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Mathematical modeling of sulfuric acid leaching of pyrite cinders after preliminary chemical activation

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ABSTRACT
Pyrite cinders, waste products of pyrite concentrate processing by firing to produce sulfuric acid, can serve as raw materials for the extraction of precious, ferrous, and non-ferrous metals. The paper considers the possibilities of obtaining non-ferrous metal concentrate from pyrite cinders by sulfuric acid leaching. This operation is one of the stages in the integrated technology. To increase the extraction of non-ferrous metals during leaching, the method of preliminary chemical activation was used. Chemical activation was carried out in a solution containing 40-120 g/dm^3 $NaHCO_3$ at temperatures of 90-230 ° C and a duration of 30-300 minutes. Sulfuric acid leaching of pyrite cinder after activation was carried out in H_2SO_4 solutions with a concentration of 5-20 % at a temperature of 60 ° C, duration of 30 minutes, and pulp density of 33 %. To determine the optimal conditions for the sulfuric acid leaching of pyrite cinders, a mathematical planning method was used, which allows to assess with a high degree of reliability the influence of the main factors (temperature, pulp density, the concentration of the solution $NaHCO_3$ and duration) and predict an increase in the efficiency of the process by analyzing the numerical values of the regression equations. As a result of sulfuric acid leaching of pyrite cinders after preliminary chemical activation under optimal conditions determined by a mathematical model, the extraction of iron and non-ferrous metals into a solution is 10-15% higher than without activation.
Keywords: pyrite cinders, non-ferrous metals, model, factor, extraction.

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Introduction

One of the methods of sulfuric acid production is the firing of pyrite concentrate to produce sulfur dioxide [[1], [2], [3], [4], [5]]. Production waste - pyrite cinders are stored, which creates a large amount of man-made waste and poses a serious environmental threat [[6], [7], [8]]. At the same time, pyrite cinders can serve as raw materials for complex processing with the extraction of precious, ferrous, and non-ferrous metals [[9], [10]]. The development of rational processing technology is relevant.

The paper considers the possibilities of obtaining non-ferrous metal concentrate from pyrite cinders by sulfuric acid leaching [[11], [12],

[13]]. This operation is one of the stages in the integrated technology.

The study of the parameters and indicators of the process of leaching pyrite cinders was carried out using the method of mathematical planning of the experiment and the selection of technologically significant factors.

The extensive use of mathematical models of technological processes is explained by the fact that the model makes it possible to establish in a phenomenon, subject, or process the main regularities that are characteristic of them, and to neglect the secondary, auxiliary features [[14], [15], [16], [17], [18]]. Development of a mathematical model of the process is directly related to the planning of the experiment.

A full factorial experiment [19] has been used to study the technology of pyrite cinders processing including preliminary chemical activation and sulphuric acid leaching. The method of the full factorial experiment includes consequent stages of mathematical modeling:

- selection of the optimization parameter and affecting factors (temperature, pulp density, $NaHCO_3$ solution concentration, and duration);
- selection of the basic level and interval of variation for each factor;
- checking the reproducibility of the experimental results;
- construction of a mathematical model with calculation of regression equation coefficients;
- testing the adequacy of the regression equation.

Chemical activation of pyrite cinders in a solution of sodium hydrogen carbonate was performed in order to increase the efficiency of sulphuric acid leaching. The use of the given method of preliminary chemical activation in the processing of mineral raw materials has a positive effect on the degree of extraction of useful components [[20], [21], [22]].

The experimental part

X-ray fluorescence analysis was performed on a Venus 200 wave dispersion spectrometer (PANalyicalB.V., Holland).

Chemical analysis was performed on an optical emission spectrometer with inductively coupled plasma (Optima 8300 DV, PerkinElmer, Waltham, MA, USA). The random error component is 2.0%.

X-ray phase analysis was performed using D8 Advance (Bruker, Billerica, Massachusetts, USA).

Discussion of results

The subject of the study was the magnetic fraction of pyrite cinders from the sulphuric acid production of the Tselinnyy Mining and Chemical Combine.

Chemical composition of the magnetic fraction of pyrite cinders wt.%: Na_2O 1.4; MgO 0.74; Al_2O_3 5.69; SiO_2 23.22; P_2O_5 1.1; $SO_3$6.24; Cl^-0.01; K_2O 0.44; CaO 2.52; $TiO_2$0.32; $Fe_2O_3$52.84; CuO 0.25; ZnO 0.53; As_2O_3 0.24; SeO_2 0.3; BaO 2.4; HgO 0.08; PbO 0.16; п.п 1.82; precious metal content, g/t: Au 2.69; Ag 19.3

The phase analysis of the magnetic fraction of the pyrite cinders is shown in Table 1 and Figure 1.

Table 1 – Phase content of the magnetic fraction of pyrite cinders

Name	Formula	%
Magemite	Fe_2O_3	25.1
Hematite	Fe_2O_3	19.1
Quartz	SiO_2	18.0
Albite	$Na(AlSi_3O_8)$	10.2
Trinatrium phosphate zinc oxide hydrate	$Na_3Zn_4O(PO_4)3(H_2O)_6$	9.5
Sodium aluminosilicate	$NaAl_3Si_3O_{11}$	6.7
Barium ferrite	$BaFe_2O_4$	4.7
Natrozharosite	$(Na_{0.67}(H_3O)_{0.33})Fe_3(SO_4)_2(OH)_6$	4.2
Dolomite	$CaMg(CO_3)_2$	2.5

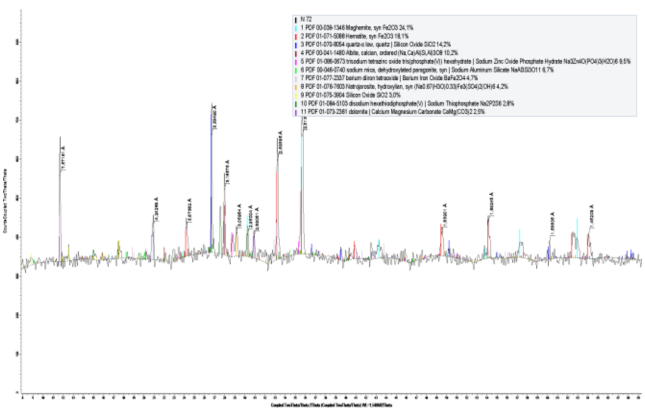

Figure 1 - X-ray diffraction of the magnetic fraction of pyrite cinders

Chemical activation of pyrite cinders was performed in a solution containing 40-120 g/dm^3 $NaHCO_3$ at pulp density from 20 to 80 % and a working temperature of 90-230 ^oC using a thermostatically controlled unit containing 6 autoclaves rotating through the head, with a working volume of 250 cm^3. The activation time was 30-300 minutes.

Sulfuric acid leaching of pyrite cinders after preliminary chemical activation was performed in H_2SO_4 solutions with a concentration of 5-20% at a temperature of 60 °C, duration of 30 minutes, and L: S=3.

A matrix of full factor experiment with the calculation of the main level and interval of variation was made up (Table 2).

The experiment was performed using the planning matrix. Each experiment was duplicated three times during the practical realization of the planning matrix. The results of Fe_2O_3, CuO, and ZnO leaching experiments are given in Tables 3, 4, and 5.

Table 2 - Matrix of the complete factor experiment

Indicators	Factors			
	X_1- temperature , °C	X_2- conc. $NaHCO_3$, g/dm^3	X_3- duration, min	X_4- ratio S:L
Basic level	160	90	180	1:6
Variation interval	70	30	120	1:4
Upper level	230	120	300	1:10
Lower level	90	60	60	1:2

Table 3 - Experimental results for Fe_2O_3 leaching matrix

Batch	**Experiment No.**	**Factor X_1**	**Factor X_2**	**Factor X_3**	**Factor X_4**	**Extracted in solution Fe_2O_3, %**	**y_{exp}**	**$S_i^{2(Fe)}$**
1	1	1	1	1	1	6.26	6.17	0.0171
	2	-1	1	1	1	6.23	6.17	
	3	1	-1	1	1	6.02	6.17	
2	1	-1	-1	1	1	5.28468	5.06636	0.041493
	2	1	1	-1	1	5.033	5.06	
	3	-1	1	-1	1	4.8814	5.06	
3	1	1	-1	-1	1	10.695	10.36	0.453225
	2	-1	-1	-1	1	10.8	10.36	
	3	1	1	1	-1	9.585	10.36	
4	1	-1	1	1	-1	4.9	4.62	0.088433
	2	1	-1	1	-1	4.67	4.62	
	3	-1	-1	1	-1	4.31	4.62	
5	1	1	1	-1	-1	6.16	5.73	0.253433
	2	-1	1	-1	-1	5.87	5.73	
	3	1	-1	-1	-1	5.18	5.73	
6	1	-1	-1	-1	-1	13.05	12.4	0.390633
	2	1	1	1	1	12.43	12.42	
	3	-1	1	1	1	11.8	12.42	
7	1	1	-1	1	1	26.0	23.89	2.838933
	2	-1	-1	1	1	22.23	23.89	
	3	1	1	-1	1	22.07	23.89	
8	1	-1	1	-1	1	10.65	10.23	0.313433
	2	1	-1	-1	1	10.54	10.27	
	3	-1	-1	-1	1	9.63	10.27	

Table 4 - Test results for CuO leaching matrix

Batch	Experiment No.	Factor X_1	Factor X_2	Factor X_3	Factor X_4	Extracted in solution Fe_2O_3, %	y_{exp}	$S_i^{2(Fe)}$
1	1	1	1	1	1	22.72	22.64	0.0148
	2	-1	1	1	1	22.7	22.64	
	3	1	-1	1	1	22.5	22.64	
2	1	-1	-1	1	1	21.35508	21.27	0.00729
	2	1	1	-1	1	21.27	21.27	
	3	-1	1	-1	1	21.18492	21.27	
3	1	1	-1	-1	1	17.9192	17.23	0.47497
	2	-1	-1	-1	1	17.23	17.23	
	3	1	1	1	-1	16.5408	17.23	
4	1	-1	1	1	-1	8.92	8.74	0.07093
	2	1	-1	1	-1	8.88	8.74	
	3	-1	-1	1	-1	8.44	8.74	
5	1	1	1	-1	-1	18.89	18.51	0.32543
	2	-1	1	-1	-1	18.8	18.51	
	3	1	-1	-1	-1	17.86	18.51	
6	1	-1	-1	-1	-1	27.96	26.63	1.7689
	2	1	1	1	1	26.63	26.63	
	3	-1	1	1	1	25.3	26.63	
7	1	1	-1	1	1	43.05	41	4.2025
	2	-1	-1	1	1	41	41	
	3	1	1	-1	1	38.95	41	
8	1	-1	1	-1	1	17.73	16.89	0.7056
	2	1	-1	-1	1	16.89	16.89	
	3	-1	-1	-1	1	16.05	16.89	

We calculated the values of regression coefficients, having received experimental data by formula $b_i = \frac{\sum Y_i}{n}$, where, Y_i-value of optimization parameter in the i-th experiment, N - number of experiments. Regression equations describing metals extraction during sulfuric acid leaching of pyrite cinders were calculated according to obtained coefficients:

y_{Fe}=12.32 + 1.4 x_1-1.65x_2+1.05x_3-7.82x (1)

y_{Zn}=-21.62 +1.07x_1+0.21x_2+0,71x_3-11x_4 (2)

y_{Cu}=-18.9 + 0.86x_1 – 0.43x_2+2.62x_3+ 11.53x_4 (3)

Factor analysis of the results was conducted, to determine the intensity of the effect of the factors under study on the optimization criteria. The effects of factors x_1 – x_4, introduced into the plan at two levels were determined by formulas for linear orthogonal plans. Significant factors were determined for each criterion and ranks which have an effect on the extraction of certain metals were drawn up:

- for iron (y_1): $x_1 > x_3 > x_2 > x_4$;
- for zinc (y_2) : $x_1 > x_3 > x_2 > x_4$;
- for copper (y_3): $x_4 > x_3 > x_1 > x_2$.

The analysis of the obtained regression equations for the extraction process allowed us to conclude that for iron and zinc extraction the greatest contribution is made by factor X_1 - temperature. The coefficient value defines a quantitative measure of the effect of the factor. The sign of the coefficient determines the nature of the effect. The plus sign shows that the value of the factor X_1 increases with an increase in the value of the factor X_1, while the minus sign shows that the value of the optimization parameter decreases. The iron and zinc extraction is less affected by the ratio L:S. The ratio L:S and $NaHCO_3$ concentration, g/dm^3 have the greatest contribution to effective extraction of copper.

Table 5 - Test results for ZnO leaching matrix

Batch	Experiment No.	Factor X_1	Factor X_2	Factor X_3	Factor X_4	Extracted in solution Fe_2O_3, %	y_{exp}	$S_i^{2(Fe)}$
1	1	1	1	1	1	22.05	21.0	1.1025
	2	-1	1	1	1	21	21.0	
	3	1	-1	1	1	19.95	21.0	
2	1	-1	-1	1	1	23.38	22.2	1.2321
	2	1	1	-1	1	22.27	22.7	
	3	-1	1	-1	1	21.16	22.7	
3	1	1	-1	-1	1	12.47	11.8	0.3481
	2	-1	-1	-1	1	11.88	11.8	
	3	1	1	1	-1	11.29	11.8	
4	1	-1	1	1	-1	11.6	11.5	0.3025
	2	1	-1	1	-1	11.05	11.05	
	3	-1	-1	1	-1	10.5	11.05	
5	1	1	1	-1	-1	8.5	8.1	0.16
	2	-1	1	-1	-1	8.1	8.1	
	3	1	-1	-1	-1	7.7	8.1	
6	1	-1	-1	-1	-1	19.95	19	0.9025
	2	1	1	1	1	19	19	
	3	-1	1	1	1	18.05	19	
7	1	1	-1	1	1	47.25	45	5.0625
	2	-1	-1	1	1	45	45	
	3	1	1	-1	1	42.75	45	
8	1	-1	1	-1	1	13.5	12.9	0.36
	2	1	-1	-1	1	12.9	12.9	
	3	-1	-1	-1	1	12.3	12.9	

The results were analyzed using the following algorithm:

- for each series of parallel experiments, the arithmetic average of the response function was calculated;

- for each series of parallel experiments, we calculated the estimation of dispersion;

- we calculated regression equation coefficients;

- we performed the equation adequacy test using Fisher's criterion (Fp) and the table test (Fт)

- we estimated the reproducibility of experiments according to Cochran's criterion G_p;

- we estimated the variance of adequacy.

Calculations according to the above algorithm are shown in Table 6.

Based on the results of the planning matrix experiments, the optimum is the preliminary chemical activation of pyrite cinders in a solution containing 60 g/dm^3 $NaHCO_3$, at a ratio L: S=4 and temperature 120^{0}C. The best results were obtained at leaching in 15 % H2SO4 solution at temperature 60^{0}C after activation of cinders in these conditions. The extraction in sulphuric acid solution was, %: CuO 43.05; ZnO 47.25, and Fe_2O_3 26.0. Further increase in concentration does not lead to an increase of extracted non-ferrous metals in a solution. The degree of extraction of non-ferrous metals in a solution is lower on 10-15 % at leaching of pyrite cinders without chemical activation.

Table 6 - Results analysis

Regression analysis criterion	Fe_2O_3	CuO	ZnO
Equation adequacy dispersion	$S_{ad}^2 = \frac{1}{N-B}\sum Y_{exp} - Y_p$ S_{ad}^2=4.1515625	$S_{ad}^2 = \frac{1}{N-B}\sum Y_{exp} - Y_p$ S_{ad}^2=3.43	$S_{ad}^2 = \frac{1}{N-B}\sum Y_{exp} - Y_p$ S_{ad}^2=1.78
Number of degrees of freedom	f=3	f=3	f=3
Repeatability dispersion	$S_y^2 = \frac{\sum S_i^2}{N}$, where S_i^2 - dispersion of experience at the i-th point S_y^2 =0.54	$S_y^2 = \frac{\sum S_i^2}{N}$, where S_i^2 - dispersion of experience at the i-th point S_y^2 =0.94	$S_y^2 = \frac{\sum S_i^2}{N}$, where S_i^2 - dispersion of experience at the i-th point S_y^2 =1.18
Fischer's criterion	$F_p = \frac{\max(S_{ad}^2, S_y^2)}{min(S_{ad}^2 S_y^2)}$ F_p= 3.09≤F_{tab}=6.59– regression equation is adequate.	$F_p = \frac{\max(S_{ad}^2, S_y^2)}{min(S_{ad}^2 S_y^2)}$ F_p=1.81 ≤F_{tab}=6.59 – regression equation is adequate.	$F_p = \frac{\max(S_{ad}^2, S_y^2)}{min(S_{ad}^2 S_y^2)}$ F_p=1.32 ≤F_{tab}=6.59 – regression equation is adequate.
Cochran's criterion	$G_{calc} = \frac{S_{max}^2}{\sum S^2}$ G_{calc}=0,429<G_{crit}=0,438 – experiments are repeatable	$G_{calc} = \frac{S_{max}^2}{\sum S^2}$ G_{calc}=0,434<G_{crit}=0,438 – experiments are repeatable	$G_{calc} = \frac{S_{max}^2}{\sum S^2}$ G_{calc}=0,436<G_{crit}=0,438 – experiments are repeatable

Conclusions

To increase the degree of extraction of non-ferrous metals from pyrite cinders during sulfuric acid leaching, the method of preliminary chemical activation in $NaHCO_3$ solution was used.

To determine the optimal technological conditions of the process of sulfuric acid leaching of pyrite cinders, a mathematical model is constructed.

Based on the results of experiments conducted on the matrix, regression equations were compiled, which determined the adequacy of the compiled mathematical model.

The analysis of the regression equations showed that for the extraction of iron and zinc, the greatest contribution is made by the X_1 factor – temperature, and for copper, the X_2 factor – pulp density and X_4 - the concentration of $NaHCO_3$, g/dm^3.

As a result of preliminary chemical activation during sulfuric acid leaching of pyrite cinders, the degree of extraction of non-ferrous metals into the solution increased by 10-15%.

Conflict of interests

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Алдын ала химиялық белсендіруден кейінгі пирит күйіктерінің күкіртқышқылды шаймалау үдерісінің математикалық модельдеуі

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ТҮЙІНДЕМЕ

Пирит күйіктері – пирит концентратын күкірт қышқылын алу арқылы күйдіру әдісімен қайта өңдеудің қалдықтары. Олар асыл, қара және түсті металдарды алу үшін шикізат болып табылады. Жұмыста күкірт қышқылды ерітінділеу әдісімен пиритті күйіктен түсті металл концентратын алу мүмкіндігі қарастырылған. Бұл операция кешенді технологияның кезеңдерінің бірі болып табылады. Ерітінділеу кезінде түсті металдарды алуды арттыру үшін алдын-ала химиялық белсендіру әдісі қолданылады. Химиялық белсендіру 40-120 г/дм^3 $NaHCO_3$ бар ерітіндіде 90-230 ° С температурада және ұзақтығы 30-300 минут ішінде жүргізілді. Белсендірілгеннен кейін пирит күйіктерін күкіртқышқылды ерітінділеу концентрациясы 5-20% H_2SO_4 ерітінділерінде 60^{o}C температурада, ұзақтығы 30 минут және С:Ж қатынасы 3:1 кезінде жүргізілді. Пириттік күйіктерді күкіртқышқылды ерітінділеудің оңтайлы жағдайларын анықтау үшін негізгі факторлардың (температура, С:Ж қатынасы, $NaHCO_3$ ерітіндісінің концентрациясы) әсерін жоғары сенімділікпен бағалауға және регрессия теңдеулерінің сандық мәндерін талдай отырып, процесс тиімділігінің артуын болжауға мүмкіндік беретін математикалық модельдеу әдісі қолданылды. Математикалық модельмен анықталған оңтайлы жағдайларда алдын-ала химиялық белсендіруден кейін пирит күйіктерін күкірт қышқылымен ерітінділеу нәтижесінде темір мен түсті металдардың ерітіндіге шығарылуы белсендірілмегенге қарағанда 10-15% жоғары болды.

Түйін сөздер: пирит күйіктері, түсті металдар, модель, фактор, бөліп алу.

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Математическое моделирование сернокислотного выщелачивания пиритных огарков после предварительной химической активации

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АННОТАЦИЯ

Пиритные огарки, отходы переработки пиритного концентрата методом обжига с получением серной кислоты, могут служить сырьем для извлечения благородных, черных и цветных металлов. В работе рассмотрены возможности получения концентрата цветных металлов из пиритных огарков способом сернокислотного выщелачивания. Эта операция является одним из этапов в комплексной технологии. Для повышения извлечения цветных металлов при выщелачивании применен метод предварительной химической активации. Химическую активацию проводили в растворе, содержащем 40-120 г/дм3 NaHCO3 при температурах 90-230оС и продолжительности 30-300 минут. Сернокислотное выщелачивание пиритных огарков после активации проводили в растворах H_2SO_4 концентрацией 5-20 % при температуре 60оС, продолжительности 30 минут и Ж:Т=3. Для определения оптимальных условий проведения сернокислотного выщелачивания пиритных огарков использовали метод математического планирования позволяющий с высокой степенью достоверности оценить влияние основных факторов (температуры, отношения Ж:Т, концентрации раствора NaHCO3 продолжительности) и прогнозировать повышение эффективности процесса, анализируя численные значения уравнений регрессии. В результате проведения сернокислотного выщелачивания пиритных огарков после предварительной химической активации в оптимальных условиях, определенных математической моделью, получено извлечение в раствор железа и цветных металлов на 10-15 % выше, чем без активации.

Ключевые слова: пиритные огарки, цветные металлы, модель, фактор, извлечение.

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