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Original article

MATHEMATICAL ANALYSIS OF THE SORPTION PROCESS OF MINE WATER PURIFICATION

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Contaminated mine water with a large amount of salts makes underground and surface water sources unsuitable for household and drinking needs. Already in 2016, there was a shortage of drinking water in the Rostov region. Water from surface sources does not meet the hygienic requirements for chemical parameters in 36.1% of the samples taken. Water from underground sources in terms of color, turbidity, total hardness, dry residue, content of iron ions, manganese, hydrogen sulfide, nitrates, ammonia, chlorides, sulfates, magnesium, sodium did not meet the required standards in 72.2% of cases. Due to the need for huge expenses for the purification of highly mineralized waters and the poor development of cheap technologies for the neutralization of large volumes, attempts to purify discharged or flowing mine waters to a safe level turn out to be practically fruitless. In this article, studies were carried out to reduce the concentration of dissolved ions of heavy metals (iron, manganese, copper and zinc) in mine wastewater by sorption in a static mode (charring) using rice husk biochar with electromagnetic treatment. The authors proposed a method for pre-treatment of the sorbent from rice husk biochar in an electromagnetic field at a process activation unit. The results of laboratory tests confirmed the high efficiency of the sorbent for the removal of heavy metal ions from mine waters. On the basis of the results obtained, optimization of the sorption purification of mine waters in the mode of carbonization was carried out using the method of mathematical planning of the experiment (full factorial experiment FFE 2k). The factors most influencing the efficiency of mine wastewater treatment from iron and zinc ions have been identified. With a probability of 0.95, the proposed models are adequate, and they can be used to describe the sorption process when using the studied sorbent to remove heavy metal ions (iron, zinc, etc.), while the optimal concentration of the sorbent from rice husks is within 0.5 mg/l. With an increase and decrease in the concentration of the sorbent above the optimal values, the efficiency of sorption decreases, and this affects the iron to a greater extent. In general, the obtained sorbent has a chemical composition similar to that of activat-

ed carbon (the most widely used sorbent for water purification), but at the same time it is a cheap production waste, which confirms its efficiency, both technological and economic, when used to purify natural and waste water. With the introduction of the proposed treatment scheme for mine water treatment facilities, it is expected that the concentrations of dissolved heavy metal ions, in particular, iron, zinc, manganese, will decrease to the concentration of the discharge into the reservoir.

Keywords: mine wastewater; sorption under static conditions; sorbents from industrial waste; biochar; rice husks; heavy metal ions; process activation unit; electromagnetic treatment; active experiment; mathematical planning

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Научная статья

МАТЕМАТИЧЕСКИЙ АНАЛИЗ СОРБЦИОННОГО ПРОЦЕССА ОЧИСТКИ ШАХТНЫХ ВОД

А.С. Смоляниченко, А.К. Халюшев, Е.В. Яковлева

Загрязненная шахтная вода с большим количеством солей делает непригодными для хозяйственно-питьевых нужд подземные и поверхностные источники воды. В Ростовской области уже в 2016 году появился дефицит питьевой воды. Вода поверхностных источников не отвечает гигиеническим требованиям по химическим показателям в 36,1% отобранных проб. Вода подземных источников по показателям цветности, мутности, общей жесткости, сухого остатка, содержанию ионов железа, марганца, сероводорода, нитратов, аммиака, хлоридов, сульфатов, магния, натрия не соответствовала требуемым нормам в 72,2% случаев. В связи с необходимостью огромных затрат на очистку высокоминерализованных вод и слабой разработанностью дешевых технологий обезвреживания крупных объемов, попытки очистить сбрасываемые или стекающие шахтные воды до безопасного уровня оказываются практически безрезультатными. В данной статье проведены исследования по снижению концентрации растворенных ионов тяжелых металлов (железа, марганца, меди и цинка) на шахтных сточных водах сорбцией в статическом режиме (углевание) с применением биоугля рисовой шелухи с электромагнитной обработкой. Ав-

торами предложен способ предварительной обработки сорбента из биоугля рисовой шелухи в электромагнитном поле на установке активации процессов. Результаты лабораторных испытаний подтвердили высокую эффективность сорбента для удаления ионов тяжелых металлов из шахтных вод. На основании полученных результатов проведена оптимизация сорбционной очистки шахтных вод в режиме углевания с применением метода математического планирования эксперимента (ПФЭ 2^к). Выявлены факторы, наиболее влияющие на эффективность очистки шахтных сточных вод от ионов железа и цинка. С вероятностью 0,95 предлагаемые модели являются адекватными, и они могут применяться для описания процесса сорбции при применении исследуемого сорбента для удаления ионов тяжелых металлов (железа, цинка и т.д.) при этом оптимальная концентрация сорбента из рисовой шелухи в пределах 0,5 мг/л. При увеличении и уменьшении концентрации сорбента сверх оптимальных значений эффективность сорбции снижается и в большей степени это отражается на железе. В целом полученный сорбент имеет химический состав близкий по составу к активированному углю (наиболее широко применяемого для очистки воды сорбента), но при этом является дешевым отходом производства, что подтверждает его эффективность как технологическую, так и экономическую при использовании для очистки природных и сточных вод. При внедрении предложенной схемы очистки на очистные сооружения шахтных вод ожидается снижение концентраций растворенных ионов тяжелых металлов, в частности, железа, цинка, марганца до концентрации ПДК сброса в водоем.

Ключевые слова: шахтные сточные воды; сорбция в статических условиях; сорбенты из отходов производств; биоуголь; рисовая шелуха; ионы тяжелых металлов; установка активации процессов; электромагнитная обработка; активный эксперимент; математическое планирование

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Introduction

It is known that the main influx of wastewater comes from mines in which underground or open pit mining is carried out. Typically, mine water is collected and stored in above-ground dams or underground caverns. Over time, the levels of wastewater and the intensity of their pollution with heavy metals are gradually increasing, which is one of the main causes of environmental pollution. In

connection with this phenomenon, there is a need to treat mine wastewater to reduce the level of negative impact on the existing ecological system [1, 6, 16, 19].

The formation of the chemical composition of mine water inflows mainly depends on hydrological, mining-geological and mining-technical factors, and the depth of occurrence of minerals has a significant effect. Mine water has a mineralization of 5 to 15 g/dm³, which is about the same times higher than the usual mineralization of river waters and exceeds the maximum allowable concentration (MPC) of salts in drinking water. Water also contains a large amount of iron, many other harmful substances (manganese, zinc, nickel, etc.) and poses a great danger to river inhabitants. In groundwater, iron and manganese are found in soluble Fe²⁺ and Mn(II) compounds. The standards and sanitary norms of the Russian Federation set the MPC in drinking water for iron - 0.3 mg/dm³, for manganese - 0.1 mg/dm³. These values are in line with the recommendations of the World Health Organization, the standards of the United States and some other countries. Somewhat more stringent standards are adopted in Sweden and in some other countries of the European Community: 0.2 and 0.05 mg/dm³, respectively [5, 7, 8, 10, 17].

A wide range of methods are observed for the purification of mine waters. In general, at the first stage of mine water treatment, mechanical methods are used, such as straining, clarification, filtration, separation of the solid phase under the action of centrifugal forces. At the next stage, chemical, physical and biological methods of water purification are used. In chemical methods, various reagents are used to change the chemical composition of pollutants or the form of their presence in wastewater (coagulation, flocculation, neutralization, neutralization, disinfection). Physical methods are the extraction and neutralization of harmful impurities by changing the state of aggregation of water, exposing water to ultrasound, magnetic field, ultraviolet, etc. Biological methods are designed to purify water containing pollution of biogenic organic origin. An obligatory stage in the purification of mine and quarry waters is their disinfection before being discharged into water bodies, since, according to sanitary standards, these waters are classified as sewage that is dangerous in an epidemic sense. Water disinfection is carried out by various chemical (ozonation) and physical (UV treatment) methods. Specific capital costs for the construction of efficient and efficient mine water treatment plants in the case of a complete supply of equipment are about \$ 6-9 thousand per 1 m³/h.

An analysis of the effectiveness of the methods used for cleaning mine waters showed that the overwhelming majority of treatment facilities available at coal enterprises do not provide cleaning of mine waters to the regulatory requirements from oil products, iron compounds, mineral salts and other contaminants due to the imperfection of the treatment schemes used [1, 3, 15].

In accordance with the requirements for the quality of mine water treatment, many enterprises need to modernize treatment facilities and introduce new and efficient technologies. The use of well-known membrane purification methods (ultrafiltration and reverse osmosis) makes it possible to achieve maximum permissible concentrations (MPC) for pollutants discharged into a reservoir. However, given the significant volume of mine water, as well as the problem of reverse osmosis concentrate disposal, it is necessary to find alternative high-performance and efficient treatment technologies based, for example, on the sorption extraction of impurities [4, 9, 10, 12, 14, 18].

In connection with the above, the purpose of the study is to intensify the purification of mine and quarry waters from heavy metal ions before discharge into surface water bodies to the maximum permissible concentrations for fishery reservoirs by sorption using a carbon-containing sorbent from agricultural waste with electromagnetic processing.

Materials and Methods

Biochar obtained from the fruit shells of rice grains (rice husks) was taken as the initial sorption carbon material.

The initial biochar was obtained by carbonizing the fruit shells of rice grains in a muffle furnace at a temperature of 600°C for 30 minutes with preliminary soaking in NaOH alkali solution for a day and subsequent washing to a normal pH value. Next, the biochar was processed in a process activation unit (PAU) (Fig. 1). A sample of biochar from the fruit shells of rice grains was stirred in distilled water, placed in a non-magnetic cylinder with ferromagnetic particles $m = 200$ g, exposed to a rotating electromagnetic field for 30 seconds in a process activation apparatus for processing materials, after which it was dried in a drying cabinet for 4 hours at $t=105^\circ\text{C}$ (Fig. 2). Ferromagnetic particles rotating in an electromagnetic field cause a magnetostrictive effect, leading to the reduction of oxides on the surface of the particles of the processed material. This method made it possible to increase the carbon content in the sorbent from 43.3 to 78.5% compared to the original biochar, to reduce the content of impurities in the sorbent, including the silicon content from 8.2% to 2.1% (Table 1, Fig. 3). It is obvious that a change in the chemical composition of biochar of this kind occurs under the influence of a complex of processes that occur in the PAU.

It can be assumed that the change in the quantitative composition of carbon is associated with the breaking of intermolecular bonds during processing in the PAU. In addition, when interacting with ferromagnetic particles, SiO_2 forms chemical compounds with their surface layer, resulting in a decrease in the Si content, %.

Also, activation in the PAU makes it possible to grind the sorbent to nanosizes of 1-50 nm with the formation of mesopores (average desorption diameter - 167Å) and micropores (average diameter - 4.92Å), thereby increasing the composition uniformity. Thus, the preparation of a carbon sorbent was carried out, which confirmed its effectiveness in the treatment of mine wastewater in laboratory conditions.

The quality indicators of the original biochar from the fruit shells of rice grains and the same with electromagnetic processing are given in Table 1.

Table 1.

Physical and chemical characteristics of the obtained samples of rice husk biochar with and without electromagnetic treatment

Characteristic	Biochar rice husk	
	with electromagnetic treatment	without electromagnetic treatment
Ash content, %	61,2	63,7
Moisture contents, %	3,3	5,4
Specific surface, m ² /g	7,93	54,6
Relative volume of pores up to 900 Å in diameter, cm ³ /g	0,042	0,049
Average mesopore diameter by desorption, Å	167	124
Micropore volume, cm ³ /g	0,0033	0,0211
Average micropore diameter, Å	4,92	3,45
Adsorption activity for iodine, %	14	19
Adsorption activity on methylene blue, %	-	52

On Fig. 1 shows photographs of the surface of biochar from the fruit shells of rice grains with electromagnetic processing and the original, taken on a ZEISS electron microscope with nanometer resolution.

Studies were carried out on mine wastewater to reduce the concentration of dissolved ions of heavy metals (iron, manganese, copper and zinc) by sorption in a static mode (charcoalization). Static conditions mean that the liquid particle does not move relative to the sorbent particle, i.e. moves with her.

The experiments were carried out under laboratory conditions in the following order:

- treatment of initial mine wastewater by carbonization using biochar from the fruit shells of rice grains with electromagnetic treatment with a variable dose of sorbent 0.1; 0.3; 0.5; 0.7 and 1.0 g/dm³ in the stirring mode at 45 rpm for 30 minutes;

- treatment of the resulting suspension with the SKIF-180 reagent (a mixture of aluminum polyoxychloride coagulant and cationic flocculant polydiallyldimethylammonium chloride (polyDADMAC)) at a dose of 1.0 mg/dm³;
- settling of the treated water for 30 minutes to carry out the coagulation process;
- filtration through a pressure filter with quartzite loading.

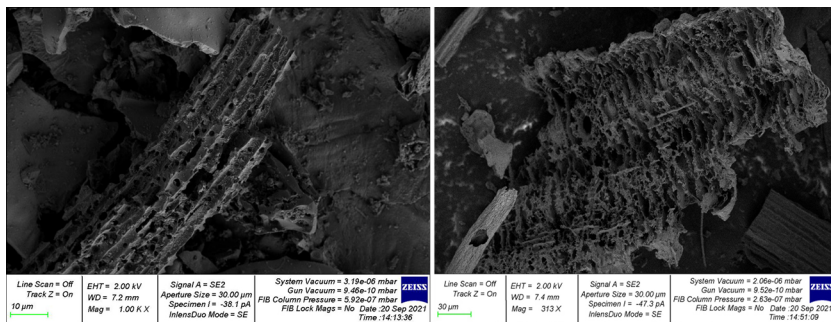


Fig. 1. Microscopic images of the surface of biochar from the fruit shells of rice grains with electromagnetic processing with a resolution of: a) 20 μm and b) 1 μm

The chemical analysis of mine wastewater was carried out by an accredited laboratory. The measurements were carried out on a UNICO 1201 spectrophotometer.

Results and Discussion

The results of experiments on mine wastewater treatment by carbonization using rice husk biochar with electromagnetic treatment are given in Table 2, 3.

Table 2.

Results of measurements of the content of heavy metal ions in treated mine wastewater

No.	Index	Source mine water	Norm	Charcoalization using rice husk biochar with electromagnetic treatment dose, mg/dm ³				
				0,1	0,3	0,5	0,7	1,0
1	2	3	4	5	6	7	8	9
1	Iron total, mg/dm ³	36,47± 3,65	0,3	20,34± 2,03	18,88± 1,89	3,99± 0,60	17,92± 1,79	11,46± 1,715
2	Manganese, mg/dm ³	5,619± 1,124	0,1	7,356± 1,471	4,150± 0,83	6,047± 1,209	4,321± 0,864	5,386± 1,077
3	Copper, mg/dm ³	0,012± 0,002	1,0	0,008± 0,002	0,023± 0,005	0,016± 0,003	0,011± 0,002	0,012± 0,002
4	Zinc, mg/dm ³	0,376± 0,128	5,0	0,064± 0,022	0,185± 0,063	0,059± 0,02	0,147± 0,05	0,153± 0,052

Table 3.

Results of measurements of redox potential, salinity and pH in treated mine wastewater

No.	Index	Source mine water	Coagulation, SKiF, D=1.0mg/l	Charcoalization using rice husk biochar with electromagnetic treatment dose, mg/dm ³				
				0,1	0,3	0,5	0,7	1,0
1	2	3	4	5	6	7	8	9
1	ORP	-0,07	+073	+080	+108	+050	+130	+130
2	pH	6,76	6,35	6,39	6,30	6,35	6,15	6,20
3	TDS	267	278	258	262	271	266	262

A visual assessment of the degree of purification of mine wastewater according to the above method is shown in fig. 2.

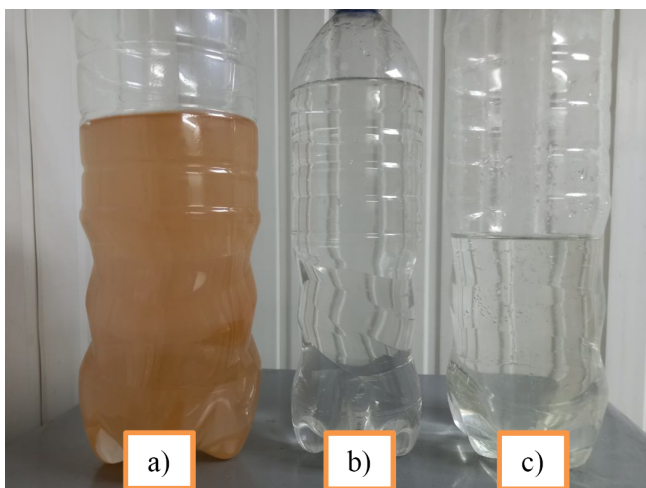


Fig. 2. Visual assessment of the degree of purification of mine wastewater in the sorption mode under static conditions: a) initial mine wastewater; b) treated mine water; c) tap water

Based on the results of the research, optimization of the sorption purification of mine waters in the mode of carbonization was carried out using the method of mathematical planning of the experiment - full factorial experiment (FFE 2^k) [13, 20].

The following parameters were taken as the response function:

$Y_1(X_1, X_2)$ - The content of iron in wastewater Fe^{2+} - 36.47 ± 3.65 mg/l, (100) %;

$Y_2(X_1, X_2)$ - Zinc content in sewage Zn^{2+} - 0.376 ± 0.128 mg/l, (100) %.

The values of the variation factors and their physical meaning are presented in Table 4.

Table 4.

The value of factors and levels of variation of FFE 2^k

No.	Factor code	The physical meaning of the factor	Units rev.	Variation interval	Levels of variation		
					-1	0	+1
1	X_1	Dose of rice husk biochar with electromagnetic treatment	mg/dm ³	$\pm 0,2$	0,3	0,5	0,7
2	X_2	Acidity of the environment	pH	$\pm 0,1$	6,3	6,2	6,1

Using the least squares method, the basic regression equations were obtained, which are presented as polynomials of the 2nd degree:

$$Y(X_1, X_2) = B_0 + B_1 \cdot X_1 + B_2 \cdot X_2 + B_3 \cdot X_1 \cdot X_2 + B_4 \cdot X_1^2 + B_5 \cdot X_2^2 \quad (1)$$

The experimental plan and the results of the optimization parameters are given in Table 5.

Table 5.

Experimental plan and results of optimization parameters

№	Variable encoding		Natural values		Optimization parameter values					
	X_1	X_2	X_1	X_2	Y_1			Y_2		
					1	2	3	1	2	3
1	-1	-1	0,3	6,30	48,2	50,7	46,5	48,2	50,7	52,9
2	-1	0	0,3	6,20	40,6	44,7	49,1	42,6	46,7	51,1
3	-1	+1	0,3	6,10	37,4	41,8	43,9	37,4	41,0	40,5
4	0	-1	0,5	6,30	89,5	94,2	83,6	83,5	90,2	80,6
5	0	0	0,5	6,20	80,9	89,4	85,8	79,3	84,4	81,8
6	0	+1	0,5	6,10	86,0	80,2	75,3	80,0	77,2	75,3
7	+1	-1	0,7	6,30	52,8	59,9	65,7	67,8	70,9	75,7
8	+1	0	0,7	6,20	60,7	63,6	69,5	80,7	74,6	72,5
9	+1	+1	0,7	6,10	47,2	50,8	54,7	57,2	60,8	63,7

Statistical data processing was performed using the Mathcad program, which made it possible to obtain regression equations in the form of polynomials of the second degree. To obtain regression equations, we calculate the coefficients, the values of which are presented in Table 6.

Table 6.

Estimated coefficients of regression equations

The name of the output parameter of the equation		Coefficients of equations					
		B ₀	B ₁	B ₂	B ₃	B ₄	B ₅
Y ₁	Iron content Fe ²⁺ in wastewater	87,219	7,289	-4,356	-33,44	-2,578	-1,533
Y ₂	The content of zinc in wastewater Zn ²⁺	84,1	11,856	-4,872	-23,87	-4,05	-0,1

We calculate the sample variance and standard deviations. We carry out a test of the significance of the coefficients according to the Student's criterion.

$$D := \frac{\sum_{i=1}^9 \sum_{j=1}^3 (Y_{i,j} - MY_i)^2}{n \cdot (n - 1)} \quad (2)$$

$$D = 5,127$$

$$S := \sqrt{D_S} \quad (3)$$

$$S = 2.264$$

We calculate the inverse distribution function of the Student's criterion for the significance equation of 0.95 and the number of degrees of freedom 18.

To determine the tabular value of the Student's criterion, the number of degrees of freedom is equal to twice the number of experiments

We calculate the confidence error and compare it with the modules of coefficient values.

$$\xi := qt(0.95, 18) \cdot S \quad (4)$$

$$\xi = 3.926$$

If the calculated values of the coefficients are less than the confidence error then are insignificant, then the regression equations take the form:

$$V(x_1, x_2) := a_1 + a_3 \cdot x_2 + a_4 \cdot x_1^2 + a_5 \cdot x_2^2 + a_6 \cdot x_1 \cdot x_2 \quad (5)$$

We check the adequacy of the model by the Fisher criterion (F):

$$SAD := \sum_{i=1}^9 \frac{(IQ_i - MY_i)^2}{1} \quad (6)$$

$$F := \frac{SAD}{D} \quad (7)$$

$$SAD = 31.132$$

$$F = 6.073$$

We calculate the inverse Fisher distribution function: $qF(0.95, 18, 1) = 247.323$

Since $F < qF$, with a probability of 0.95 the resulting equation is adequate to the data.

The results of the statistical tests obtained (Fisher's test F ; variance D_0^2 ; standard deviation S_0 and standard error in determining the coefficients ξ) are summarized in Table 7.

Table 7.

Statistical optimization criteria

The name of the output parameter of the equation		Statistical criteria			
		F	D_0^2	S_0	ξ
Y_1	Iron content Fe^{2+} in wastewater	6,07	5,127	2,26	3,93
Y_2	The content of zinc in wastewater Zn^{2+}	12,83	3,009	1,735	3,008

Based on the equations obtained, the response surfaces were constructed using the Mathcad software.

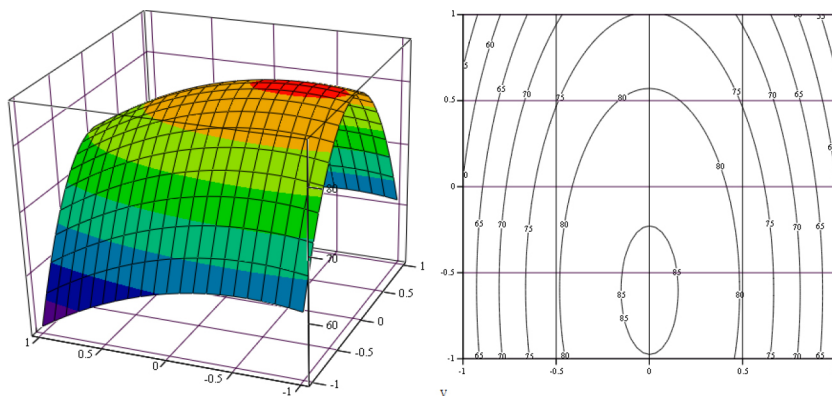


Fig. 3. The dependence of the zinc content in the treated mine water on the dose of the sorbent (X_1 - the axis of values is vertical on a flat graph) and the pH of the medium (X_2 - the axis of categories is horizontal on a flat graph)

Analysis of the results of the obtained model, presented in the form of regression equations, shows that the coefficient B_1 with the factor X_1 (the dose of the applied sorbent) has a positive effect on the process of reducing iron and zinc in water. Moreover, the efficiency of sorption is higher for zinc ($B_1=11.856$) than for iron ($B_1=7.289$). At the same time, the negative value of the coeffi-

cient B_2 at the factor X_2 (change in the acidity of the medium) indicates that a slight decrease in pH leads to a negative effect on the efficiency of sorption both in the case of iron ($B_2 = -4.356$) and in the case of zinc ($B_2 = -4.872$). The joint interaction of these factors (X_1, X_2) also significantly affects the sorption efficiency in the first ($B_3 = -33.44$) and in the second case ($B_3 = -23.87$). The remaining coefficients can be neglected, since they are insignificant for iron (B_4, B_5) and zinc (B_5), because they are less than the standard error in determining the coefficients.

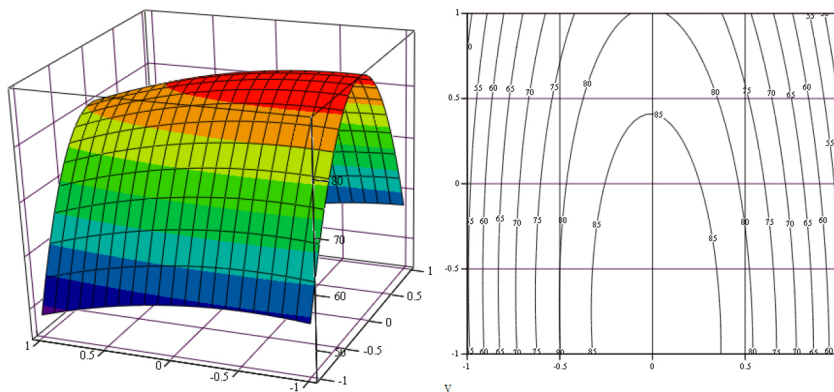


Fig. 4. The dependence of the iron content in the treated mine water on the dose of the sorbent (X_1 - the axis of values is vertical on a flat graph) and the pH of the medium (X_2 - the axis of categories is horizontal on a flat graph)

In general, based on the inequality $F < qF$ with a probability of 0.95, the proposed models are adequate and they can be used to describe the sorption process when using the studied sorbent to remove heavy metal ions (iron, zinc, etc.), while the optimal concentration of the sorbent from rice husk within 0.5 mg/dm^3 . With an increase and decrease in the concentration of the sorbent above the optimal values, the efficiency of sorption decreases, and this affects the iron to a greater extent.

Conclusions

In conclusion, the following conclusions can be drawn from the conducted research.

The proposed method for activating rice husk biochar (rice straw) by electromagnetic means has confirmed its effectiveness in the preparation of this carbon sorbent for the treatment of mine wastewater in laboratory conditions. In general,

the obtained sorbent has a chemical composition similar to that of activated carbon (the most widely used sorbent for water purification), but at the same time it is a cheap production waste, which confirms its efficiency, both technological and economic, when used to purify natural and waste water. With the introduction of the proposed treatment scheme for mine water treatment facilities, it is expected that the concentrations of dissolved heavy metal ions, in particular, iron, zinc, manganese, will decrease to the concentration of the discharge into the reservoir.

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