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Decomposition of Magnesite-Sparing Waste in Sulfuric Acid with a High Concentration: Empirical Modeling and Determination of Optimal Conditions

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ABSTRACT
This article describes the processes of decomposition of magnesite waste in sulfuric acid, which is formed during flotation enrichment of the Zinelbulak talc-magnesite deposit. As a result of the research, the influence of concentration and temperature of the reaction medium on the dissolution of magnesite in H_2SO_4 solution was investigated and melting degrees were determined. Based on the data obtained, a mathematical model of the process was formed: the melting kinetics were described by equations, and the parameters were calculated using regression analysis. The modelling results were based on the rate at which magnesite melts, with a focus on acid concentration and temperature factors. The value of the coefficient of determination of the constructed mathematical model justified the fact that this model was 97.2% accurate. Based on the reliability of the mathematical model, optimal conditions are determined through experimental and model analysis. The optimum conditions are a temperature of T = 81.45 °C and an acid concentration of C = 74.05%, resulting in a decomposition rate of Y_{max} = 93.57%. The results of this work establish a scientific foundation for treating waste products containing magnesium and producing products such as magnesium sulfate from them. Initially, the article describes the composition of raw materials and experimental methods, then the mathematical model and the results obtained are analysed, and conclusions and proposals are presented.

Keywords: Zinelbulak mine, talc-magnesite waste, magnesite, dissolution in sulfuric acid, mathematical model, kinetic analysis, regression analysis.

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Introduction

The Zinelbulak talc-magnesite deposit reserve is the only major talc rock deposit in Central Asia with an output of 200 million tonnes. Mining ore has a complex composition containing 52% talc, 43% carbonate minerals (mainly magnesite) and 5% iron oxides [[1], [2], [3]]. Qualitative and quantitative analyses showed that the main mineralogical composition of ore consists of talc and magnesium components, and the magnesium content is 31.7 wt%. It has also been found that such impurities as serpentine, quartz, hematite, and magnetite are found in this raw material [[1], [2]].

When talc concentrate is isolated from the talc-magnesite raw material by gravitational or flotation methods, a magnesite waste consisting mainly of magnesite is formed. This magnifying waste can be chemically processed into a valuable product. In particular, when the magnesium is decomposed in sulfuric acid, magnesium sulfate ($MgSO_4$) is formed, a compound that is widely used in industry and agriculture. Therefore, obtaining a variety of products through the recycling of magnetic waste can provide additional economic benefits as well as reduce environmental problems.

Globally, it has been reported that by dissolving magnesite waste in hydrochloric acid, the process kinetics obeys the "pseudo-secondary" model and the activation energy is 62.4 kJ/mol [4]. However, there are also several general studies on the melting kinetics of magnesite feedstock in acids and the optimization of process parameters. Specifically, [5] in their study, they set the reaction duration to 60 min with a solid-liquid part ratio of 1:20 at an acid concentration of 2 M at a temperature of 65°C, as optimal conditions for dissolving magnesite in H_2SO_4

solution. The melting kinetics of magnesite in sulfuric acid were studied by the process of magnesium extraction in sulfuric acid on the example of the mineral serpentinite. As a result of the process, a magnesium release of 98.35% was achieved. As a result, the authors found that high-purity $Mg(OH)_2$ and $4MgCO_3$· Synthesis of $Mg(OH)_2 \cdot 4H_2O$ obtained [6]. The influence of indicators such as acid concentration, temperature, mixing rate, and particle size as factors influencing the efficiency of the magnetite acid refining process was investigated, and conclusions about optimal conditions were drawn [[7], [8], [9]].

According to the above-mentioned sources of literature analysis, the process of decomposition of magnesite waste formed by flotation enrichment of talc-magnesite from the Zinelbulak mine using sulfate, nitrate or hydrochloric acids and its mathematical model have not been studied. In this regard, this study investigated experimentally and theoretically the process of disintegration of magnite-sparing wastes by processing in H_2SO_4, the main component of which is magnesite.

The purpose of the research is to define its parameters and equations by mathematical modeling of a process and determine the optimal technological conditions for the complete process.

Experimental part

The object of the study was the magnesite-containing waste generated during the flotation beneficiation process of the Zinelbulak talc-magnesite rock. The material composition of the Zinelbulak talc-magnesite rock has been studied in our previous research [2] using modern physico-chemical methods. The magnesite-containing waste sample formed during the flotation beneficiation of this raw material was comprehensively analyzed using an X-ray fluorescence spectrometer (Figure 1).

Based on the analyses, the chemical composition of the raw material is presented in Table 1.

The phase composition of this raw material was analyzed using X-ray diffraction, and the results were processed using the BGMN/Profex Rietveld software package to determine the qualitative and quantitative mineralogical composition of the samples (Figure 2 and Table 2) [[10], [11], [12]].

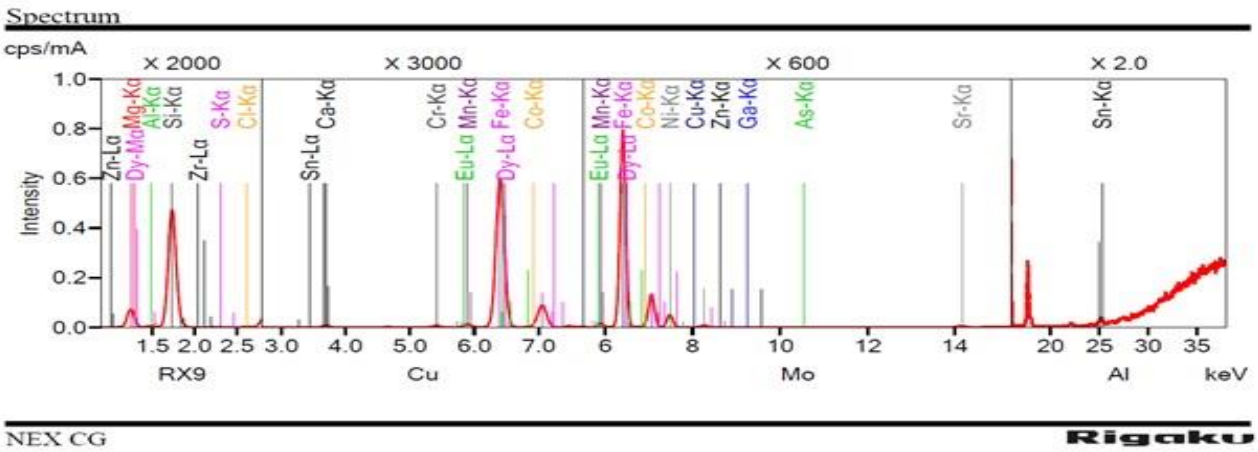

Figure 1 - Extended analysis of the magnesite-containing waste using an X-ray fluorescence spectrometer

Table 1 - Chemical composition of the magnesite precipitate obtained from the flotation process as determined by X-ray fluorescence spectrometry

Tarkibdagi oksidlarning ulushi, %										
SiO_2	MgO	Fe_2O_3	$A1_2O_3$	CaO	Cr_2O_3	MnO	SO_3	NiO	ZrO_2	N_2O
39.15	39.46	5.93	2.42	2.78	0.14	1.62	1.34	1.56	1.78	3.82

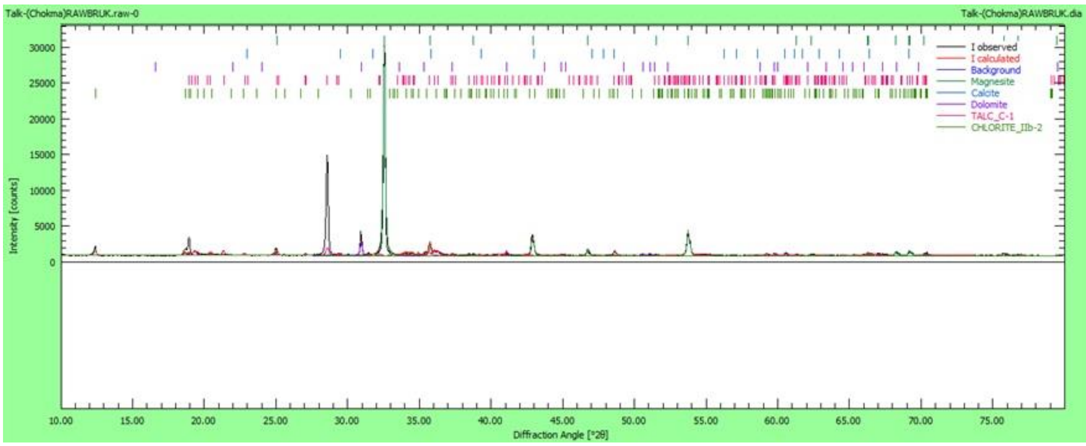

Figure 2 - X-ray diffraction pattern of the magnesite-containing waste

Table 2 - Mineralogical composition of the magnesite-containing waste sample

Mineral name	Chemical formula	Content in the waste, %
Magnesite	$MgCO_3$	53.70
Talc	$3MgO{\cdot}4SiO_2{\cdot}H_2O$	27.20
Kemmererite	$5MgO{\cdot}5FeO{\cdot}Al_2(SiO_3)_3{\cdot}H_2O$	10.01
Dolomite	$MgCO_3{\cdot}CaCO_3$	7.75
Calcite	$CaCO_3$	1.34
Total		**100**

For conducting the research, a static chemical solvent (liquid–solid leaching) method was used in the laboratory. Dried to a constant mass were weighed at an extraction of 5g from a sample with a moderate particle size to −100µm. Disintegration processes were carried out with a solid-to-liquid ratio of 5:100 (g/ml) with the sample measured in a glass tube with a volume of 100 or 250 ml of sulfuric acid solution. For preparation of the acid solution, chemically clean high concentration H_2SO_4 was used, and concentrations of 20–96% were prepared by dilution.

Theoretically, in the process of the decomposition of magnitous boar with sulfuric acid, the following reactions have been observed:

$MgCO_3$ + H2SO4=$MgSO_4$ + H_2O + CO_2

$CaMg(CO_3)_2 + 2H_2SO_4 + (n-2)H_2O = CaSO_4{\cdot}nH_2O + MgSO_4 + 2CO_2$

$CaCO_3+H_2SO_4=CaSO_4+H_20+CO_2$

$2Mg(OH)_2{\cdot}MgSiO_3{\cdot}3SiO_2+2H_2SO_4=2MgSO_4 + 4H_2O+ MgSiO_3+3SiO_2$

$Mg(OH)_2{\cdot}4MgFeO_2{\cdot}FeO{\cdot}Al_2(SiO_3)_3+10H_2SO_4=5 MgSO_4+5FeSO_4+Al_2(SiO_3)_3+11H_2O$

The amount of magnesium contained in talc-magnesite raw materials and separated magnesite porridges was determined according to the requirements of State standart 19728.8-2001, and the amount of calcium oxide was determined according to the requirements of State standart 19728.7-2001 using volumetric complexometric methods of titration with 0.05. normal Trilon-B solution, chromium black-blue and fluorescein indicators were used. The results were calculated using the following calculation formulas (1 and 2) [[13], [14]].

$$X_{CaO} = \frac{VCV_1 100}{V_2 m} \quad (1)$$

Here: V- Volume flow rate of trilon-B, $см^3$; *C*- concentration of trilon-B, g/$см^3$; V_1- total volume of solution, $см^3$; V_2- volume of solution taken for titration, $см^3$; *m*- amount of sample, g.

$$X_{MgO} = \frac{VCV_1}{V_2 m} \times 100 - 0{,}719X_1 \quad (2)$$

Here: *V*- volumetric flow rate of Trilon-B used for titration of calcium and magnesium content in the control solution, $см^3$; *C*- concentration of Trilon-B, g/$см^3$; V_1- total volume of solution, $см^3$; V_2- volume of solution taken for titration, см^3; *m*- amount of sample, g; X_1- mass fraction of calcium oxide, %.

During the analytical processes, a solution is prepared according to the method specified in the literature [15] for determining the general form of P_2O_5 in phosphate raw materials and magnesium phosphate fertilizers. The filtered sample was measured in a photo colourimeter at a wavelength of λ=440 and the results were calculated using the following formula 3:

$$C_{P_2O_5} = \frac{a \times 250 \times 100}{g \times V \times 1000} \qquad (3)$$

Here: *a*-the amount of P_2O_5 found from the calibration graph, mg; g - the amount of the analyzed sample, g; V-solution volume, см^3

Calcium and magnesium oxides contained in phosphate raw materials and magnesium phosphate fertilizers were determined by titration with a Trilon-B solution using the complexometric method presented in the literature [[16], [17]]. To determine the general form of P_2O_5 for analysis, a fixed amount of 250 cm^3 of a 0.05 N solution of trilon-B was taken from the prepared solution and analyzed in the presence of chromium black-blue. The results were calculated using the following formulas (4 and 5).

$$C_{CaO} = \frac{a \times 0.0014 \times 50 \times 100}{g \times V \times 50}; \qquad (4)$$

$$C_{MgO} = \frac{(b-a) \times 0.001 \times 250 \times 250 \times 100}{g \times V \times 50}; \qquad (5)$$

Here: α-volume of Trilon-B used to determine the amount of calcium, ml; β-the volume of Trilon-B used to determine the amount of magnesium, ml; g - amount of analyzed sample, g; V-Volume of solution, cm^3.

In order to determine the optimal conditions for the sulfuric acid decomposition process of this magnifying waste with a complex composition, a quadratic regression model with dependence on the kkita independent variable C-acid concentration (%) and T - process temperature (°C) was used to predict.

In the creation of a mathematical model, the mathematical model equation is expressed only from a mathematical point of view, depending on their quadratic, i.e. curved effect on the degree of decay when the basic initial values, i.e. temperature and concentration are 0, when the acid concentration and process temperatures increase by one unit value, depending on their quadratic, i.e. curved effect:

$$Y = a + bC + cT + dC^2 + eCT + fT^2$$

Where *a, b, c, d, e, f are the coefficients determined on the basis of experimental data.*

To predict the maximum extent of the disintegration process, the two main independent variables are the concentration of acidity (%) and T (°C) by solving all the maximum values and conditions of the process temperature.

$$\frac{\partial Y}{\partial C} = 0; \ \frac{\partial Y}{\partial T} = 0$$

The model determines the coefficient of determination depending on the total $-T_{dis}$ and residual $-R_{des}$ dispersions, i.e

$$R^2 = 1 - \frac{T_{dis}}{R_{des}}$$

The total and residual dispersions in the formula are determined depending on the actual observed value as well as the values predicted by the model. In this case, the general dispersion $T_{dis} = \sum_{i=1}^{n} (y_i - \bar{y})^2$ and residual dispersion $R_{dis} = \sum_{i=1}^{n} (y_i - \hat{y})^2$ are determined from the dispersion formulas [[18], [19]].

Results and Discussion

The prepared reaction mixture was carried out in a digital tube heater with magnetic stirring brand MS7-H550-Pro for 180 min at a temperature interval of 10°C, temperature range 30-100°C (with thermostat control ±1 °C accuracy) and constant stirring at 300 rpm. The resulting solid residue was filtered, separated, washed and dried. The amount of magnesium in the samples was determined by the complexometric method (using EDTA).

The results obtained during the studies are shown in Table 3 below, which shows that the degree of decomposition of the magnetite ash relative to MgO is due to a variation of 20–96% concentration of kp acid (C%) and temperature of 30–100°C (T).

Table 3 - Degrees of decomposition of magnesite precipitate at different concentrations, absolute values, and temperatures of H_2SO_4

Acid concentration, %	Degree of brocade of magnesite chips, %, $k_P = \frac{MgO_{сув.}}{MgO_{ум.}}$;							
	30°C	40°C	50°C	60°C	70°C	80°C	90°C	100°C
20	39.33	50.32	51.02	51.03	53.12	54.48	55.12	55.32
30	48.21	74.64	71.24	71.65	72.15	73.52	74.15	74.32
40	49.48	76.64	74.14	74.03	77.97	78.01	78.07	83.89
50	50.38	79.04	79.21	79.46	80.06	80.26	83.94	84.56
60	54.78	79.86	79.64	82.04	82.76	82.02	85.36	85.65
70	57.56	82.01	82.95	83.02	84.56	85.01	87.46	87.92
80	53.35	83.02	83.76	84.16	85.02	85.21	88.56	88.72
90	58.82	83.89	84.50	85.02	86.01	87.01	88.69	89.08
96	59.01	84.40	85.12	85.56	87.08	87.92	89.02	90.14

When the acid concentration was 20% in the process, the rate of decomposition increased by 1.40 times compared to MgO water (k_P), reaching from 39.33% to 55.32%. When the concentration was 30%, the decomposition rate was observed to increase from 48.21% to 65.66% to 1.54 times, and these dependencies increased to 1.69 and 1.68 times, respectively, at concentrations of 40% and 50% of sulfuric acid. It was found that in processes where the acid concentration was increased from 60% to 96%, these values increased by 1.56 and 1.53 times, respectively. At all concentrations of acid, an increase in decomposition at the temperature range of 30-40°C was observed by 1.23 and 1.43 times, and at the temperature range of 50-100°C, up to 1.08 times. Based on the research carried out, it was found that the process is desirable to conduct the process at high concentrations of acid, 80-96%, at a temperature of 40°C.

In the studies, the maximum value was determined at the boundary points, having the concentration of acid (C) in the range of 20% ≤ C ≤ 96% and the process temperature ranges (T) between 30°C ≤ T ≤ 100°C.

$$Y = a + bC + cT + dC^2 + eCT + fT^2$$

The coefficients *a, b, c, d, e, and f in the mathematical model* were calculated using the method of least squares and were determined to be equal to *a = -27.21; b = 0.6257; c = 2.4094; d = -0.0023; e = -0.0035* and *f = -0.0132*. Based on these coefficients, the following equation was formed.

$$Y = -27.721 + 0.6257C + 2.4094T - 0.0023C^2 - 0.0035CT - 0.0132T^2$$

The number of *values of the kP* – experimental decomposition levels in Table 1 above is 9 on the acid, concentration – *9* on C and temperature – 8 on T. So, in the calculations, it will be equal. On the basis of this value, the levels of decay calculated according to the model were determined by the formula $\hat{Y}n = CT = 9 \times 8 = 72$

$$\hat{Y} = \frac{1}{n}\sum_{i=1}^{n}(Y_i) = \frac{1}{72} \times (5780.88) = 80.29\%$$

The determination coefficient of this structure. model – R^2 total $-T_{dis}$ and residual – R_{des} was determined depending on the dispersions.

$$R^2 = 1 - \frac{T_{dis}}{R_{des}} = 1 - \frac{\sum_{i=1}^{n}(y_i - \bar{y})^2}{\sum_{i=1}^{n}(y_i - \hat{y})^2} = \frac{365.12}{13067.93} = 0.972$$

The value of the coefficient of determination indicated that this model was suitable for experimental data with 97.2% accuracy. In the graph shown in Figure 3 below, the experimental values of the degree of decomposition were compared with the values of the degree of decomposition calculated according to the model. The fact that all the points in the graph are located close to it along the regression line once again justified the accuracy of this model. $R^2 = 0.972$

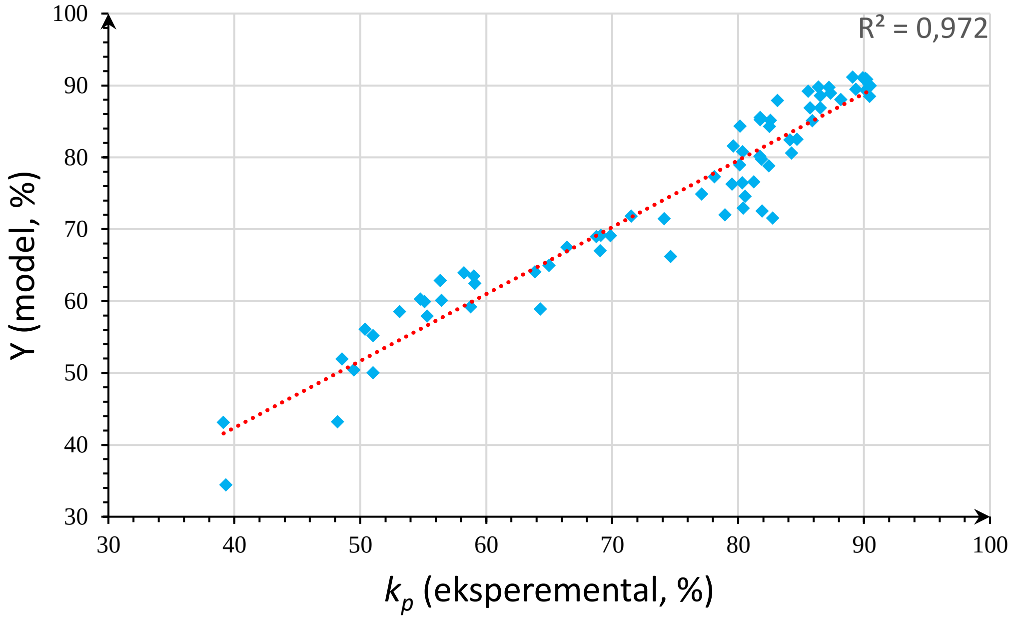

Figure 3 - Comparative comparison of model and experimental values

It has been proven that the model is suitable for research to predict optimal conditions in terms of acid concentration and process temperature parameters.

Conclusion

Based on the above model equation, the optimal conditions and conditions of acid concentration and temperature were deduced. $\frac{\Delta Y}{\Delta C} = 0$; $\frac{\Delta Y}{\Delta T} = 0$

$$\frac{\Delta Y}{\Delta C} = 0.6257 - 2 \times 0.0023C - 0.0035T$$

$$\frac{\Delta Y}{\Delta T} = 2.4094 - 2 \times 0.0132T - 0.0035C$$

Calculations were performed by zeroing the first-order derivatives. As a result of the calculations, the decomposition rate is Y_{max} = 93.57% when the temperature is T = 81.45°C and the concentration of acidity required for the process is C=74.05%.

On the basis of the studies conducted, the model created to determine the possibility of processing magnesitic waste in sulfuric acid under optimal conditions was found and proved to be theoretically useful. This model has made it possible to use the acid decomposition process efficiently and efficiently in the studies because it operates at high precision.

Conflicts of interest. The author declares that there is no conflict of interest regarding the conduct and publication of this study.

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Құрамында магнезит қоспасы бар қалдықтардың жоғары концентрациялы күкірт қышқылында ыдырауы: эмпирикалық модельдеу және оңтайлы шарттарды анықтау

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ТҮЙІНДЕМЕ

Бұл мақалада Зинелбулак тальк-магнезит кен орны шикізатын флотациялық байыту кезінде алынатын магнезит қалдығының күкірт қышқылында ыдырау процестері қарастырылған. Зерттеу нәтижесінде магнезиттің H_2SO_4 ерітіндісінде еруіне реакциялық орта концентрациясы мен температурасының әсері зерттеліп, еру дәрежелері анықталды. Алынған мәліметтер негізінде процестің математикалық моделі жасалды: еру кинетикасы теңдеулер арқылы сипатталып, параметрлері регрессиялық талдау көмегімен есептелді. Модельдеу нәтижелері магнезиттің еру жылдамдығы негізінен қышқыл концентрациясы мен температура факторларына тәуелді екенін көрсетті. Құрылған математикалық модельдің детерминация коэффициентінің R^2=0,972 мәні бұл модельдің 97,2% дәлдікпен жұмыс істейтінін дәлелдеді. Математикалық модельдің дәлдігінің негізінде оңтайлы шарттар температура T=81,45°C және қажетті қышқыл концентрациясы C=74,05% кезінде ыдырау деңгейі Y_{max}=93,57% тең болатыны тәжірибелік және модельдік талдаулар арқылы анықталды. Бұл зерттеу нәтижелері магнезит құрамындағы қалдықтарды қайта өңдеу және олардан магний сульфаты сияқты өнімдерді алу үшін ғылыми негіздерді құруға мүмкіндік береді. Мақалада алдымен шикізат құрамы мен эксперименттік әдістер баяндалып, кейін математикалық модель мен алынған нәтижелер талданып, қорытындылар мен ұсыныстар берілді.

Түйін сөздер: Зинелбулак кені; тальк-магнезит қалдығы; магнезит; күкірт қышқылында еріту; математикалық модель; кинетикалық талдау; регрессиялық талдау.

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Разложение магнезитсодержащих отходов в высококонцентрированной серной кислоте: эмпирическое моделирование и определение оптимальных условий

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АННОТАЦИЯ

В данной статье рассматриваются процессы разложения магнезитсодержащих отходов, образующихся при флотационном обогащении тальк-магнезитовой руды Зинелбулакского месторождения, в серной кислоте. В результате проведённых исследований изучено влияние концентрации реакционной среды и температуры на растворение магнезита в растворе H_2SO_4, а также определены степени растворения. На основе полученных данных была разработана математическая модель процесса: кинетика растворения описана уравнениями, а параметры рассчитаны с использованием регрессионного анализа. Результаты моделирования показали, что скорость растворения магнезита в основном зависит от факторов концентрации кислоты и температуры. Коэффициент детерминации построенной математической модели R^2=0,972 подтвердил, что точность модели составляет 97,2%. На основе достоверности математической модели определены оптимальные условия: при температуре T=81,45°C и концентрации кислоты C=74,05% степень разложения составляет Y_{max}=93,57%, что установлено на основе экспериментальных и модельных анализов. Результаты данной работы закладывают научные основы для переработки отходов, содержащих магнезит, и получения из них таких продуктов, как сульфат магния. В статье сначала изложены состав сырья и экспериментальные методы, затем приведён анализ математической модели и полученных результатов, а также даны выводы и предложения.

Ключевые слова: Зинелбулакское месторождение, тальк-магнезитовый отход, магнезит, растворение в серной кислоте, математическая модель, кинетический анализ, регрессионный анализ.

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