90°C. The cerium recovery in this experiment reached 96.5%.

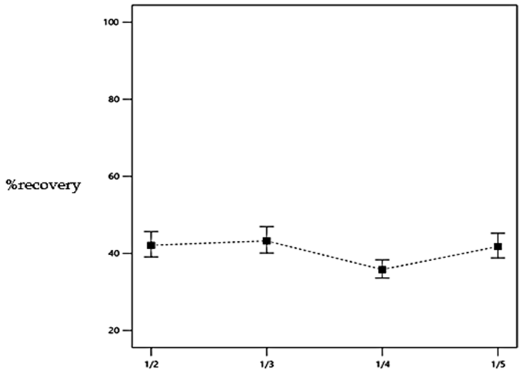

Figure 7. Acid concentration

3.4. Solvent extraction

The optimized leach solution is used in the leaching step for solvent extraction. As shown in Table 2, In this solvent extraction experiment, all parameters—including temperature, time, stirring speed, pH, and the organic-to-aqueous phase ratio—were kept constant, while only the ratio of D2EHPA (di-(2-ethylhexyl) phosphoric acid) to TBP (tributyl phosphate) was varied. The main objective of this study is typically to investigate the effect of changes in the ratio of these two extractants on recovery, compound transfer, and the thermodynamic aspects of the extraction process.

Table 1. Solvent extraction results.

number	(RPM) Stirrer speed	Temperature Centigrade	o/a	Dehpha/TBP Ratio	Time (min)	Extraction (%)
1	300	25	1:1	D2EHPA	20	81.46
2				TBP		80.28
3				TBP 1:1D2EHPA		69.25
4				TBP 1:3 D2EHPA		35.50
5				TBP 3:1 D2EHPA		25.33

According to the results of the solvent extraction, as observed, the highest extraction percentage is 81%, which corresponds to the case where D2EHPA is used alone as the extractant in the reaction.

4. Conclusions

The results obtained from the pre-processing using the spiral and shaking table:

The results obtained from the spiral and shaking table tests, as presented in the table, show that the cerium grade increased from 320 ppm to 1364 ppm. Additionally, the weight recovery reached 6%, and the metallurgical recovery was 25%, indicating that the performance of the spiral and shaking table was relatively good.

The results obtained from the leaching and solvent extraction processes.

1. The effects of key operational parameters including leaching agent concentration, leaching temperature, leaching time, acid concentration, type of acid, and particle size—were investigated. The findings indicated that among the studied factors, leaching temperature, acid concentration, acid type, and leaching time had the most significant impact on cerium recovery through acidic leaching.
2. The maximum cerium recovery rate in the leaching experiments was 95%, achieved under the operating conditions of 400 RPM stirring speed, a water-to-acid ratio of 1:3(concentration of hydrochloric acid became 9 Molar), leaching temperature of 90°C, a liquid-to-solid ratio of 10, and a leaching duration of 4 hours.
3. The optimized leaching experiment resulted in the dissolution of 96.5% of cerium, with all optimal conditions being considered in this test.
4. The highest cerium extraction rate (81%) using the organic solvent D2EHPA was obtained under the conditions of 300 rpm stirring speed, pH = 3, temperature of 25°C, an organic-to-aqueous phase ratio of 1, and an extraction time of 20 minutes.
5. The synergistic effect of organic solvents on cerium extraction was studied using two binary mixtures of organic extractants: tributyl phosphate (TBP) and D2EHPA. In both cases, the results showed that the cerium extraction rate using D2EHPA alone was higher than that achieved using the synergistic solvent extraction system. Therefore, based on these findings, cerium extraction using D2EHPA alone was preferred over the synergistic extraction method.

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استحصال سریم از باطله معدن سنگ آهن به روش لیچینگ و استخراج حلالی

محمد کریمی و محمد کارآموزیان*

دانشکده مهندسی معدن، نفت و ژئوفیزیک ، دانشگاه صنعتی شاهرود، شاهرود، ایران

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چکیده

این تحقیق به بررسی فرآیند استحصال سریم از باطله‌های خردایش شده که به وسیله درام سپراتور مغناطیسی از آهن جدا شده اند به دو روش لیچینگ اسیدی و استخراج حلالی پرداخته است. در ابتدا، با استفاده از مقطع نازک-صیقلی و آزمایش XRD خصوصیات کانی‌شناسی نمونه‌ها مورد تجزیه و تحلیل قرار گرفت. کانی های اصلی تشکیل دهنده فلدسپار، گارنت، کلسیت، ژیپس، آمفیبول وکانی های فرعی کلریت، کوارتز، اپک و همچنین کانی های فلزی پیریت، کالکوپیریت، مگنتیت، هماتیت بودند. نمونه‌ی معرف گرفته شده از دپو باطله به سایز منفی ۸۰۰ میکرون خردایش شد به طوری که d۸۰ نمونه ۸۰۰ میکرون شد. سپس نمونه در یک مخزن همزن دار همراه با آب به دستگاه اسپیرال و بعد از آن به میز لرزان با پمپ خوراک دهی شد. نتیجه آنالیز ها به این صورت بود که عیار سریم از ppm ۳۲۰ در خوراک به ppm۱۳۶۴ افزایش پیدا کرد. در آزمایش لیچینگ تأثیر پارامترهای مختلف شامل دما، غلظت اسید، نوع اسید، زمان لیچینگ و اندازه ذرات بر روی نرخ لیچینگ سریم در فرآیند لیچینگ اسیدی ارزیابی شد. نتایج نشان داد که دما و غلظت اسید بیشترین تأثیر را بر نرخ لیچینگ سریم داشته است. در این مرحله ۹۵ درصد سریم حل شد. آزمایش‌های بهینه‌سازی شرایط لیچینگ نشان داد که بهترین شرایط برای لیچینگ سریم شامل استفاده از اسید هیدروکلریک با غلظت ۱ به ۲ با آب، دمای ۹۰ درجه سانتی‌گراد و زمان ۴ ساعت است. در مرحله بعدی یک آزمایش بهینه با عوامل ذکر شده انجام گرفت که در این مرحله به ۹۶/۵ درصد حل شد. سپس فرآیند استخراج حلالی با استفاده از حلال‌های آلی دی (اتیل هگزیل) فسفریک اسید و تری بوتیل فسفات مورد بررسی قرار گرفت. نتایج نشان داد که بالاترین نرخ استخراج سریم در استفاده از دی (اتیل هگزیل) فسفریک اسید با در نظر گرفتن عواملی نظیر pH 3، نسبت فاز آلی به آبی یک، زمان ۲۰ دقیقه، دما ۲۵ درجه و سرعت همزن rpm۳۰۰ به بازیابی ۸۱ رسید. در نهایت، نتایج این تحقیق به بهینه‌سازی فرآیند استخراج سریم کمک کرده و پیشنهاداتی برای بهبود راندمان این فرآیند ارائه شده اس.

کلمات کلیدی

باطله‌های آهن
استخراج سریم
لیچینگ اسیدی
استخراج با حلال

نویسنده مسئول مکاتبات: *m.karamoozian@shahroodut.ac.ir* **(م. کارآموزیان)**