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ПОЛУЧЕНИЕ СУЛЬФАТА МАГНИЯ ИЗ ТЕХНОГЕННОГО ОТХОДА ПЕРЕРАБОТКИ ХРИЗОТИЛОВОГО СЫРЬЯ ЖИТИКАРИНСКОГО МЕСТОРОЖДЕНИЯ

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Аннотация: На основе проведенных технологических исследований и анализа материального баланса процессов получения сульфата магния ($MgSO_4$) из серпентинита Житикаринского месторождения предложена новая принципиальная технологическая схема, включающая ряд стадий сернокислотного выщелачивания и последующей очистки сульфатного раствора с применением термоактивированного серпентинита, обработанного при температуре 750 °С. Полученные результаты исследований показали, что предложенная схема позволяет существенно повысить эффективность использования серной кислоты (H_2SO_4), достигая выхода магния по серной кислоте, близкого к 100%. При этом серная кислота является единственным расходуемым реагентом в разработанной новой технологии. Получаемый по новой технологии сульфат магния отличается высокой степенью чистоты, а выход продукта в виде сульфата магния относительно содержания магния в исходном серпентините составляет до 56%. Экспериментально полученные данные открывают новые перспективы для промышленного применения серпентинитовой руды и отходов ее переработки в качестве перспективного источника магния для производства магнийсодержащих соединений. Внедрение данной новой технологии может способствовать более рациональному использованию минеральных ресурсов и техногенных отходов, снижению экологической нагрузки за счет их переработки в качестве вторичного сырья.

Ключевые слова: сульфат магния, серпентинит, серная кислота, кислотная обработка, термоактивированный серпентинит.

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Production magnesium sulfate from man-made waste from the processing of chrysotile raw materials from the Zhitikara deposit

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Abstract: In this article, based on the conducted technological research and the analysis of the material balance of the processes for obtaining magnesium sulfate ($MgSO_4$) from serpentinite of the Zhitikara deposit, a new principal technological scheme has been proposed. The technological scheme includes a number of stages of sulfuric acid leaching and subsequent purification of the sulfate solution using thermally activated serpentinite treated at a temperature of 750 °C. The research results have shown that the proposed scheme can significantly improve the efficiency of sulfuric acid utilization (H_2SO_4), reaching a magnesium sulfuric acid yield close to 100%. In this process, sulfuric acid is the only consumable reagent in the developed technology. The magnesium sulfate obtained by the new technology is characterized by a high degree of purity, and the product yield, relative to the magnesium content in the original serpentinite, reaches up to 56%. The experimentally obtained data open up new prospects for the industrial use of serpentinite as a promising source of magnesium for the production of magnesium-containing compounds. The introduction of this new technology can contribute to the more rational use of mineral resources and technogenic wastes, as well as to the reduction of environmental impact through their processing into secondary raw materials.

Key words: magnesium sulfate, serpentinite, sulfuric acid, acid treatment, thermally activated serpentinite.

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Introduction

The Zhitikara deposit is one of the largest sources of chrysotile asbestos located in the Kostanay region, northwest Kazakhstan. Serpentinites from the Zhitikara deposit are an exceptionally valuable mineral raw material with significant potential for the industrial extraction of magnesium compounds in various chemical forms. These rocks, formed as a result of long-term geological processes, are characterized by a unique chemical composition, which makes them especially in demand in modern conditions of rapid growth in the consumption of magnesium compounds. In recent decades, there has been a steady increase in demand for magnesium derivatives in various sectors of the national economy. This need is particularly acute in the agricultural sector, where magnesium fertilizers play a key role in increasing crop yields and improving soil quality [1, 2]. Magnesium compounds are equally important in industrial production — they are widely used in metallurgy [3—5], the chemical industry [6—8], the production of building materials [9, 10] and many other technological processes. In the light of these trends, the problem of integrated and rational processing of local serpentinite resources is becoming particularly relevant. The traditional approach, which considers serpentinites only as a by-product of asbestos production, seems economically unjustified today. Modern technologies allow us to consider these

wastes as a full-fledged multicomponent raw material capable of providing a significant part of the region's needs for magnesium products. For 65 years, chrysotile raw materials have been mined and processed here, resulting in huge dumps — millions of tons of so-called serpentinite waste. Serpentinite ores of this deposit are magnesian silicate minerals rich in magnesium and silica. The main minerals of chrysotile-asbestos raw materials and the wastes of its processing are minerals of the serpentine group (chrysotile, antigorite, lizardite), which are described by the general formula $(Mg, Fe)_3Si_2O_5(OH)_4$. The more valuable components in serpentinites, from the point of view of their use in obtaining commercial products with market values, are magnesium and silicon, which are found in rather large amounts (MgO — up to 43% and SiO_2 — up to 45%, respectively). The development of serpentinite processing technologies is of particular importance in the context of:

Resource conservation — the most complete extraction of all valuable components from mineral raw materials;

Environmental safety — solving the problem of accumulated landfills and preventing their negative impact on the environment;

Economic efficiency — creation of new industries with high added value based on local raw materials.

In the current historical period, the search for acceptable ways to use low-quality raw materials and especially industrial waste is relevant for the chemical [6—8], metallurgical [3—5], construction [11—13], energy [14] industries, and especially for mining [15], as one of the most waste-generating industries. In this regard, the search for ways to involve chrysotile-containing raw materials and especially waste from the processing of chrysotile-asbestos raw materials for the production of magnesium compounds in the world is mainly aimed at finding the possibility of acidic processing methods [16, 17] using traditional known mineral acids: sulfuric, hydrochloric, nitric and phosphoric. It is obvious that the choice of mineral acid depends on the target product and technological requirements. From the analysis of literature sources, it is known that sulfuric acid remains the most economical and available reagent. Hydrochloric acid provides more complete dissolution of magnesium from serpentinite. Nitric acid has high efficiency but its use is limited due to cost. Phosphoric acid is interesting for agrochemical applications, but is limited due to the formation of insoluble phosphate. Many studies are aimed at improving development efficiency, reducing costs and minimizing environmental impacts. It should be noted that, despite the numerous cited studies, there is still no acceptable technology for processing serpentinite using acid methods. One of the main problems of acid decomposition of serpentinite is the resistance of silica, which, due to its physicochemical behavior in an acidic medium, can form a protective film on the surface of serpentinite particles, slowing down the reaction and, therefore, the dissolution of serpentinite in solutions of mineral acids [17—19]. The results of studies of physical and chemical aspects of the behavior and dissolution of serpentinite from the Zhitikara deposit are given in [20—22]. Kinetic aspects and dissolution mechanism of serpentinite in various mineral acids are described in detail in [23, 24], where it is noted that the main limiting factor is the formation of colloidal forms of silica at the interface [25, 26]. The process of acid treatment of serpentinites, especially with the use of sulfuric acid, is accompanied by characteristic chemical transformations of great technological importance. When mineral raw materials interact with acidic reagents, selective dissolution of the components is observed:

magnesium-containing compounds pass into the liquid phase with the formation of an aqueous solution of magnesium sulfate, while silicon predominantly remain in the solid phase as insoluble compounds [25, 26].

$$Mg_3Si_2O_5(OH)_4 + 3H_2SO_4 \rightarrow$$
$$\rightarrow 3MgSO_4 + 2SiO_2\downarrow + 5H_2O \quad (1)$$

This process underlies the majority of modern technological schemes for processing serpentinite raw materials. Of particular interest for industrial use are serpentinites from the Zhitikara deposit, which are characterized by an abnormally high content of magnesium oxide — from 40 to 43% by weight [27, 28]. This concentration of MgO significantly exceeds the average values for this type of mineral raw materials and creates exceptionally favorable conditions for the organization of cost-effective production of magnesium salts. It is important to note that magnesium in serpentinites is in a chemically active form, which greatly facilitates the process of its extraction compared to other magnesium-containing minerals. Magnesium sulfate as a final product of processing has a wide range of applications in various industries. In agriculture, it is used as a highly effective magnesium fertilizer that helps to increase yields and improve the quality of agricultural products.; In the medical industry, purified forms of magnesium sulfate are used in pharmaceutical production.; The food industry uses it as a regulatory additive; In the chemical industry, it serves as a raw material for the production of various magnesium-containing compounds. The development of technologies for obtaining magnesium sulfate from local serpentinite raw materials is of strategic importance for the region's economy, as it allows solving several important tasks simultaneously:

- reduce dependence on imported magnesium products;
- to increase the economic efficiency of mining waste processing;
- create new high-tech production facilities;

To solve the environmental problems associated with the accumulation of landfills [29, 30].

Based on these considerations, the purpose of this work is to develop a new technological scheme for the production of magnesium sulfate, including the use of low-concentration sulfuric acid solutions. The use of sulfuric acid results in the formation of an insignificant amount of silica in the "serpentinite — sulfuric acid" system, which slows down the dissolution of serpentinite in the acid and reduces the consumption of thermally activated serpentinite (TA-Sp) used for neutralizing the leaching solution and purifying it from impurity ions of metals.

Materials and methods

In the studies, a Pyrex flask equipped with a reflux condenser, propeller stirrer and thermostat served as a reactor. Serpentinite waste thermally activated at 750 °C for an hour was taken as a reagent for neutralization and purification of sulfate leaching solution. Thermally activated serpentinite exhibits enhanced alkaline properties acquired during calcination due to phase transformations of the brucite components of serpentinite into magnesium oxide forms.

Magnesium oxide is characterized by stronger basic properties and higher solubility in water compared to brucite, which leads to an increased rate of acid–base interactions during the neutralization of the sulfuric acid medium. Consequently, this alkaline reagent ensures the achievement of acidity up to pH = 8.3, at which practically all impurity ions such as Al^{3+}, Fe^{3+}, Fe^{2+}, Cr^{3+}, present in the initial composition of serpentinite, precipitate in the form of hydroxides.

100 g of serpentinite waste was suspended in water for 10 minutes at a liquid-to-solid (L/S) ratio of 2.1. To leach magnesium from the waste, of 115 g H_2SO_4 was gradually added over 10 – 15 minutes, resulting in an L/S ratio of 3.25. After 2 – 3 minutes, the temperature of the suspension was increased to 90 – 100 °C, and the suspension was stirred for 30 minutes, since the further rate of magnesium leaching from serpentinite is only slightly dependent on the duration. Upon completion of leaching, a suspension of thermally activated serpentinite (TA-Sp) was added until the pH of the medium reached 8.3, with the TA-Sp mass amounting to 100 g. The total consumption of serpentinite raw material [Sp + (TA-Sp)] was therefore 200 g. The suspension was cooled to 20 – 25 °C to precipitate hydroxides of impurity metals and subsequently filtered. The resulting filtrate together with the wash waters (900 mL in total) represented a magnesium sulfate solution with a concentration of 161.4 g/L ($MgSO_4$), containing an insignificant amount of calcium sulfate.

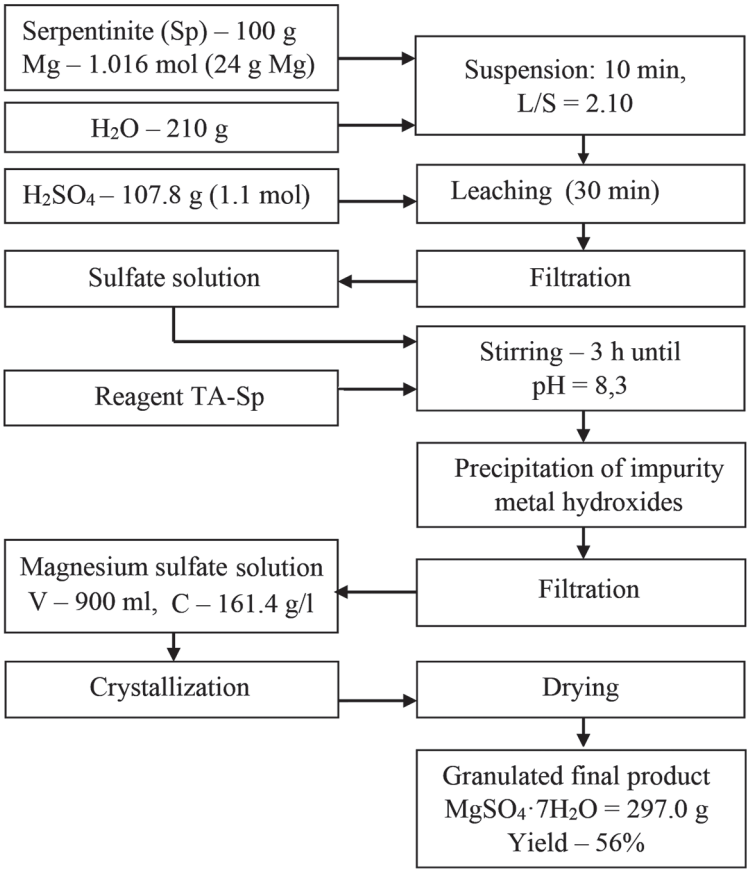
All analytical procedures were performed using the capabilities of the scanning electron microscope JSM-6490LV instrument, JEOL (Japan), complete with INCA Energy 350 energy dispersive microanalyzer systems.

Results and discussion

The nature of quantitative interaction in the "serpentinite – sulfuric acid" system, as was shown earlier in [16] shows that the amount of magnesium extracted into the sulfate solution from serpentinite in some range from the beginning to 0.35 – 0.4 SRQ (stoichiometric required quantity) of H_2SO_4 of acid concentrations has an equivalent proportionality, that is, the amount of dissolved magnesium corresponds to an equivalent of SRQ of H_2SO_4, according to the reaction equation: $Sp(Mg) + H_2SO_4 \rightarrow MgSO_4 + 2H^+$ where, Sp(Mg) is magnesium in serpentinite. At the same time, it was observed that within these concentrations the formation of colloidal forms of silica consisting of associated polysilicic acids ($SiO_2 \cdot nH_2O$) does not occur so significantly, causing the limitation of the dissolution rate of serpentinite. It was found that sulfuric acid interacts with serpentinite in the most effective way with an approximate utilization efficiency close to 100%; when using concentrations of sulfuric acid up to 0.4 SRQ of H_2SO_4 the dissolution rate of serpentinite decreases due to the passivating effect of associates formed from oligo- and polysilicic acids arising from the transition of silicon in solution in the form of silica as a result of acid–base interaction between serpentinite and the acid.

These findings formed the basis for laboratory studies aimed at establishing the optimal technological conditions and efficiency of magnesium extraction from serpentinite waste in the form of magnesium sulfate. At the same time, it was taken into account that when assessing the economic efficiency of the use of the sulfuric acid method the cost of purchasing sulfuric acid will be important in the material balance. At the same time, special attention was paid to the stages of leaching and purification of the leach solution from impurity metals. The use of thermally activated serpentinite (TA-Sp) for this purpose is explained by the fact that intra-phase transformations [17], with the formation of periclase, occur at a temperature of 750 °C, which significantly increases its alkaline properties (which can be represented schematically as [MgO + insoluble silicates]).

Based on the research results and the obtained experimental data, a scheme for obtaining magnesium sulfate has been developed and proposed (Figure), which shows the conditions, material balances and yield of the target product $MgSO_4$, including the stages of leaching, neutrali-



Scheme of magnesium sulfate production using thermally activated serpentinite (TA-Sp) for neutralization and purification of the sulfate solution

Схема получения сульфата магния с использованием термически активированного серпентинита (ТА-Sp) для нейтрализации и очистки сульфатного раствора

zation and purification of the productive solution.

The proposed scheme of magnesium sulfate production using assumes the use of the leach solution for neutralization and purification and the following raw materials as a source of magnesium: serpentinite ores, chrysotile-asbestos mining and beneficiation waste (particle size — 1.0—1.25 mm); reagent — sulfuric acid, water and as a reagent for neutralization and purification of sulfate solution from impurity metal ions — thermoactivated serpentinite waste at 750 °C (TA-Sp) (particle size — 1.0—1.25 mm).

The scheme includes two technological operations:

1. Magnesium leaching from serpentinite waste with sulfuric acid solution.

2. Neutralization of the suspension to pH=8.3 and precipitation of impurity metal ions, using for this purpose TA-Sp, by the following reaction:

- $[TA\text{-}Sp(MgO)] + 2H^+ \rightarrow$
$\rightarrow Mg^{2+} + H_2O$ + insoluble residues
- $[TA\text{-}Sp(MgO)] + H_2O +$
$+ X^{n+}(Fe^{3+}, Fe^{2+}, Ni^{2+}, Al^{3+}$ etc.) $\rightarrow$
$\rightarrow Mg^{2+}$ + metal hydroxides$\downarrow$
(pH = 8.3).

The enhanced alkaline and adsorption properties of TA-Sp are utilized both for the neutralization of acidic suspensions and for the purification of the productive sulfate solution from impurity metal ions. Based on the results of laboratory studies of the interaction between sulfuric acid and serpentinite waste, a technological scheme for the production of magnesium sulfate has been developed, involving the use of thermally activated serpentinite as a reagent for neutralization and purification (Figure).

Simple calculations of the yield of total Mg^{2+} and, consequently, magnesium sulfate ($MgSO_4$) show that the yield of $MgSO_4$, taking into account the consumption of TA-Sp is 56%, and for acid (H_2SO_4) yield close to 100%. A probable explanation of the observed fact can be formulated as follows, when serpentinite ($Mg_3Si_2O_5(OH)_4$) is used as raw material, the amount of magnesium (3 mol) interacts with 6 mol of H_2SO_4 according to the reaction equation:

$$Mg_3Si_2O_5(OH)_4 + 6H_2SO_{4(initial)} \rightarrow$$
$$\rightarrow 3Mg(HSO_4)_2 + 2H_4SiO_4 + H_2O \quad (2)$$

As a result, 3 mol of $Mg(HSO_4)_2$ and 2 mol of H_4SiO_4 are formed. During neutralization with thermally treated serpentinite (TA-Sp), the acidic salt $Mg(HSO_4)_2$ is neutralized by MgO from TA-Sp according to the reaction:

$$3Mg(HSO_4)_{2(initial)} +$$
$$+ 3MgO(TA\text{-}Sp)_{(initial)} \rightarrow$$
$$\rightarrow 6MgSO_{4(final)} + 3H_2O \quad (3)$$

A comparison of the quantities shows that Mg(initial) = 6 mol, Mg(final) = 6 mol in the products, and H_2SO_4 (initial) = 6 mol, which corresponds to the initial amount of $[H_2SO_4]$ = 6 mol. As a result, when calculating the magnesium yield relative to the total sulfuric acid content, the sulfuric acid yield approaches 100%

The table presents the analysis results of the initial serpentinite, the filtrate after acid leaching, and the $MgSO_4$ solution obtained using TA-Sp for the neutralization and purification of the acidic solution, which indicate the effectiveness of TA-Sp in improving the quality parameters of magnesium sulfate.

Taking into account the fact that at present the volume of accumulated serpentinite waste (as a source of magnesium) is measured in millions of tons, obtaining magnesium sulfate even with such a yield (56%) and a high coefficient of utiliza-

Composition of the initial serpentinite, the filtrate after acid leaching, and the purified $MgSO_4$ (using TA-Sp, wt.%)
Составы исходного серпентинита, фильтрата после кислотного выщелачивания и раствора $MgSO_4$, полученного с использованием TA-Sp, мас.%

Element	Initial serpentinite	Filtrate after acid leaching	Purified $MgSO_4$ solution (TA-Sp)
Mg	24.04	13.66	16.22
Al	0.44	0.45	–
Si	17.85	0.10	–
S	–	19.09	23.62
Ca	0.53	0.46	0.21
Cr	0.21	0.18	–
Mn	0.11	–	–
Fe	4.99	2.30	–
Cl	0.24	–	0.25

tion of sulfuric acid as the only consumable reagent can have a positive economic effect.

The results of experimental studies show that the proposed scheme for obtaining magnesium sulfate, which includes treatment of serpentinite using half the amount of sulfuric acid required, calculated by the ratio of the mole amount of magnesium in the total (Sp + TA-Sp) and thermally activated serpentinite (TA-Sp) for neutralization and purification of sulfate solution can claim to be one of the options for the use of serpentinite of the Zhitikara deposit as a source of magnesium. This allows for the evaluation of new possibilities for practical use of serpentinite rocks and chrysotile-asbestos processing waste.

Conclusions

The use of serpentinite waste as a source of magnesium in the production of magnesium salts, including magnesium sulfate, is a promising direction. The most effective method for magnesium sulfate production can be the variant of the scheme, which includes the use of serpentinite waste thermally activated at 750 °C as a reagent for neutralization and purification of leaching solutions. The scheme allows the extraction of magnesium from serpentinite waste in the form of magnesium sulfate with a total yield of up to 56%. The proposed scheme differs from others by a sufficiently high coefficient of use of the main reagent — sulfuric acid and high purity of the final product, magnesium sulfate.

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