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ҚАЗАҚСТАН РЕСПУБЛИКАСЫ  
ҰЛТТЫҚ ҒЫЛЫМ АКАДЕМИЯСЫ  
Satbayev University

# Х А Б А Р Л А Р Ы

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**ИЗВЕСТИЯ**

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК  
РЕСПУБЛИКИ КАЗАХСТАН  
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**RESEARCHING OF SULFURIC ACID LEACHING OF MAGNESIUM FROM  
SERPENTINES**

**Abstract.** The processes of magnesium extraction from waste – MPMP (multi-purpose mineral powder 0.025-0.25 mm in diameter) being the residual of technological processes on extraction of fibrous chrysotile asbestos by ventilation pumps and sulfuric acid solutions – have been studied. It is shown that the rational concentration of sulfuric acid needed to extract magnesium, where its use is close to 100%, is a solution containing 0.3 SRQ of sulfuric acid taken relative to the content of the amount of magnesium in MPMP. Apparent activation energy of MPMP dissolution reactions in a sulfuric acid solution being equal to 45.0 kJ/mol corresponding to the activation energy of diffusion-controlled processes was determined by kinetic studies and processing of obtained data. It is shown that the rate and amount of extractable magnesium from MPMP limit complex associates from silica that appear as a result of MPMP and sulfuric acid reactions at the initial stage of interactions.

**Key words:** serpentine, silica, chrysotile-asbestos waste, sulfuric acid, magnesium, and sulfuric acid extraction.

**Introduction.** Serpentine represent groups of minerals comprising a subclass of layered hydrosilicates, including structural modifications having the following chemical composition:  $Mg_3Si_2O_5(OH)_4$ . The main structural varieties of serpentine corresponding to this composition are chrysotile, antigorite and lizardite; and  $Fe^{+2}$ ,  $Fe^{+3}$ , Al, Ni, Ca, etc. are normally present as impurities. A more valuable component of serpentine in the view of chemical industry is magnesium (up to 25-26 wt.%), and silica part ( $SiO_2$  – 44-45%), because they are most demanded for production of materials for various purposes. As you know, the chrysotile asbestos mining and beneficiation cycle is accompanied with formation of a huge amount of dumps and wastes normally consisting of serpentine [1].

As a source of magnesium, scientists have been interested in these technogenic wastes for a long time. However, despite the multiplicity and diversity of the above studies, today there are no stable industrially developed technologies for their utilization in the world. Numerous proposed acidic methods and schemes have not been practically implemented. This is equally applicable to Kazakhstan.

The Kazakhstan Zhitikara serpentine deposit is located in the Kostanay region, and Kostanay Minerals JSC, a large enterprise for mining and production of various grades of chrysotile asbestos, is in the CIS. It is nationally crucial to seek and utilize accumulated technogenic serpentine wastes (about 400 million tons) being acceptable from the view of environmental viability and processability. Firstly, there are hundreds of millions of tons of such wastes, which increases the environmental hazard, and secondly, there are no process facilities in Kazakhstan for production of magnesium compounds (magnesium oxide, magnesium hydroxide or magnesium salts –  $MgSO_4$ ,  $MgCl_2$ ,  $Mg(NO_3)_2$ ) being industrially important for the development of various sectors of industry. They are used for production of steel, refractories, rubber and polymer products, as well as in construction, leather, chemical, food, pharmaceutical, oil and gas and other sectors of industry. There are no deposits of high-quality magnesian ores in Kazakhstan.

The key problem faced by researchers of acid treatment of serpentine is associated with the formation of gel-like silicic acid at interaction of  $H^+$  ions and magnesium hydrosilicate. Consequently, many of the proposed methods for processing of serpentine wastes are characterized by a resource-intensive and complex process

at all stages of the technological chain, which requires further researching of interaction of non-organic acids and serpentine in order to develop rational and acceptable methods for processing of technogenic wastes, including industrial wastes of Kostanay Minerals JSC.

According to the analysis of the literature covering development of methods for processing of serpentines using various non-organic acids (mainly sulfuric, hydrochloric, nitric acids), besides the advantages of each of the method, they also have some disadvantages preventing development of practical technologies.

Thus, [2] proposes a method of serpentine treatment with sulfuric acid solutions (20-50 wt.%) using electromagnetic separation to separate amorphous silica from chromomagnetite concentrate; the ions of iron, chromium and nickel remaining in the solution are precipitated in the form of hydroxides at pH=7-8.5. After separation of the precipitate, the productive solution is carbonized with soda ash, and the separated precipitate ( $\text{MgCO}_3$ ) is decomposed at 700°C, thus producing magnesium oxide. Sodium sulfate is obtained as a by-product. As a result, the process is time-consuming, and every technological stage involves complex processes, and the obtained silica is characterized by a limited scope of application. Two-stage leaching with sulfuric acid proposed in [3], including filtration of pulp, neutralization with ammonia, dilution of solution, introduction of magnesium carbonate nucleus and carbonation with carbon dioxide at pH=9.5-11.0 to form  $\text{MgCO}_3$ , and its subsequent drying and calcination to produce magnesium oxide, is also characterized by a time-, money- and power-consuming process.

The methods used for processing of serpentine using hydrochloric acid [4,5] and nitric acid [6] to obtain end products such as magnesium oxide, red pigment, concentrate containing nickel, chromium and iron, and amorphous silica are characterized by similar practical disadvantages.

There are also other methods for studying the decomposition of serpentine characterized by different schemes and reagents involved, for example, ammonium sulfate [7,8], which also have some disadvantages in terms of processability and economic feasibility of their use for processing of serpentine wastes to obtain a magnesium compounds and magnesium-containing materials.

Therefore, relevant researches are in progress. New approaches in the study of the processes of complex processing of serpentines, including chrysotile asbestos mining and beneficiation wastes are emerging.

Thus, the purpose of this work is to study the processes of sulfuric acid extraction of magnesium from MPMP being a dusty waste that does not require additional grinding to extract magnesium by sulfuric acid leaching to produce magnesium sulfate.

**Experimental procedure.** MPMP (multi-purpose mineral powder) waste is used in the work. This waste is generated by Kostanay Minerals JSC (Zhitikara). MPMP is a greenish-gray granular polydisperse fibrous material being the residual of technological processes of extraction of fibrous chrysotile asbestos by ventilation pumps. Grain composition: 0.025 ÷ 0.25 mm.

Average chemical composition of MPMP (wt.%): Mg - 25.80; Al - 0.40; Si - 17.64; Ca - 0.50; Cr - 0.20; Fe - 4.64; Mn - 0.03; and Ni - 0.05 (according to the chemical analysis using JSM-6490 LV complete with INCA Energy 350 energy dispersive microanalysis systems).

To study kinetic parameters of magnesium sulfate extraction from MPMP using sulfuric acid, samples of all fractions were averaged, ground using planetary ball mill Retch GmbH PM - 200 with eleven W-tungsten balls d=20 mm, at a speed of 350 rpm for 20 min. Fractions 0.074-0.104 mm in diameter were used in the studies.

MPMP was treated with solutions of sulfuric acid (1.0-5.0 M) in 1000 ml thermostatted glass reactor equipped with reverse cooler, propeller stirrer, sampler and pH meter.

For the purpose of the leaching process at a certain temperature, a portion of 500 ml 1.0M-5.0M acid was heated to the required temperature. The propeller stirrer at 350 rpm was activated when adding 10 g of a weighed sample of waste to the acid solution, and started time counting. Five milliliter samples for analysis were taken the desired time intervals depending on the process conditions (in 2, 5 and 10 minutes at the beginning, and in 20 and 30 minutes at the process end). The sample of pulp was quickly filtered using a vacuum pump and measured filtrate weight using analytical balance. The filtrate was then dried at 105°C until constant weight is achieved, and the dry filtrate weight was measured again. Chemical composition of dry samples was determined using JSM-6490LV scanning electron microscope (JEOL Ltd, Japan) complete with INCA Energy 350 energy dispersive microanalysis systems (Oxford Instruments).

The degree of magnesium extraction ( $X_{\text{Mg}}$ ) from MPMP using sulfuric acid solutions was calculated on the basis of the ratio of recovered amount of magnesium in filtrate to its amount in the initial sample of MPMP.

**Results and discussion.** Preliminary determination of the optimal grain composition of MPMP for the purpose of sulfuric acid extraction of magnesium showed that the degree of magnesium extraction reaches

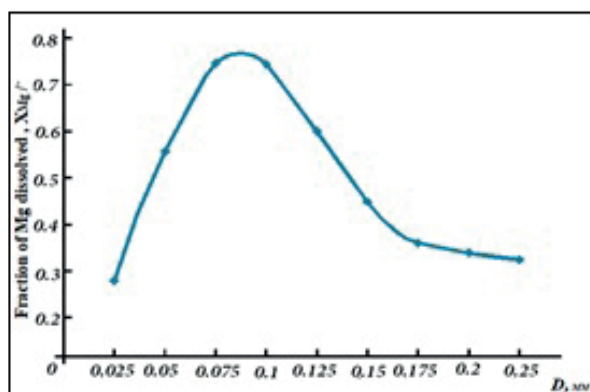
its peak value (Figure 1); the most optimum grain composition is 0.104-0.074 mm. Taking this into account, all further studies of the processes of magnesium extraction from MPMP with sulfuric acid solutions were carried out using MPMP samples with a grain fraction in this range.

Based on the results of the chemical analysis of MPMP (Mg - 25.8 wt.% and Si - 17.64 wt.%), it was assumed that this waste (MPMP) consists mainly of layered magnesium hydrosilicate – varieties of the serpentine subclass, with the general formula  $Mg_3Si_2O_5(OH)_4$ , and the MPMP reaction using sulfuric acid can be described by the following equation:

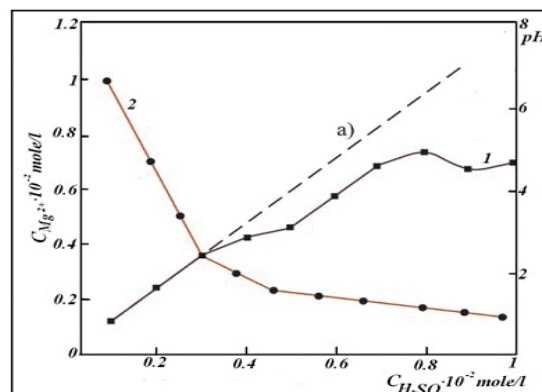


According to the processes of quantitative interaction of MPMP (molar content of magnesium in a MPMP sample) and sulfuric acid (calculated stoichiometric quantity of  $H_2SO_4$ ) according to equation (1) shows that the theoretical proportional dependence, i.e. the amount of extractable magnesium on the stoichiometrically required quantity (SRQ) of  $H_2SO_4$  (Figure 1, (a) dotted line) can only be maintained up to concentrations of 0.3 SRQ  $H_2SO_4$ . In this case, as one can see from Figure 1, an equivalent amount (0.3 SRQ  $Mg^{+2}$ ) of magnesium ions passes from the MPMP into the solution. Further, when using the concentration (0.4-0.5) SRQ  $H_2SO_4$  for extraction, this regularity breaks and decreases the equivalent extraction of magnesium into solution. Starting from 0.6 SRQ  $H_2SO_4$  and up to the proportions taken in the equivalent molar ratio of magnesium in MPMP and  $H_2SO_4$  (1:1) according to equation (1), the amount of extractable magnesium is always lower than the theoretically calculated one on the amount (SRQ)  $H_2SO_4$  in solution prepared for extraction of magnesium from MPMP. In case of 1:1 ratio, the degree of extraction or the amount of extractable magnesium in the solution does not exceed 76% of its total amount in MPMP.

The processes of interaction of MPMP and  $H_2SO_4$  at different ratios of MPMP (Mg): $H_2SO_4$  (SRQ) differ not only in the amount of dissolved magnesium, but also in the kinetics of the reactions occurring on the surface of MPMP.



**Figure 1** Dependence of the degree of Mg extraction on grain composition of MPMP. Concentration  $C_{H_2SO_4}=2.0M$ ; 1-MPMP waste, ( $\tau = 120$  min,  $t = 60^\circ C$ ,  $S:L = 1:10$ )



**Figure 2** Dependence of extractable amount of magnesium from MPMP (1) and pH factor (2) on amount of  $H_2SO_4$  in solution,  $m$  (MPMP) = 10 g,  $\tau = 10$  min,  $t = 96^\circ C$

Kinetic studies of the interaction of MPMP and aqueous solutions of  $H_2SO_4$  in quantitative ratios, SRQ  $H_2SO_4$  (mol) in relation to the molar amount of magnesium in a sample of MPMP (Mg): $H_2SO_4$  (SRQ) in the range from  $1.0:0.1$  to  $1.0:1.0$  shows that the nature of the dependence of the amount of recovered magnesium on the amount of  $H_2SO_4$  in the recovery solution correlates quite well with the features of the layered structural and molecular serpentine. As it is known [9], the structure of serpentine consists of folded kaolin-like layers, each layer of which consists of alternately located interconnected tetrahedral silicon-oxygen and octohedral brucite-like layers. The two-layer packets that make up the structure are characterized by disproportionality of trioctahedral “brucite” layers with hexagonal patterns of silicon-oxygen tetrahedrons, which leads to an uneven distribution of magnesium ions in individual packets of layered hydrosilicate.



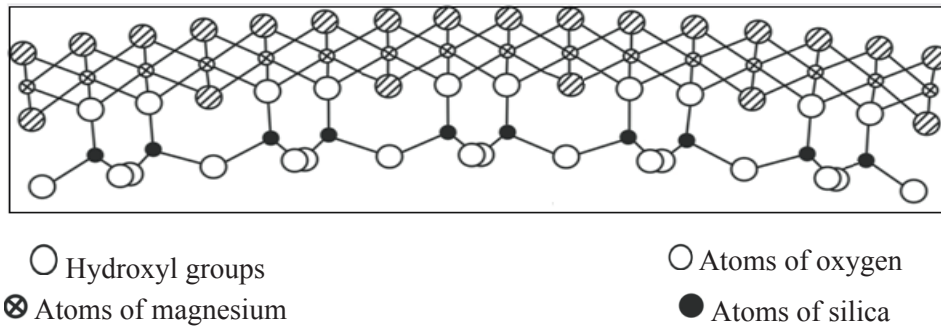


Figure 3. Ideal structure of crystalline chrysotile asbestos pattern [9]

Estimation of distribution of magnesium ions in serpentine structure (chrysotile) [9] shows that 1/3 portion of magnesium in its molecular structure of the total amount in chrysotile is in the octahedral “brucite” layer, while the other 2/3 is in the tetrahedral layer. Based on this, the chemical formula of serpentine (chrysotile) can be more clearly described as  $Mg(OH)_2(MgOH)_2Si_2O_5$ . It is assumed that that 1/3 portion of magnesium in the form of  $Mg(OH)_2$ , which is in the “brucite” octahedral layer, is more easily accessible to the effect of  $H^+$  acid ions, and 2/3 portion in the form of  $(MgOH)_2$ , which is in tetrahedral form, is relatively structurally difficult to extract. Thus, extraction of magnesium into MPMP solution can be described by the following reactions (2,3) having different kinetic character.



As one can see from Figure 2, in case of interaction of serpentine (chrysotile) and  $H_2SO_4$ , the equivalent extraction of magnesium from the equivalent amount of acid is only observed up to 0.3 SRQ  $H_2SO_4$ . This acid-base interaction proceeds relatively quickly, which can be seen from Figures 4 and 5 showing the influence of  $H_2SO_4$  concentration and process temperature on degree of magnesium dissolution from MPMP. It should be noted that in the both cases the portion of recovered magnesium in  $H_2SO_4$  solution increases only up to a certain level, in our case up to 0.76 of magnesium portion of its original weight in MPMP.

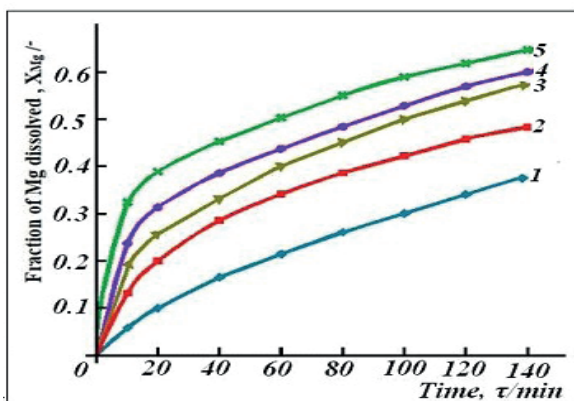


Figure 4. Influence of  $H_2SO_4$  concentration on the degree of Mg ion extraction from MPMP  
 $C_{H_2SO_4}$ : 1-1,0M; 2-2,0M; 3-3,0M; 4-4,0M; 5-5,0M,  $t^{\circ}$  - 40°C.

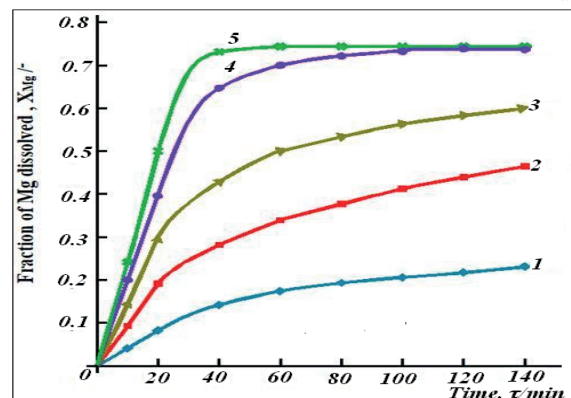


Figure 5. Influence of temperature on the degree of Mg ion extraction from MPMP at  $t^{\circ}C$  1-20; 2-40; 3-60; 4-80; 5-90,  $C_{H_2SO_4} = 2,0M$

Influence of temperature on the degree of magnesium extraction affects the time to reach the maximum fraction of magnesium extraction from MPMP, so at 90°C,  $X_{Mg} = 0.76$  is achieved in 45 minutes, and at 80°C –it is achieved in 120 minutes. The shown times to reach the maximum recovery rate are obviously explained by diffusion barriers preventing the movement of  $-H_3O^+$  ions to new reaction zones through

interlayer channels of modified initial structure of the surface layers of serpentine as a result of the action of sulfuric acid solution on them. Yander equation is normally used to describe kinetic regularities of such hydrochemical processes[10], where the fraction of dissolved magnesium (in our case) from MPMP on time of interaction with acid is related by the following equation:

$$[1 - (1 - X_{Mg})^{1/3}]^2 = K_{eff} \cdot \tau \quad (4)$$

where,  $X_{Mg}$  – portion of dissolved magnesium;  $\tau$  – interaction time (min);  $K_{\phi\phi}$  – effective or apparent speed constant ( $\text{min}^{-1}$ ).

If the process of interaction of MPMP and sulfuric acid solution proceeds according to the laws described by Yander's equation, then the graph of the dependence of  $[1 - (1 - X_{Mg})^{1/3}]^2$  on  $\tau$  should be linear, where  $\text{tg}\alpha$  is the slope of the straight lines corresponds to  $K_{eff}$ .

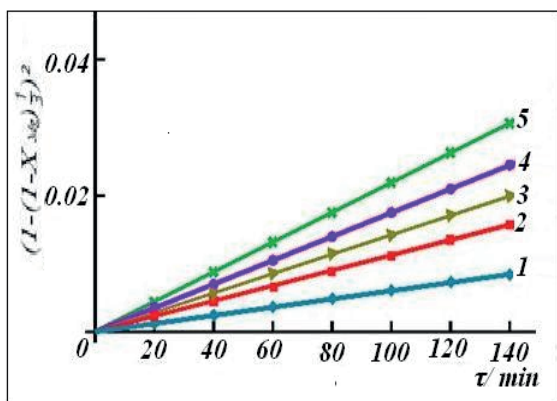


Figure 6. Dependency graph  $(1 - (1 - X_{Mg})^{1/3})^2$  on  $\tau$  at various concentrations of acid on the results of Figure 4 according to equation 4.

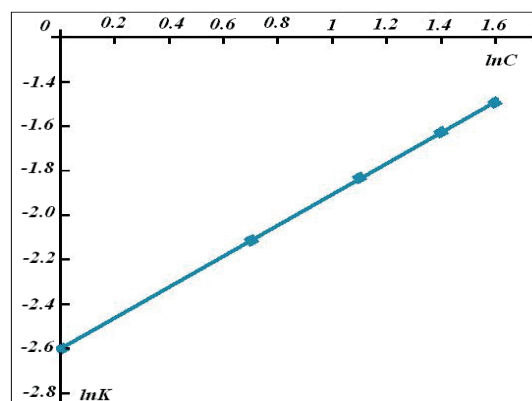


Figure 7. Dependence of Mg dissolution apparent rate constant on  $\text{H}_2\text{SO}_4$ .

The corresponding graph (Figure 6) was built by processing the curves shown in Figure 4, on the basis of which the  $K_{eff}$  values were determined. A graph of  $\ln K_{eff}$  versus  $\ln C_{\text{H}_2\text{SO}_4}$  dependence was plotted, where  $\text{tg}\alpha$  is the slope of the straight lines (Figure 7), the reaction order was  $n=0.71$ , to determine the order of the reaction on sulfuric acid.

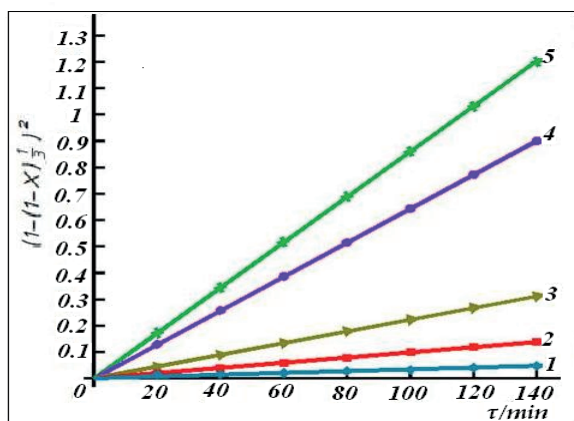


Figure 8. Graph of  $(1 - (1 - X_{Mg})^{1/3})^2$  dependence on  $\tau$  on the results of Fig. 5, equation 4.

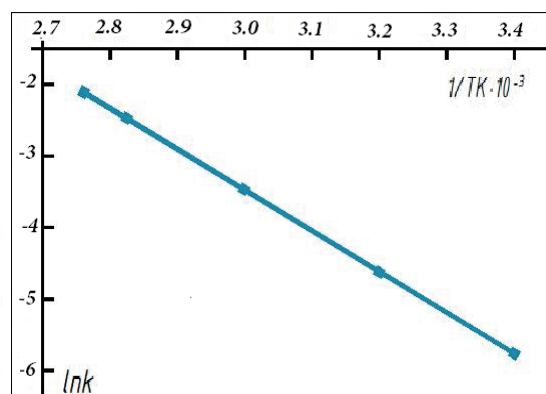


Figure 9. Graph of  $\ln K$  dependence on  $T^{-1}K^{-1}$  according to Arrhenius equation,  $E_{app} = 45$  kJ/mole

A graph of dependence of  $[1 - (1 - X_{Mg})^{1/3}]^2$  on  $\tau$  at different temperatures (Figure 8) was plotted by initial processing of experimental data shown in Figure 4 to find the apparent activation energy of the reactions of interaction of MPMP and sulfuric acid; the values of  $\ln k$  were determined on the basis of  $\text{tg}\alpha$  – on angle of

slope; then the apparent activation energy of the interaction reaction process was determined on the basis of the dependence of  $\ln k$  on  $1/T$  (Figure 9) being equal to 45 kJ/mol, which corresponds to the activation energy of diffusion-controlled processes. Diffusion-controlled can be, as noted earlier, the diffusion of  $H_3O^+$  ions through thin interlayer channels. An additional obstacle can also be created by the emerging new aggregate formations enriched with silica as a result of acid-base interaction of MPMP and  $H_2SO_4$  and removal of magnesium from the octahedral brucite layer. These aggregate formations, in an acidic environment, will obviously have the properties of silicic acids, which do not dissolve in a sulfuric acid solution, but are actively protonized absorbing a certain equivalent amount of  $H^+$  ions. Acidic environment promotes the formation of cationic hydroxyls ( $Si-O-H^+$ ) [11], especially in an acidic medium at  $pH=1-4$ , which easily form associates using H-bonds. Depending on the rate and amount of emergence of new aggregate formations, two-, three- and more complex associates (n-layer oligomers) can be formed from silica that can more tightly block the channels of penetration into the structure of serpentine preventing the diffusion of  $H_3O^+$  ions. This assumption is also supported by the fact that even when using high concentrations of sulfuric acid (5M) and temperature ( $90^\circ C$ ), the ceiling value of the fraction of maximum magnesium recovery ( $X_{Mg}=0.76$ ) remains unchanged with these parameters of the process of MPMP dissolution in sulfuric acid solutions.

**Conclusions.** By studying the process of magnesium extraction from MPMP using sulfuric acid solutions it was found that the maximum consumption of sulfuric acid (close to 100%) is achieved at concentration of  $H_2SO_4$  in the solution used in the amount of 0.3 SRQ relative to the amount of magnesium in the MPMP. It is assumed that this is explained by location of partially extractable magnesium ions in brucite-like layers of layered magnesium hydrosilicates, the total amount of which in these layers is 1/3 of the total magnesium content in the molecular structure of serpentine ( $Mg_3Si_2O_5(OH)_4$ ). At higher concentrations of  $H_2SO_4$ , the yield of magnesium sulfate and the rate of dissolution decrease due to complex associates from silica arising from acid-base interactions between MPMP and sulfuric acid solution. The research results can be useful in the development of acid methods for magnesium extraction from serpentinite wastes of chrysotile-asbestos beneficiation, especially when assessing the economic feasibility of the sulfuric acid method, since according to the material balance, it is determined, first of all, by the cost of the reagent – sulfuric acid.

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### СЕРПЕНТИНДЕРДІ КҮКІРТ ҚЫШҚЫЛЫМЕН ӨНДЕУ АРҚЫЛЫ МАГНИЙДІ АЛУ ПРОЦЕСТЕРІН ЗЕРТТЕУ

**Аннотация.** Талшықты хризотил-асбестті желдеткіштік сорғыштармен алудың технологиялық процестерінен кейін қалатын қалдық – ӘМҰ-тан (эмбебап минералды ұнтақ 0,025-0,25 мм) күкірт қышқылының ерітінділерімен магнийді алу процестері зерттелді. Магнийді алу үшін күкірт қышқылының ұтымды концентрациясы, яғни оны қолдану 100%-ға жақындайтын кезі, ӘМҰ құрамындағы магний мөлшеріне қатысты алынған стехиометриялық қажетті мөлшері 0,3 болатын күкірт қышқылының ерітіндісі екендігі көрсетілген. Кинетикалық зерттеулер және алынған деректерді өңдеу арқылы диффузиялық бақыланатын процестердің активтену энергиясына сәйкес келетін 45,0 кДж/моль тең, ӘМҰ-дың күкірт қышқылының ерітіндісіндегі еру реакцияларының айқын активтендіру энергиясы анықталды. ӘМҰ-дан магнийді алудың жылдамдығы мен мөлшерін ӘМҰ және күкірт қышқылының реакциясының бастапқы өзара әрекеттесулерінің кезеңінің нәтижесінде пайда болатын кремнеземнің күрделі ассоциаттары шектейтінін көрсетілді.

**Түйінді сөздер:** серпентин, кремний, хризотил-асбест қалдықтары, күкірт қышқылы, магний, күкірт қышқылымен өңдеу.

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## ИССЛЕДОВАНИЕ ПРОЦЕССОВ СЕРНОКИСЛОТНОГО ИЗВЛЕЧЕНИЯ МАГНИЯ ИЗ СЕРПЕНТИНОВ

**Аннотация.** Исследованы процессы извлечения магния из отхода – ПМУ (порошок минеральный универсальный 0,025-0,25 мм), который остается после технологических процессов извлечения волокнистого хризотил-асбеста вентиляционными насосами, растворами серной кислоты. Показано, что рациональной концентрацией серной кислоты для извлечения магния, где ее использование приближается к 100%, является раствор, содержащий 0,3 СНК серной кислоты, взятой относительно к содержанию количества магния в составе ПМУ. Кинетическими исследованиями и обработкой полученных данных определена кажущаяся энергия активации реакций растворения ПМУ в растворе серной кислоты, равным 45,0кДж/моль, что соответствует энергии активаций диффузионно-контролируемых процессов. Установлено, что скорость и количество извлекаемого магния из ПМУ ограничивают сложные ассоциаты из кремнезема, появляющиеся в результате реакций ПМУ и серной кислоты на начальном этапе взаимодействий.

**Ключевые слова:** серпентин, кремнезем, отход хризотил-асбеста, серная кислота, магний, сернокислотное извлечение.

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