

ВЛИЯНИЕ КОНЦЕНТРАЦИЙ СЕРНОЙ КИСЛОТЫ НА ОБРАЗОВАНИЕ ГЕЛЯ В ПРОЦЕССЕ ВЫЩЕЛАЧИВАНИЯ СЕРПЕНТИНА

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Аннотация: Представлены результаты исследования влияния концентрации серной кислоты на процесс выщелачивания серпентина, с особым акцентом на механизм образования геля поликремниевых кислот. Проведен и рассмотрен детальный анализ кинетических особенностей растворения серпентинита в различных условиях и соотношениях в минеральной кислоте, в частности, в серной. Установлено, что ключевым фактором, ограничивающим скорость процесса, является формирование коллоидных форм кремнезема на поверхности частиц, что приводит к пассивации реакционной зоны. С использованием методов ИК-Фурье спектроскопии и химического анализа показано, что трансформация кремнезема из кристаллической структуры серпентинита в аморфное состояние начинается при достижении концентрации серной кислоты 40–50% от стехиометрически необходимого количества. Обнаружено, что при таких условиях на поверхности частиц образуется устойчивый слой геля, существенно замедляющий дальнейшее растворение магния. Полученные данные позволяют определить оптимальные параметры кислотной обработки серпентинитов, обеспечивающие максимальное извлечение целевых компонентов при минимальном гелеобразовании. Результаты исследования имеют важное практическое значение для разработки эффективных технологий переработки серпентинитового сырья, включая получение соединений магния и кремнезема.

Ключевые слова: серная кислота, магний, серпентинит, кремнезем, ИК-Фурье спектроскопия, окружающая среда.

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Effect of sulfuric acid concentrations on gel formation during serpentine leaching

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Abstract: This article presents the results of a study of the effect of sulfuric acid concentration on the leaching process of serpentinite, with a special focus on the mechanism of polysilicon acid gel formation. A detailed analysis of the kinetic features of the dissolution of serpentinite under various conditions and ratios in mineral acid, in particular, in sulfuric acid, has been carried out and considered. It has been established that the key factor limiting the rate of the process is the formation of colloidal forms of silica on the surface of the particles, which leads to passivation of the reaction zone. Using the methods of IR-Fourier spectroscopy and chemical analysis, it is shown that the transformation of silica from the crystalline structure of serpentinite to an amorphous state begins when the concentration of sulfuric acid reaches 40–50% of the stoichiometrically required amount. It was found that under such conditions, a stable gel layer forms on the surface of the particles, which significantly slows down the further dissolution of magnesium. The data obtained make it possible to determine the optimal parameters of acid treatment of serpentinites, ensuring maximum extraction of target components with minimal gelation. The results of the study are of great practical importance for the development of effective technologies for processing serpentinite raw materials, including the production of magnesium and silica compounds.

Key words: sulfuric acid, magnesium, serpentinite, silica, IR-Fourier spectroscopy, environment.

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Introduction

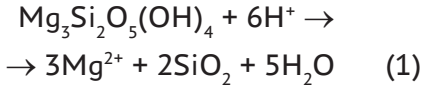
Serpentinite is a multifunctional raw material, it is magnesian silicate minerals rich in magnesium (up to 43.0 wt.% MgO) and silica (44–45 wt.% SiO₂) [1, 2], contain impurity metals such as Fe, Ni, Cr, Al and Mn [3, 4]. They are widely studied in order to use them in the production of magnesium and its compounds, silica [5, 6], and nickel, when nickel-containing compounds (up to 0.8–1.0 wt.% Ni) serpentinites, where nickel is in the oxide form [7, 8]. In addition, carbon dioxide capture technology is being developed [9, 10]. In all these cases, one of the important aspects of their treatment is acidic application, the effectiveness of which depends on the nature of the acid used. The effect of various mineral acids (sulfuric, hydrochloric, nitric, and phosphoric) on the dis-

closure of the structural structure of rather durable serpentinite, the kinetics of dissolution, and the advantages and disadvantages of using acidic processing methods to achieve a particular goal are considered.

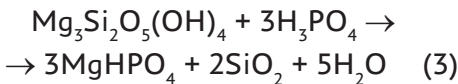
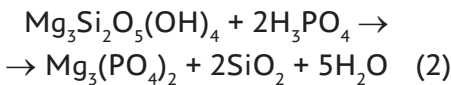
Serpentinites are mainly represented by serpentine group minerals (chrysotile, antigorite, and lizardite), with the basic formula (Mg,Fe)₃Si₂O₅(OH)₄. As is known, the main problem of acid decomposition of serpentinites is the resistance of silica when exposed to acids, which can form a protective film consisting of gels of polysilicon acids (SiO₂ · nH₂O) on the surface of particles that slow down the acid-base interaction in the serpentinite–acid system, leading to the dissolution of serpentinite in acids [10, 11].

The mechanism of decomposition of serpentinite by acids (H₂SO₄, HCl and HNO₃)

includes the ionization of magnesium and the formation of silica:



At the same time, the main parameters affecting the reaction rate are: temperature (activation of decomposition at 50–70 °C); acid concentration (an increase leads to an increase in velocity, but at high concentration levels surface passivation ($\text{SiO}_2 \cdot n\text{H}_2\text{O}$) is possible); particle size (fine powders decompose faster due to the increased contact surface). In the kinetic aspects of the dissolution of serpentine, it is noted that in the case of sulfuric acid, the reaction often follows a pseudo-first-order model, the limiting factor being diffusion and formation on the surface ($\text{SiO}_2 \cdot n\text{H}_2\text{O}$) [12]. In the case of hydrochloric acid, the reaction proceeds faster than in sulfuric acid, while the reaction is attributed to an autocatalytic kinetic model, often described by Schrauder equations [13], the limiting factor is also associated with the formation of colloidal forms of silica. Soluble magnesium nitrate is formed in nitric acid, and the kinetics of dissolution follows a diffusion-controlled model [14]. A more significant difference in the nature of dissolution from other acids occurs in the case of phosphoric acid. The reaction proceeds according to the scheme:



Unlike sulfate, chloride, and magnesium nitrate, the solubility of phosphates is low, which determines the limiting factor limiting the rate of dissolution in phosphoric acid. An analysis of the kinetic aspects of the dissolution and interaction of serpentine with acids of various kinds shows that two phases occur in an acidic

environment: first, the "bruxite" layer rapidly dissolves to form Mg^{2+} ions, then the silica framework much more slowly, which leads to the formation of a passivating $\text{SiO}_2 \cdot n\text{H}_2\text{O}$ layer. Naturally, the appearance of colloidal silica in the serpentine–acid reaction system is associated with the effect of H_3O^+ ions on the structural crystal lattice of serpentine, and its amount, capable of limiting the dissolution process, should a priori depend on its quantity. That is, it depends on the concentration of acid. Acid leaching of serpentine is: magnesium dissolution (surface reaction) \rightarrow release of H_4SiO_4 and/or formation of a Si-rich layer (aggregation of silicic acid H_4SiO_4 – primary SiO_2 particles-nanoscale \rightarrow flocculation \rightarrow gel).

The kinetics of the reaction on the surface is described by the classical equation:

$$\begin{aligned} 1 - (1 - X)^{1/3} &= k_r t, \\ k_r &= k_{r,0} e^{-E_a/RT} [\text{H}^+]^n, \end{aligned}$$

where X – is the degree of leaching Mg; k_r – reaction rate constant; $[\text{H}^+]$ – is the acid concentration; n – is the order of reactions by acid.

The kinetics of gelation from the released H_4SiO_4 can be written as:

$$\begin{aligned} dC_m/dt &= -k_r C_m^m, \\ k_r &= k_{r,0} e^{-E_a/RT} f(pH), \end{aligned}$$

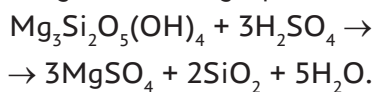
where C_m – monomer concentration [H_4SiO_4], $m \approx 2$ (effective order). The minimum polycondensation rate is about $pH \approx 2$. With a further increase in acidity, rapidly forming gel ($\text{SiO}_2 \cdot n\text{H}_2\text{O}$)/the layer puts the process into diffusion mode. Theoretically, it becomes difficult to determine the critical concentrations of gelling acid. In this regard, in this work we studied the nature of the quantitative acid-base interaction of serpentine and sulfuric acid using the possibility of IR-Fourier spectroscopy, the purpose of which was to record the appearance of silica in the system during an increase in the quantitative acid content.

Materials and methods

Chemical analysis was performed on a JSM-6490LV device (JEOL, Japan), complete with INCA Energy 350 energy dispersion microanalyzer systems. Error: ± 1 wt. % for the main components (10–100 wt. %), up to $\pm 10\%$ relative error for minor (1–10 wt. %). Infrared spectra (X-Fourier) of samples were recorded on a Shimadzu IR Prestige-21c spectrophotometer with the prefix of the disturbed total internal reflection (NPVO) Miracle from Pike Technologies." in the frequency range of 400–4000 cm^{-1} , with its own program and database of files. The ATR-IR method was used (Attenuated Total Reflection, ATR).

Serpentinite samples were provided by Kostanay Minerals JSC (Zhitikara, Republic of Kazakhstan). About 20 g of serpentinite was crushed, sieved, and a fraction of particles < 0.14 mm in size was selected. For the experiment, 10 g of a sample containing 26.6 wt. % Mg, 18.8 wt. % Si, 2.7 wt. % Fe and 0.49 wt. % Ca. The amount of magnesium was 0.11 mol, and the amount of iron was 0.005 mol.

The stoichiometrically required amount (SRA) of sulfuric acid for interaction in the serpentinite–acid system was calculated using the following equation:



Solutions containing various amounts of SRA H_2SO_4 (from 10% to 100% SRA calculated from the actual magnesium content in a 10-gram serpentinite sample) were prepared by diluting the initial solution containing 100% SRA H_2SO_4 . The treatment of serpentinite with sulfuric acid solutions in the concentration range of 10–60% SRA H_2SO_4 was carried out in a 300 ml thermostatically controlled glass reactor equipped with a propeller mixer and a sampler.

The experiments were carried out at a ratio of solid/liquid (T/W) = 1:10. A flask with a solution of sulfuric acid was preheated to a temperature of 90 °C, then 10 g of serpentinite was introduced through a sampler and a stopwatch was turned on. When serpentinite was added to the H_2SO_4 solution heated to 90 °C, the temperature rose slightly to 94 °C. The filtrate acquired a pale green-blue hue. After 5 minutes, the hot suspension was quantitatively transferred to a folded paper filter with a white ribbon. After filtration, the total volume, mass, and *pH* of the filtrate were determined.

The degree of extraction of magnesium (PMg) and other elements from 10 g of serpentinite with H_2SO_4 solutions was calculated as the ratio of the analytical amount of the element in the filtrate to its content in the initial serpentinite sample according to the formula:

$$P_{\text{Mg}} = (m \cdot \text{Mg}, \%) / 2.538,$$

where P_{Mg} – is the mass fraction of extracted magnesium; m – is the mass of the dry residue of the filtrate, g; Mg, % – is the magnesium content in the dry residue of the filtrate according to the results of chemical analysis; 2,538 – is the magnesium content in the initial 10-gram serpentinite sample (according to the analysis). The degree of Si extraction was determined similarly.

The filtrate and insoluble precipitate after preliminary preparation (drying at 100 °C) were subjected to chemical analysis and examined by IR spectroscopy.

Results and discussion

Sulfuric acid is one of the most studied reagents for the decomposition of serpentinites. The revealed advantage of its use is the high efficiency of magnesium dissolution from serpentinite, accessibility and relatively low resistance. The main disadvantage is the formation of difficult-to-

filter silica gels during processing, which generally limits the dissolution of serpentinite. At the same time, the interaction and ongoing reactions in the serpentinite – H_2SO_4 system have an acid-base character is demonstrated at the initial stage by a rapid change in the pH of the suspension [15].

Observations of the dynamics of changes in the ratio of silicon oxide to magnesium oxide (SiO_2/MgO) in acid-insoluble residues obtained by treating serpentinite with sulfuric acid (H_2SO_4) solutions of various concentrations show that the formation of silica in the system directly depends on the amount of acid reagent introduced. Experimental data indicate that the appearance of SiO_2 in the insoluble residue begins only when a certain concentration of sulfuric acid is reached, calculated based on the stoichiometric ratio necessary for the complete dissolution of the magnesium-containing components of serpentinite (in accordance with reaction 1). At the same time, there is a clear correlation between the amount of acid added

and the degree of silica release, which indicates a chemical relationship between these processes. Thus, it can be concluded that the initial stage of SiO_2 formation in the system is determined precisely by the amount of sulfuric acid used to process the mineral. This dependence confirms that the decomposition of serpentinite under the action of HSO_4 is accompanied by a sequential transition of magnesium compounds into solution, whereas silica begins to accumulate in the insoluble phase only after reaching certain concentrations of acid in the reaction medium. The formation of a passivating silica layer is a common problem that slows down the leaching process, which determines the relevance of a more detailed study of this process (Fig. 1).

During the research, the value of the activation energy (E_a) of the dissolution of serpentinite in H_2SO_4 was experimentally determined, which was 82 kJ/mol, and after the mechanical activation process 76.7 kJ/mol, which confirms the diffusion limitation of the process. The acid

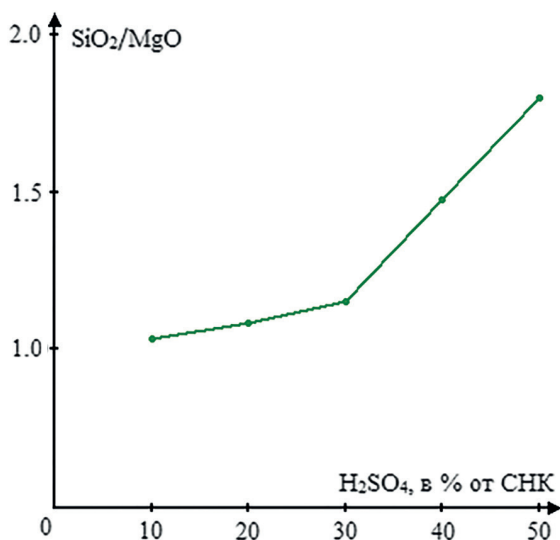


Fig. 1. The trend of SiO_2/MgO changes in acid-insoluble residues depending on the H_2SO_4 SRA in the system «serpentinite- H_2SO_4 »

Рис. 1. Тенденция изменения SiO_2/MgO в кислотонерастворимых остатках в зависимости от ЧК H_2SO_4 в системе «серпентинит – H_2SO_4 »

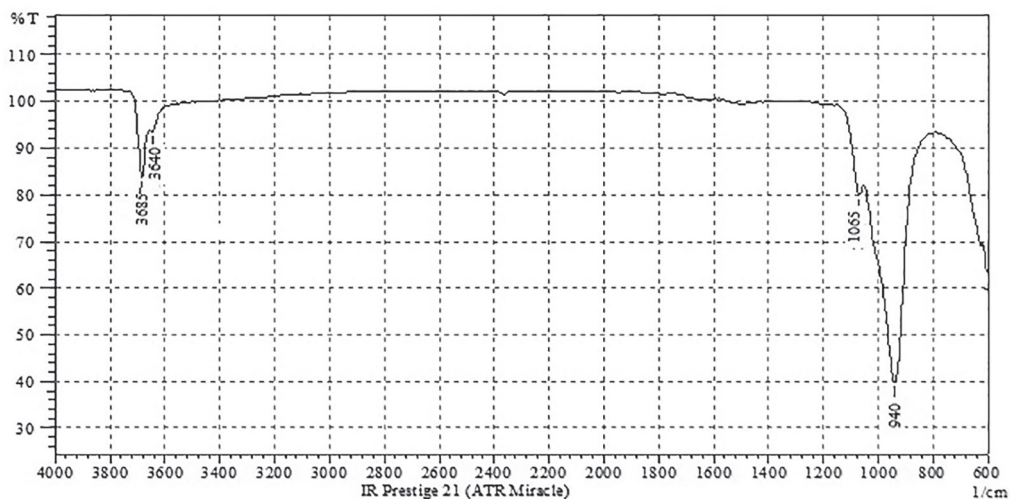


Fig. 2. The IR spectrum of the initial serpentinite [11]
 Рис. 2. ИК-спектр исходного серпентинита [11]

reaction order is $n = 0.57$ [12]. In the case of hydrochloric acid, the reaction proceeds faster than sulfuric acid [13]. In comparative experiments, H_2SO_4 , HCl , and HNO_3 (2M, 30–70 °C) activation energy values were 68, 70, and 74 kJ/mol, respectively [14]. For a more detailed study of this phenomenon, the method of IR-Fourier spectroscopy was used, since the silica, the presence of which had to be detected, was highly likely to be in an X-ray amorphous state. Acid-soluble residues obtained after each stage of serpentinite treatment with sulfuric acid (H_2SO_4) solutions with different but strictly fixed concentrations (SRA H_2SO_4) were subjected to IR Fourier spectroscopic analysis. All experiments were conducted under the same external conditions (temperature, reaction time, mixing, etc.) in order to exclude the influence of side effects on the research results. This approach made it possible to trace the dynamics of changes in the composition of the insoluble phase depending on the amount of acid introduced and reliably identify the appearance of silica even in amorphous form, which is difficult to do using other analytical methods [15].

Fig. 2 shows the IR spectrum of the initial serpentinite. In the high-frequency region, a doublet of asymmetric bands is observed at $\nu = 3680 \text{ cm}^{-1}$ and $\nu_{\text{as}} = 3643 \text{ cm}^{-1}$, which corresponds to valence vibrations of OH groups. In this case, the symmetrical valence vibration of the OH group of tetrahedral silica is recorded at $\nu_{\text{S}}\text{H} = 2930 \text{ cm}^{-1}$ [16, 17]. Spectrum analysis makes it possible to attribute the band at $\nu_{\text{ON}} = 3685 \text{ cm}^{-1}$ to intraglobular OH groups, whereas the oscillation at $\nu_{\text{ON}} = 3640 \text{ cm}^{-1}$ corresponds to the surface OH groups of the mineral structure [18, 19]. The absence of a narrow band $\nu_1\text{OH} = 3752 \text{ cm}^{-1}$ and a concomitant deformation peak at $\delta\text{OH} = 871 \text{ cm}^{-1}$ indicates that there are no free or noninteracting surface OH groups of tetrahedral silica in the serpentinite structure. A characteristic deformation oscillation $\delta(\text{OH})$ is also observed in the spectrum, which manifests itself as a wide depression in the region of 1445–1565 cm^{-1} . In this case, the signal at $\delta(\text{OH}) = 1642 \text{ cm}^{-1}$ corresponds to the deformation vibrations of the adsorbed water. In the mid-IR region, two narrow absorption bands are detected at 942 cm^{-1}

(with a shoulder at 1645 cm^{-1}), which are characteristic of SiO_4 groups of layered silicates [20]. The band at 940 cm^{-1} corresponds to valence asymmetric vibrations of $\nu_{\text{as}} = \text{O}-\text{Si}-\text{O}(\text{Mg})$ caused by the influence of an octahedral brucite-like layer on tetrahedral silica. Additionally, the shoulder at 1065 cm^{-1} is present in the spectrum, associated with asymmetric fluctuations of $\nu_{\text{as}} = \text{Si}-\text{O}-\text{Si}$ in the tetrahedral structure. Bands at 600 , 550 , and 454 cm^{-1} are observed in the low-frequency region, which, according to the literature data [15, 20], correspond to deformation vibrations $\delta(\text{SiO}_4)$ of tetrahedr.

A comparison of the IR spectra of the initial serpentinite and the acid-insoluble residue [15] obtained after treatment with a solution containing 10–20% SRA H_2SO_4 , as can be seen from Fig. 3 (Spectrum 2), does not show any significant differences in the spectra. However, on the spectrum of the sample treated with a solution containing 30–50% SRA H_2SO_4 , there is a noticeable decrease in the intensity of the peak of the surface part of the serpentinite

structure ($\nu_{\text{as}}\text{OH} = 3644\text{ cm}^{-1}$), while the intraglobular peak $\nu_{\text{as}}\text{OH} = 3685\text{ cm}^{-1}$ does not change. A noticeable change is found at the threshold of valence vibrations of $\nu_{\text{as}}(\text{Si}-\text{O}-\text{Si})$ in the region of $1125-1255\text{ cm}^{-1}$, where a wide absorption stage appears, which deepens with an increase in the concentration of SRA H_2SO_4 from 20 to 50%. According to [15], it is the appearance of wide absorptions in the region of $\nu_{\text{as}}\text{OH}$ and $\nu_{\text{as}}(\text{Si}-\text{O}-\text{Si})$ oscillations that indicates the appearance of associated various acidic groups on the surface of particles. It becomes noticeable that the silica arm $\nu_{\text{as}}(\text{Si}-\text{O}-\text{Si}) = 1063\text{ cm}^{-1}$ begins to stand out with a more pronounced peak, and the asymmetric valence vibrations of $\nu_{\text{as}}\text{Si}-\text{O}-\text{Si}(\text{Mg})$ at 942 cm^{-1} , which, according to [15, 20], refers to symmetrical valence vibrations of $\nu_{\text{as}}(\text{Si}-\text{O}-\text{Si})$ connections.

These changes confirm that as the concentration of H_2SO_4 increases, the surface OH groups gradually degrade and the silica framework transforms to form new acid-modified structures. The most distinct evidence of structural transformations in

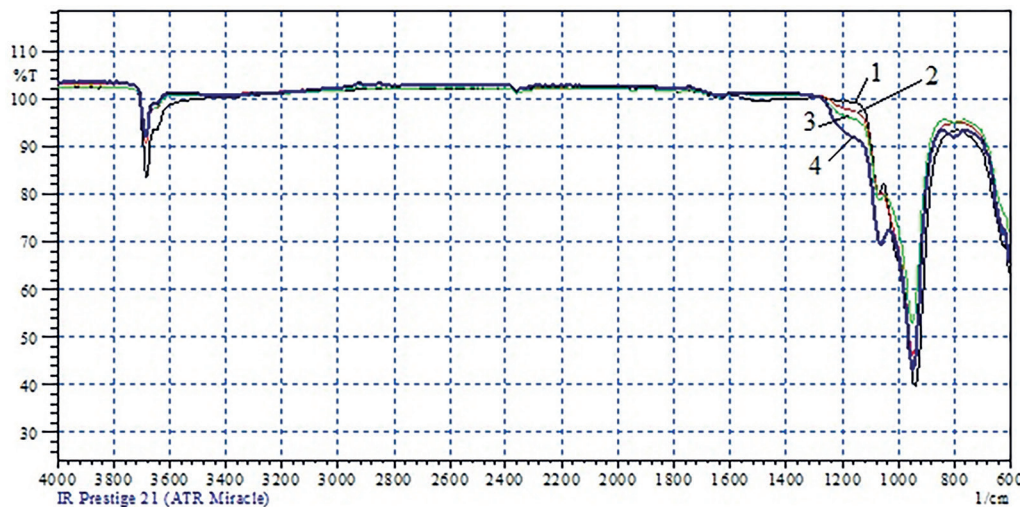


Fig. 3. IR spectra of serpentinite treated with solutions containing SRA H_2SO_4 : 1 – 10%; 2 – 20%; 3 – 30%; 4 – 50%

Рис. 3. ИК-спектры серпентинита обработанных растворами, содержащие $\text{CHK H}_2\text{SO}_4$: 1 – 10%; 2 – 20%; 3 – 30%; 4 – 50%

the serpentinite crystal lattice, namely, the transformation of silicate tetrahedral and octahedral structural elements into silica under the influence of acid-base interaction with sulfuric acid, was recorded in the IR spectra of insoluble residues after treatment of the mineral with a solution with 50% SRA H_2SO_4 (Fig. 3, spectrum 4). In this case, there is a significant increase in the intensity of the intraglobular peak at $\nu_{\text{OH}} = 3680 \text{ cm}^{-1}$, while its position in the spectrum remains unchanged. This effect indicates the preservation of intrastuctural hydroxyl groups, while their enhanced absorption may be associated with a change in the environment of these groups as a result of partial destruction of the crystal lattice. Such changes confirm that acid treatment leads to a selective modification of the serpentinite structure, with the intraglobular OH groups demonstrating greater resistance to acid attack compared with surface hydroxyls. The peak characterizing the surface part of serpentinite significantly deepens and expands in the low-frequency range of $3622 - 2845 \text{ cm}^{-1}$, recorded by a large and wide hump, which may be caused by the appearance of hydrated polysilicon acids.

The formation of new characteristic bands is observed in the IR spectra: a distinct peak at 1065 cm^{-1} ($\nu_{\text{as}}(\text{Si-O-Si})$) and absorption bands at 804 cm^{-1} corresponding to the valence symmetric vibrations of Si-O-Si bonds characteristic of silica (SiO_2) [15, 20]. The intensity of these bands increases progressively with an increase in the concentration of HSO_4 to 40–50% of the SNA, which clearly indicates the beginning of silica formation in the serpentinite- HSO_4 system. A further increase in the acidity of the medium leads to sequential polymerization of the formed silica with the formation of colloidal particles of polysilicon acids ($\text{SiO}_2 \cdot n\text{H}_2\text{O}$), which ultimately initiates the gelation process. These structural changes confirm that:

- silica formation is a step-by-step process;
- its release begins when a critical acid concentration (40–50% SNA) is reached;
- the subsequent aggregation of silica particles into colloidal forms significantly affects the kinetics of the entire process of acid decomposition of serpentinite.

The interaction of acid with the serpentinite surface begins with protonation of hydroxyl groups (OH^-), which are located in layered structures, which leads to the rupture of Si-O-Mg bonds, weakening the structure of the mineral. There is a certain range of acid concentrations to which the amount of leached Mg^{2+} has a proportional dependence on the amount of acid. Silicon (SiO_2) is released from the silicate matrix more slowly than magnesium. At low pH values, magnesium remains in solution, and silicon can form a gel. This circumstance indicates the possibility of magnesium leaching from serpentinite with a yield of no more than 40–50%, under conditions without gelation in the serpentinite – H_2SO_4 system using low-concentration sulfuric acid.

Thus, the studies have confirmed that monitoring the concentration of sulfuric acid and monitoring structural changes by IR spectroscopy can optimize the process of acid decomposition of serpentinite, minimizing the negative effects of silica.

Conclusions

In the course of the conducted research, the following conclusions and conclusions can be drawn, in particular:

- The decomposition of serpentinite by sulfuric acid proceeds by an acid-base mechanism with the ionization of magnesium and the formation of amorphous silica ($\text{SiO}_2 \cdot n\text{H}_2\text{O}$);
- The rate of dissolution depends on the acid concentration, temperature and particle dispersion, however, at high concentrations of H_2SO_4 the process slows

down due to the formation of a passivating layer of silica;

- IR-Fourier spectroscopy confirmed that silica appears in the system when 30–50% of the stoichiometrically required amount of acid (SRA H_2SO_4) is reached;

- The formation of $SiO_2 \cdot nH_2O$ gel on the surface of serpentinite particles is the main limiting factor reducing the efficiency of magnesium leaching;

- When treated with H_2SO_4 , the destruction of the surface OH groups ($\nu_{OH} = 3644 \text{ cm}^{-1}$) is observed, while the intraglobular ones ($\nu_{OH} = 3685 \text{ cm}^{-1}$) remain stable;

- The appearance of a wide absorption band in the region of $1125–1255 \text{ cm}^{-1}$ and an increase in the peak at 1065 cm^{-1} ($\nu_{Si-O-Si}$) indicate the transformation of silicon-oxygen tetrahedra and the formation of acid-modified structures;

- The maximum magnesium yield (40–50%) without gelation is achieved by using dilute sulfuric acid ($\leq 50\%$ SRA);

- The value of the activation energy of the dissolution of serpentinite was experimentally established, which was 82 kJ/mol, and after mechanical activation 76.7 kJ/mol, which confirms the diffusion limitation of the process.

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