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## Assessment of the prospects for processing oxidized nickel ores using microwave energy

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Abstract: The Ural region holds an estimated 1.5 million tons of nickel reserves, located in the industrially developed Chelyabinsk, Sverdlovsk, and Orenburg regions. At present, however, these deposits are not being exploited, and metallic nickel is not produced in the Urals, as metallurgical facilities have been completely shut down. The reserves are represented by oxidized nickel ores (ONO) — a complex raw material with low nickel and cobalt contents, whose processing by existing technologies is economically unfeasible. The challenge is compounded by the absence of a beneficiation method for ONO that yields a concentrate; therefore, all current technologies require processing the entire ore mass, which results in high reagent consumption and substantial energy costs. Research is ongoing to develop new technological approaches, including alternative methods for extracting nickel and cobalt from ONO in the Ural deposits. One promising option is the use of microwave (MW) energy to unlock nickel-bearing minerals and accelerate the dissolution of nickel and cobalt. This study evaluates the effect of microwave energy on nickel recovery from oxidized nickel ores of the Ural region. Comparative data are presented for conventional sulfuric acid leaching and for the process with microwave energy applied. A series of test studies was carried out to assess the feasibility of using microwave energy for ONO processing. The comparison of technological parameters demonstrated the advantage of atmospheric sulfuric acid leaching with microwave energy, which achieved nickel recovery of up to 95 % in a relatively short time. These results identify this approach as the most promising for practical implementation.

Keywords: microwave energy, oxidized nickel ores, leaching, sulfuric acid, nickel.

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# Оценка перспективности переработки окисленных никелевых руд с использованием СВЧ-энергии

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**Аннотация:** Урал обладает запасами никеля на уровне 1,5 млн т. Месторождения находятся в промышленно развитом регионе на территории Челябинской, Свердловской и Оренбургской областей. Однако в настоящее время их не разрабатывают, и металлический никель на Урале не производят, так как металлургические предприятия полностью остановлены. Причиной является то, что запасы никеля представлены окисленными никелевыми рудами (ОНР), которые являются сложным сырьем с низким содержанием никеля и кобальта, переработка которого по существующим технологиям нерентабельна. Осложняет задачу и то, что на сегодняшний день не существует метода обогащения ОНР с получением концентрата, поэтому все технологии предусматривают переработку всей массы руды, что ведет к значительным расходам на реагенты и энергетическим затратам. В то же время не прекращается поиск новых технологических подходов, ориентированных на применение альтернативных вариантов извлечения никеля и кобальта из ОНР уральских месторождений. Одним из подобных методов является применение СВЧ-энергии для вскрытия никелевых минералов и интенсификации перевода в раствор никеля и кобальта. В настоящей работе оценено влияние воздействия СВЧ-энергии на извлечение никеля из окисленных никелевых руд Уральского региона. Приводятся данные по сравнению показателей классического серно-кислотного выщелачивания и процесса с наложением СВЧ-энергии. Выполнен комплекс тестовых исследований, цель которых — оценить перспективность применения СВЧ-энергии для переработки ОНР. Сравнение технологических параметров обоих подходов выявило преимущество атмосферного серно-кислотного выщелачивания ОНР с наложением СВЧ-энергии, в ходе которого было достигнуто извлечение никеля в раствор до 95 % за небольшой промежуток времени. На основании полученных результатов данное направление выбрано как наиболее перспективное для практической реализации.

Ключевые слова: СВЧ-энергия, окисленные никелевые руды, выщелачивание, серная кислота, никель.

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#### Introduction

In both global and domestic practice, nickel is extracted from sulfide and oxidized ores. Sulfide ores are subjected to flotation beneficiation, and the resulting flotation concentrate is processed by pyrometallurgical methods [1]. Oxidized (lateritic) ores account for 60-70 % of the world's nickel reserves. These ores cannot be beneficiated by conventional methods, and nickel extraction directly from raw ore is associated with high specific costs. Therefore, the search for efficient technologies for processing oxidized nickel ores (ONO) remains highly relevant [2-4]. Abroad, laterite ore processing is generally carried out by hydrometallurgical or combined methods [5-8].

Hydrometallurgical technologies implemented in practice for ONO processing are based on direct leaching of nickel and cobalt with sulfuric acid in two main variants:

- heap leaching of raw ore with sulfuric acid solutions [9—12];
- high-pressure acid leaching under autoclave conditions (HPAL) [13;14].

In some cases, heap leaching is combined with autoclave leaching to reduce specific costs: raw ore is subjected to pressure leaching, while the residue from the autoclave stage is processed by heap leaching.

The main challenges in applying hydrometallurgical technologies to ONO from the Ural region stem from the low contents of valuable components (nickel and cobalt). The specific mineralogical features of these ores result

in high reagent consumption, low recovery of target metals, and the formation of large volumes of solid waste requiring disposal. These factors render traditional hydrometallurgical technologies economically unfeasible and hinder the adaptation of foreign processing schemes to domestic ores.

It is known that in ONO, most nickel occurs as an isomorphic substitute for iron in poorly soluble silicate minerals [14]. To intensify solid—liquid reactions, elevated temperatures, pressure, and vigorous mixing are used.

The greatest potential for accelerating hydrometallurgical interactions under optimal reagent conditions in liquid—solid systems lies in enhancing mass transfer. Various approaches have been proposed to accelerate heterogeneous solid—liquid processes, including the application of oscillations of different frequencies acoustic and ultrasonic waves, industrial-frequency current, as well as high and ultra-high frequencies.

Microwave (MW) energy is widely applied across diverse industries such as petrochemistry, agriculture, food processing, wood drying, medicine, and others. In these applications, it is generally used solely for accelerated heating of materials. Only in petrochemistry and catalytic chemistry has microwave energy been applied to intensify mass transfer processes.

The aim of the present work is to evaluate the potential for intensifying acid leaching of nickel from oxidized ore by applying microwave energy to the reaction mass

and to compare the results with those of conventional nickel leaching methods..

#### Research methods and results

The object of study was an increment of oxidized nickel ore from the Ural region. The increments were composited, and a laboratory sample was prepared and ground to a particle size below 0.074 mm. Phase analysis revealed that the main minerals in the ore were goethite (Fe(OH)<sub>2</sub>·nH<sub>2</sub>O), silica (SiO<sub>2</sub>), and nickel silicate (NiO<sub>2</sub>·SiO<sub>2</sub>). In addition, alumina (Al<sub>2</sub>O<sub>3</sub>), manganese hydroxides, calcium compounds, and other phases were present. Alumina and silica constituted the basis of the clay-forming minerals. According to chemical analysis, the ore contained (wt. %): Ni - 0.92, Co - 0.064, and Fe - 39.32.

To investigate the effect of microwave irradiation on ONO, a laboratory unit was assembled based on a commercially available inverter-type microwave oven, which maintains constant output power during operation. This allowed more accurate evaluation of specific energy consumption and minimized pulp overheating.

The pulp (ore mixed with acidic solution) was placed in a heat-resistant laboratory beaker and set inside the microwave oven directly over the waveguide outlet. To achieve this configuration, the oven was oriented vertically. To evaluate the effect of microwave irradiation, it was necessary to ensure that the entire microwave energy generated by the magnetron was focused on the reaction mass. For this purpose, the beaker was wrapped in an aluminum foil shield. These methodological arrangements allowed reliable determination of specific energy consumption, optimal pulp layer thickness (i.e., effective penetration depth of microwave energy), and the overall efficiency of the method.

A schematic of the laboratory setup is presented in Fig. 1.

Concentrated sulfuric acid solutions (≥80 g/dm³) were used in the experiments, heated directly by microwave irradiation. To protect the magnetron from thermal and chemical effects of the reaction mass, the waveguide outlet was additionally covered with a screen made of mica and asbestos, both transparent to microwaves.

At the first stage, the effect of microwave energy on the kinetics of nickel leaching was examined by conducting comparative tests under identical conditions: liquid-to-solid ratio (L/S) = 5:1, stirring intensity 400 rpm, temperature  $\leq 75$  °C, and  $H_2SO_4$  concentra-

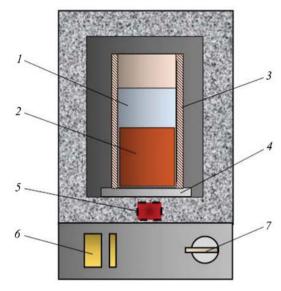


Fig. 1. Schematic of the laboratory setup

I – reaction beaker, 2 – pulp, 3 – aluminum foil shield, 4 – support for the reaction beaker made of microwave-transparent material, 5 – magnetron, 6 – timer, 7 – power controller

#### Рис. 1. Схема лабораторной установки

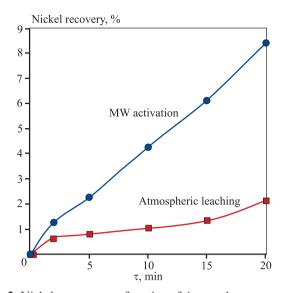
I— реакционный стакан, 2— пульпа, 3— экранирующий кожух, 4— подставка под реакционный стакан из СВЧ-прозрачного материала, 5— магнетрон, 6— датчик времени, 7— регулятор управления

tion 200 g/dm<sup>3</sup>. Parallel ore charges of equal mass were leached in a beaker on a conventional heating plate and in the microwave-assisted setup. Microwave activation was performed at an output power of 180 W for 20 min in both cases.

At specified time intervals, aliquots of the pregnant solution were withdrawn and analyzed for nickel content by atomic absorption spectroscopy (Analytik Jena, Germany). Nickel recovery into solution was then calculated (Fig. 2).

As expected, microwave irradiation significantly accelerated nickel dissolution compared with conventional thermal leaching, increasing the leaching rate by approximately fourfold. Although the maximum achievable nickel recovery was not determined in this series, the observed trend suggests that microwave activation substantially shortens the time required to achieve high extraction.

For practical implementation of the proposed method and reactor design, it was important to estimate the penetration depth of microwave energy into the solid—liquid system (a pulp consisting of sulfuric acid solution and ground ore at a liquid-to-solid ratio of 1:1). To vary the pulp layer thickness, different volumes of pulp were loaded into the reaction vessel. A separate beaker containing a fixed volume of distilled



**Fig. 2.** Nickel recovery as a function of time under microwave-assisted and atmospheric leaching of ONO

**Рис. 2.** Сравнение динамики выщелачивания никеля из OHP

water was placed above the reaction vessel. When the microwave oven was switched on, the extent of heating of the water indicated the fraction of microwave energy that had not been absorbed by the pulp. Thus, by comparing the temperature rise in the water at different pulp heights, the penetration depth of the microwave energy flux through the reaction mass was assessed. The chosen pulp density (L/S=1:1) minimized particle settling during the test, ensuring reliable evaluation of microwave penetration depth.

The absorbed energy was calculated from the temperature difference before and after irradiation according to the equation:

$$E = \frac{mC\Delta t}{\tau},$$

where E is the absorbed energy, W/s; m is the mass of water, g;  $C = 4.18 \text{ J/g} \cdot ^{\circ}\text{C}$  is the specific heat capacity of water;  $\Delta t$  is the temperature difference,  $^{\circ}\text{C}$ ; and  $\tau$  is time, s.

The amount of microwave energy absorbed by the reaction mass at different layer thicknesses is presented in the table.

The results show that in dense pulp, the microwave flux is fully absorbed within a 4—6 cm layer. Naturally, this parameter depends on microwave power, pulp density, and mixing conditions. The penetration depth of microwave energy — where heating occurs and reactions are intensified — is referred to as the effective microwave depth. Within this zone, heating, the skin effect, and accelerated dissolution of target metals can be expected.

In the next stage, the effect of specific sulfuric acid consumption on nickel leaching was evaluated. Since an additional aim was to estimate the maximum achievable recovery, experiments were performed at maximum magnetron power (900 W) with deliberately excess acid addition.

The leaching solution was prepared by dissolving the required mass of acid in water and diluting to a fixed volume. Ore charges weighed 30 g, and the solution volume was 60 ml. At this power, the reaction mass rapidly evaporated, forming a dense sinter that fundamentally altered the process. To avoid this, the experiment duration was limited to 4 minutes. Monitoring the temperature of the reaction mass during microwave activation was not feasible; post-experiment measurements showed that under steady-state conditions the temperature rapidly reached 95—100 °C.

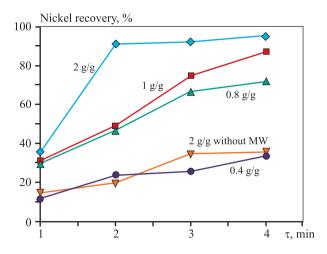
Given the expected high reaction rate between nickel-bearing minerals and sulfuric acid, the acid concentration was the critical factor in the leaching process. To enable proper comparison of experimental results at equal slurry volumes (effective layer thickness of 4 cm) and different acid concentrations, the specific acid consumption was varied from 0.4 to 2.0 g  $\rm H_2SO_4$  per gram of ore.

The ore charges were mixed with sulfuric acid solution and subjected to microwave irradiation for a set leaching time (e.g., 1 min). The pulp was then removed, diluted with water, filtered, and the filtrate analyzed for nickel concentration and residual acid. Leaching was repeated at the same acid consumption but with different leaching times. Thus, each point on the curves in Figs. 3 and 4 corresponds to a separate test. For comparison, additional experiments at 2 g H<sub>2</sub>SO<sub>4</sub> per g ore were performed by heating the pulp to 95 °C without microwave activation. Based on these results, nickel recovery into solution and the fraction of sulfuric acid consumed in the target reaction were calculated.

### Microwave energy absorbed by the reaction mass at different pulp layer heights

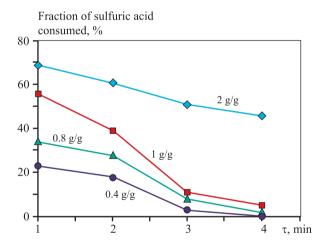
Количество СВЧ-энергии, поглощенной реакционной массой при разной высоте слоя пульпы

Pulp height, mm	Absorbed energy, J	Energy loss, %
10	253.6	71.8
20	612.3	32.0
40	891.4	1.0
60	895.1	0.5



**Fig. 3.** Dependence of nickel recovery on sulfuric acid consumption under microwave-assisted leaching of ONO (g/g ore)

**Рис. 3.** Зависимость извлечения Ni в раствор при выщелачивании с наложением СВЧ-энергии при различном расходе серной кислоты (г/г руды)



**Fig. 4.** Dependence of sulfuric acid consumption on time under microwave-assisted leaching of ONO (g/g ore)

**Рис. 4.** Зависимость расхода серной кислоты при СВЧ-выщелачивания ОНР (г/г руды)

The results show that under excess (i.e., high concentration of) sulfuric acid, the nickel leaching rate under microwave activation increased severalfold compared with conventional methods.

The experiments were not aimed at determining reagent consumption coefficients or performing an economic assessment of this approach, but the results demonstrate that under certain conditions, near 100 % nickel recovery can be achieved in a very short time. With countercurrent leaching — widely used in hydrometallurgy — this method could achieve the

required metal recovery at high rates. Overall, the findings confirm the potential of microwave energy for enhancing the efficiency of metallurgical ore processing.

Further studies will focus on the features of microwave-assisted leaching of oxidized nickel ore under countercurrent liquid—solid conditions.

#### **Conclusions**

- **1.** An original methodology was proposed for studying metal leaching from mineral raw materials using microwave energy.
- **2.** It was established that the effective penetration depth of microwave energy through a dense solid—liquid system ("ground ore leaching solution") at a liquid-to-solid ratio of 1:1 does not exceed 40 mm.
- **3.** Atmospheric sulfuric acid leaching of ONO with microwave activation was shown to accelerate the process severalfold, achieving up to 95 % nickel recovery into solution.

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#### **Contribution of the authors**

- **S.E. Polygalov** development of the main concept, definition of research objectives and tasks, preparation of the manuscript, formulation of conclusions.
- **V.G. Lobanov** scientific supervision, revision of the manuscript and conclusions.
- $\begin{tabular}{ll} \textbf{D.S. Sedelnikova}-\text{experiment preparation and experimental}\\ work. \end{tabular}$
- ${f O.B.}$  Kolmachikhina calculations and preparation of the manuscript.
- $\mbox{O.Yu.}$   $\mbox{Makovskaya} \mbox{calculations}$  and analysis of research results.

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