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OPTIMIZATION OF COAGULATION WASTEWATER TREATMENT OF LEATHER INDUSTRY

The object of the study was the process of coagulation treatment of wastewater of leather industry enterprises. The treatment of samples of real wastewater from a leather production was investigated. Such wastewater is characterized by high concentrations of pollutants of varying degrees of dispersion, which is due to the use of a large number of various chemicals in the leather production process. During technological operations related to the skinning and processing of skins, these substances enter the wastewater, which is discharged into the sewer. A peculiarity of leather production wastewater is large fluctuations in composition and volume, high concentrations of pollutants, and an increased content of suspended solids. Such waters have certain toxicity. The existing methods of wastewater treatment of leather industry enterprises do not provide the necessary treatment efficiency, do not allow to create a closed circulation of water at enterprises, to use or regenerate valuable components of wastewater, and to ensure their economically feasible disposal. Therefore, the research is aimed at improving the process of coagulation wastewater treatment of leather factories using mathematical modeling and optimization methods. This will make it possible to modernize existing wastewater treatment schemes. The experiments were carried out on the Niva Jar-test laboratory unit, which allows simultaneous examination of 6 samples. Samples of wastewater from leather production after the fattening-filling and dyeing stages were studied. The effectiveness of the coagulation process was determined by the degree of wastewater treatment. The factors were analyzed and the intervals of variation of factors that have a significant impact on the process of coagulation treatment of wastewater from leather factories were established. An experiment plan was drawn up and implemented in order to study the effect of coagulants based on aluminum and ferrum, as well as flocculants on the quality of wastewater treatment. The choice of quality indicators of the coagulation process is substantiated. A statistical analysis of the results of experimental studies was performed, a correlational analysis of the interdependence of parameters and indicators of the quality of the coagulation process was performed. Aluminum-based coagulant has been found to be more effective than iron-based coagulant. The results of mathematical modeling were used to determine the optimal parameters of the coagulation process: pH 10, coagulant dose – 2.5 g/dm^3, flocculant dose – 100 mg/dm^3.

Keywords: *wastewater from leather factories, coagulation, degree of treatment, optimal parameters of the coagulation process.*

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1. Introduction

Ensuring water quality is extremely important because it not only meets basic human needs, but also ensures that they are met in sufficient quantity. In order to effectively overcome the current water shortage crisis, there is an urgent need to understand the imbalance in urban water supply systems, to manage the movement of water in urban areas. Water is inherently cyclical in nature, an example of which is the water cycle, which illustrates the cyclical nature of nature. Therefore, the application of circular economy principles in the areas of water supply and drainage is a potential solution to these problems and can contribute to sustainable water management practices [1].

The circular economy assumes that the flow of materials and waste becomes circular in order to achieve their revaluation throughout the production system. Thus, the risk of supply of resources and materials is reduced, increasing the ability of the system to respond to shortages of raw material sources.

The circular economy has a positive effect not only on production processes, but also on the ecosystem and society. In terms of water reuse, wastewater from treatment plants is being re-evaluated and considered as a new non-traditional source of water, taking a step forward in water management. For this reason, water reuse is a fundamental axis of the circular economy in the water sector not only to meet water demand, but also to strengthen the water-energy-ecosystem link [2].

The leather industry plays a significant role in the world economy, its volume of world trade is estimated at approximately 100 billion USD per year [3]. Leather production consists of the transformation of hides and skins of animals into valuable materials used in the production of such products as shoes, leather goods, etc., which require a number of chemical and mechanical processes [4].

Tanning and other leather processing methods use large amounts of fresh water, dyes, chemicals, and salts and generate toxic waste [5].

Water consumption by leather enterprises is from 25 to 60 m^3 per ton of fresh leather, and the output is 500 kg of finished leather per ton of leather. Depending on the process, more than 440 kg of chemicals can be used per ton of leather [6].

These chemicals dissolve in the process water, which creates wastewater with a very high content of organic substances and polluting inorganic compounds (chromium, chlorides, ammonium, sulfides, sulfates). Total suspended solids, total dissolved solids, and chemical oxygen demand make leather production wastewater extremely toxic to all living species. A significant part of these effluents, approximately 90 %, enters the environment [7, 8]. In addition, the water has an unpleasant smell, a dark color and foams.

Groundwater pollution occurs when wastewater from these industries seeps through the soil from unlined pipes, drains, and ponds, or from spills and landfills [9, 10].

At present, the problem of wastewater treatment of leather factories remains relevant, since the existing technologies do not provide the necessary quality of treatment of such waters – multi-component, concentrated and toxic for microorganisms active sludge of biological treatment facilities.

The use of traditional methods of wastewater treatment of leather industry enterprises, which include mechanical, biochemical, chemical or reagent treatment [11, 12], do not provide the necessary treatment efficiency. In addition, classic schemes do not allow to create a closed circulation of water at enterprises, to use or regenerate valuable components of wastewater, and to ensure their economically feasible disposal. It should be noted that there are still no reliable and effective schemes for the treatment of wastewater from leather factories [13, 14].

The aim of research is to improve the process of coagulation treatment of wastewater from leather factories using mathematical modeling and optimization methods. This will make it possible to modernize existing wastewater treatment schemes.

To achieve the aim, the following objectives were set:

- to conduct an a priori analysis of the parameters of the coagulation process, which affect the quality of wastewater treatment of leather production;
- to conduct experimental studies of the effectiveness of the use of selected coagulants and flocculants on the quality of coagulation treatment of leather production wastewater in accordance with the plan of a full factorial experiment;
- to formalize the task of optimizing the coagulation process and determine the optimal conditions for the leather industry wastewater treatment process.

2. Materials and Methods

The object of research is the process of coagulation wastewater treatment of leather industry enterprises.

The subject of research is the influence of the parameters of the coagulation process on the wastewater treatment quality of leather enterprises to ensure the regulatory values of residual concentrations.

The work assumes the linearity of the correlation between the parameters of the coagulation process, as well as the fact that the response surface is quite smooth.

Samples of wastewater from leather production after the fattening-filling and dyeing stages were studied. To conduct the experiment, samples of 400 cm^3 were taken and the pH was adjusted with a KOH solution until pH values of 10 were reached. To measure the pH of the obtained solutions, a universal ionometer AI-125 (Ukraine) was used. In accordance with the plan of the full factorial experiment (FFE) [15], solutions of coagulants $Al_2(SO_4)_3$ or $FeCl_3$ with a concentration of 25 g/dm^3 and 5 % solutions of flocculants were added to each sample. As flocculants, polyhexamethyleneguanidine (PHMG) in chloride and acetate forms and flocculant P228, which was obtained by splitting polyethylene terephthalate waste under the action of monoethanolamine and glycerin at a temperature of 180–200 °C, were used.

Normalization of parameters for the FFE implementation is carried out according to the formula:

$$\bar{x}_i = \frac{2x_i - x_{i\,\max} - x_{i\,\min}}{x_{i\,\max} - x_{i\,\min}}, \qquad (1)$$

where $\bar{x}_i$ and x_i – coded and natural value of the i-th parameter, respectively; $x_{i\,\max}$ and $x_{i\,\min}$ – the maximum and minimum values of the i-th parameter, respectively.

The prepared samples were placed in the NIVA (Norway) laboratory facility (Fig. 1), which allows simultaneous examination of 6 samples.

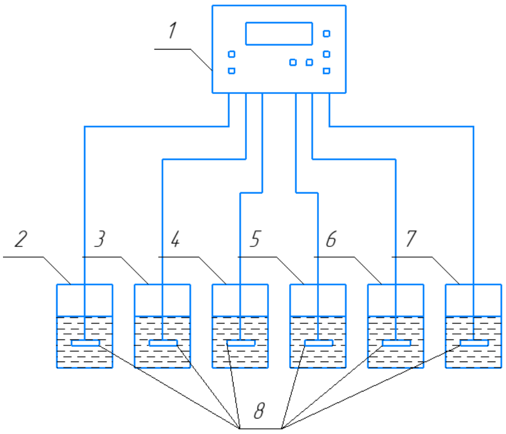


Fig. 1. Laboratory unit for conducting the Niva Jar-test: 1 – power supply unit and control of the unit; 2–7 – laboratory beakers with a volume of up to 1 dm^3; 8 – mechanical stirrers

Coagulation was carried out for 30 minutes according to the program shown in Fig. 2.

The parameters chosen as indicators of the quality of the coagulation process are: turbidity (y_1, NUT), optical density of the solution (y_2 at $\lambda=400$ nm), sediment height (y_3, mm).

The turbidity of purified water samples was measured with a CyberScan WL Turbidimeter TB1000 (USA); determination of color was carried out using a spectrophotometer ULAB 101 (China) according to standard methods [16]. The treatment degree of the studied samples is determined by equation (1), which describes the relationship between the degree of treatment X, % and the optical density A:

$$A = -0.98X + 1.16. \qquad (2)$$

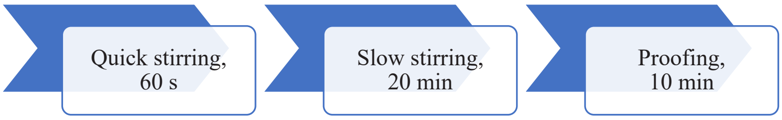


Fig. 2. Stages of the coagulation process

Equation (1) is obtained according to the graduation graph, built on the results of measuring the optical density of model solutions of different degrees of treatment.

In addition, the analysis of waste water samples before and after the experimental studies was carried out by the Central Biochemical Laboratory (Kyiv, Ukraine) according to the indicators specified in the Rules for receiving waste water from subscribers to the sewage system of the city of Kyiv (order No. 1879 dated 12.10.2011).

Statistical processing of the experiment results involved the rejection of results containing gross errors and the identification of correlations between parameters and indicators of the quality of the coagulation process.

Detection of results containing gross errors was performed according to the 3σ rule [15]. According to the rule, the lower x_l and upper x_u limits of the confidence intervals for each indicator of the quality of the coagulation process are calculated according to the formulas:

$$x_l = \bar{x} - 3\sigma,\ x_u = \bar{x} - 3\sigma, \tag{3}$$

where $\bar{x}$ – the average value of the quality indicator obtained as a result of the experiment; σ – the standard deviation calculated by the formula:

$$\sigma = \sqrt{\frac{1}{n}\Sigma\left(x_i - \bar{x}\right)^2}, \tag{4}$$

where n – the number of values that do not cause doubt.

If a questionable value falls outside the confidence interval, it is excluded from the series of measurements.

The tightness of the correlation relationship was estimated by the value of the multiple correlation coefficient R:

$$R = \sqrt{1 - \frac{\Delta_r}{\Delta_{r11}}}, \tag{5}$$

where Δ_r – the determinant of the matrix of paired correlation coefficients; Δ_{r11} – the determinant of the interfactor correlation matrix.

The closer the R value is to 1, the stronger the relationship.

Modeling of the coagulation process and assessment of the adequacy of the obtained models was performed using the STAR system [17]. An equation of the form was chosen as the basic model:

$$y_j = f\left(x_i, \ldots, x_n\right),\ j = 1 \ldots k, \tag{6}$$

where y_j – indicators of the quality of the coagulation process; x_1, ..., x_n – process parameters that have the greatest influence on the degree of extraction of pollutants.

The search for optimal parameters of the coagulation process was performed using the «OPTIMIZ-M» program [18].

3. Results and Discussions

3.1. Results of a priori analysis of the coagulation process parameters. Preliminary experiments were conducted in which the content of coagulants and flocculants varied widely. As a result, the intervals of variation of factors that significantly affect the quality of coagulation treatment were established (Table 1).

Table 1

Intervals of variation of factors that affect the coagulation process

Variation interval	Factors, units and designation				
	Coagulant 1 $Al_2(SO_4)_3$	Flocculant 1 PGMG	Flocculant 2 PGMG acet.	Flocculant 3 P228	Coagulant 2 $FeCl_3$
	ml	ml	ml	ml	ml
	x_1	x_2	x_3	x_4	x_5
x_{min}	0	4	2	2	0
x_{max}	10	10	6	6	10

The obtained results were used to plan the experiment.

3.2. Results of experimental studies. Experimental studies were divided into four stages, at each of which the influence of one of the coagulants and flocculants was investigated. The content of the stages is given in the Table 2. For each stage, a separate FFE plan of 12 experiments was drawn up.

Table 2

Stages of conducting experimental research

Stage No.	Parameters that were studied
1	Coagulant 1 $Al_2\left(SO_4\right)_3$, flocculant 1 (PGMG) and pH
2	Coagulant 2 $FeCl_3$, flocculant 1 (PGMG) and pH
3	Coagulant 1 $Al_2\left(SO_4\right)_3$, flocculant 2 (PGMG acet.) and pH
4	Coagulant 1 $Al_2\left(SO_4\right)_3$, flocculant 3 (P228) and pH

3.2.1. Dependence of the coagulation treatment quality on the type of coagulant. The treatment degree of the studied samples and the change in sediment height for two coagulants, as well as without the addition of a flocculant, were studied. The results are given in the Tables 3, 4.

Table 3

Dependence of the degree of treatment on the type and coagulant dose

Dose and type of coagulant	Treatment degree X, %	
	$Al_2\left(SO_4\right)_3$	$FeCl_3$
without flocculant addition	85	80
the maximum flocculant dose	85	55

Table 4

Dependence of sediment height on the type and coagulant dose

Dose and type of coagulant	Sediment height, mm	
	$Al_2(SO_4)_3$	$FeCl_3$
without flocculant addition	20	6
the maximum flocculant dose	18	1

3.2.2. Dependence of the quality of coagulation treatment on the type of flocculant and the coagulant dose. The results of experimental studies of the influence of the type of flocculant and the coagulant dose on turbidity, the treatment degree and the sediment height are shown in Table 5–7.

Table 5

Dependence of turbidity on the type of flocculant and dose of $Al_2(SO_4)_3$

Dose and type of coagulant	Turbidity, NUT		
	flocculant 1	flocculant 2	flocculant 3
without flocculant addition	1.02	11.7	35.1
1.5 g/dm^3	1.74	4.89	38.8
3.1 g/dm^3	2.45	5.65	14.1

Table 6

Dependence of the treatment degree on the type of flocculant and dose of $Al_2(SO_4)_3$

Coagulant dose	Treatment degree *X*, %		
	flocculant 1	flocculant 2	flocculant 3
without flocculant addition	80	24	66
1.5 g/dm^3	89	54	83
3.1 g/dm^3	85	42	84

Table 7

Dependence of sediment height on flocculant type and dose of $Al_2(SO_4)_3$

Coagulant dose	Sediment height, mm		
	flocculant 1	flocculant 2	flocculant 3
without flocculant addition	6	10	1
1.5 g/dm^3	25	15	10
3.1 g/dm^3	30	20	17

3.2.3. Dependence of the quality of coagulation treatment on the type and dose of flocculant. The results of the study of the influence of the type and dose of flocculant on turbidity, the treatment degree and the sediment height are shown in Tables 8–10.

Table 8

Dependence of turbidity on the type and dose of flocculant

Flocculant dose	Turbidity, NUT		
	flocculant 1	flocculant 2	flocculant 3
20 mg/dm^3	3.15	4.88	1.36
40 mg/dm^3	2.45	5.65	14.1
60 mg/dm^3	1.75	5.51	6.25

Table 9

Dependence of the treatment degree on the type and dose of flocculant

Flocculant dose	Treatment degree *X*, %		
	flocculant 1	flocculant 2	flocculant 3
20 mg/dm^3	40	29	68
40 mg/dm^3	85	42	84
60 mg/dm^3	83	58	79

Table 10

Dependence of sediment height on the type and dose of flocculant

Flocculant dose	Sediment height, mm		
	flocculant 1	flocculant 2	flocculant 3
20 mg/dm^3	20	21	17
40 mg/dm^3	30	20	17
60 mg/dm^3	20	20	20

3.2.4. Statistical processing of experimental data. The limits of the confidence intervals were calculated according to the formula (3) and the standard deviation (4) for the quality indicators of the coagulation process (Table 11).

Table 11

Statistical characteristics

Quality indicator	Limits of the confidence interval		Standard deviation
	x_H	x_B	
turbidity (y_1, NUT)	0	75.75	0.72
treatment degree (y_2, %)	0	100	0.22
sediment height (y_3, mm)	0	31	1.39

The values of the determinants of the matrix of paired correlation coefficients and the value of the multiple correlation coefficient *R* were calculated according to formula (5):

$$\Delta_r = \begin{vmatrix} 1 & 0.722 & 0.1167 \\ 0.722 & 1 & 0.019 \\ 0.1167 & 0.019 & 1 \end{vmatrix} = 0.468;$$

$$\Delta_{r11} = \begin{vmatrix} 1 & 0.019 \\ 0.019 & 1 \end{vmatrix} = 1,$$

$$R = \sqrt{1 - \frac{0.468}{1}} = 0.73.$$

3.3. Optimization of the coagulation process. The mathematical description of the coagulation process was obtained by means of the STAR program based on the results of experimental studies:

$$\begin{cases} y_1 = 91.48 - 2.06x_1 - 88.06x_2 - 1.58x_6, \\ y_2 = 1.95 - 0.16x_1 - 0.76x_2 - 5.6\cdot 10^{-2}x_6, \\ y_3 = -4.98 + 1.25x_1 + 0.37x_2 + 17.05x_6. \end{cases} \quad (7)$$

Standard deviation characteristics for each equation, respectively: $\sigma_1 = \pm 1.7$ NUT, $\sigma_2 = \pm 0.22$ %, $\sigma_3 = \pm 1.4$ mm. Therefore, the obtained mathematical model describes the experimental data quite accurately.

Let's formulate the problem of optimizing the coagulation process: it is necessary to find the coordinates of the optimal point $\left(x_1^{opt}, x_2^{opt}, x_6^{opt}\right)$, taking into account the following restrictions:

$$2 <= x_1 <= 10, \ 1 <= y_1 <= 50,$$

$$4 <= x_2 <= 10, \ 0 <= y_2 <= 2,$$

$$9 <= x_6 <= 10, \ 0 <= y_3 <= 30,$$

if the response surface has the form (7).

The output data and results of the «OPTIMIZ-M» program are shown in Fig. 3.

Comparing the results of cleaning with different coagulants (Fig. 4), it can be seen that the aluminum-based coagulant has higher values of the degree of cleaning even without the addition of flocculants.

Fig. 5 shows that a high treatment degree can be achieved for each of the studied flocculants even without the addition of coagulants, which is explained by the presence of a large amount of suspended substances in wastewater.

In Fig. 6, it can be seen that increasing the flocculant dose does not significantly affect the degree of water treatment.

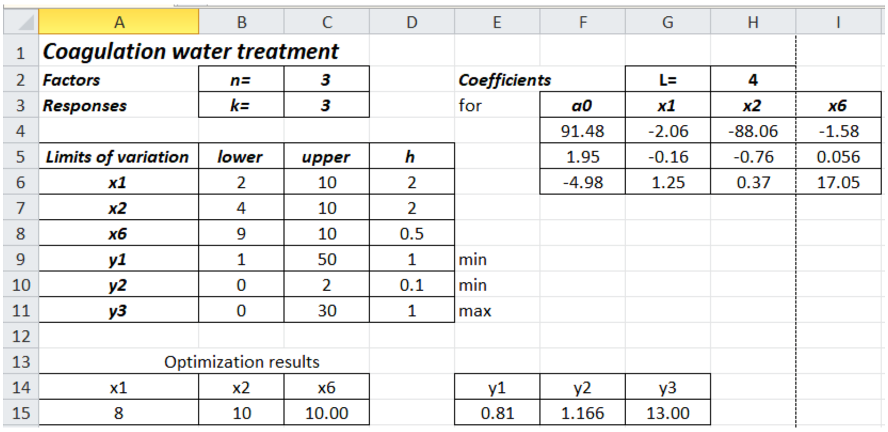
The results of optimization of the process of coagulation treatment of wastewater from leather factories are shown in Fig. 3, allow determining the optimal parameters of the process: pH 10, coagulant dose – 2.5 g/dm^3, flocculant dose – 100 mg/dm^3. The results of solving the optimization problem are confirmed by the graphical dependencies shown in Fig. 4–6 and can be quickly adjusted in real production conditions thanks to the application of the «OPTIMIZ-M» program.

Further development of this study involves expanding the list of coagulants and flocculants.

The conditions of martial law in Ukraine affected the conduct of the research, namely:

1) the difficulty of moving around the country led to a limitation of the number of samples of wastewater from leather enterprises, so not all parameters could be investigated;

2) there was no opportunity to test the proposed technology, it will need to be adapted after the end of the war to real operating conditions.



	A	B	C	D	E	F	G	H	I
1	***Coagulation water treatment***								
2	***Factors***	***n=***	***3***		***Coefficients***		***L=***	***4***	
3	***Responses***	***k=***	***3***		for	***a0***	***x1***	***x2***	***x6***
4						91.48	-2.06	-88.06	-1.58
5	***Limits of variation***	***lower***	***upper***	***h***		1.95	-0.16	-0.76	0.056
6	***x1***	2	10	2		-4.98	1.25	0.37	17.05
7	***x2***	4	10	2					
8	***x6***	9	10	0.5					
9	***y1***	1	50	1	min				
10	***y2***	0	2	0.1	min				
11	***y3***	0	30	1	max				
12									
13		Optimization results							
14	x1	x2	x6		y1	y2	y3		
15	8	10	10.00		0.81	1.166	13.00		

Fig. 3. Optimum conditions for the wastewater treatment process, obtained by means of the «OPTIMIZ-M» program

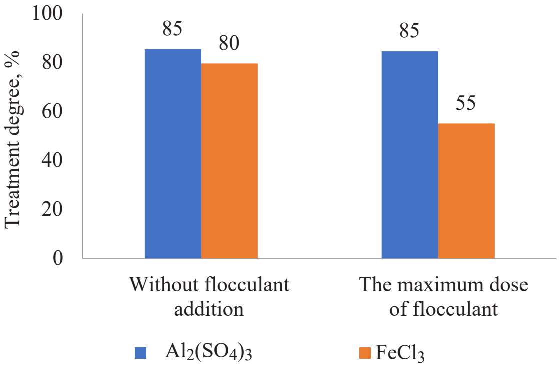


Fig. 4. Wastewater treatment degree depending on the type of coagulant

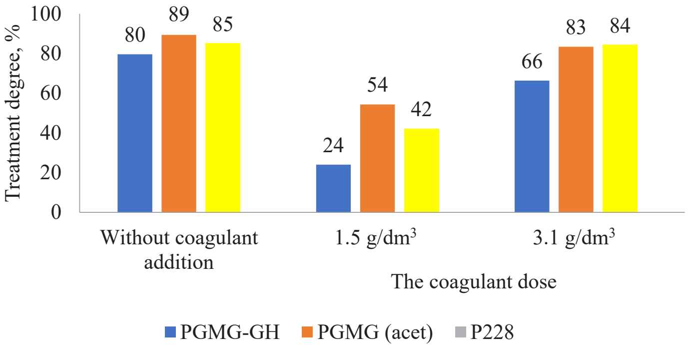


Fig. 5. Wastewater treatment degree depending on the type of flocculant

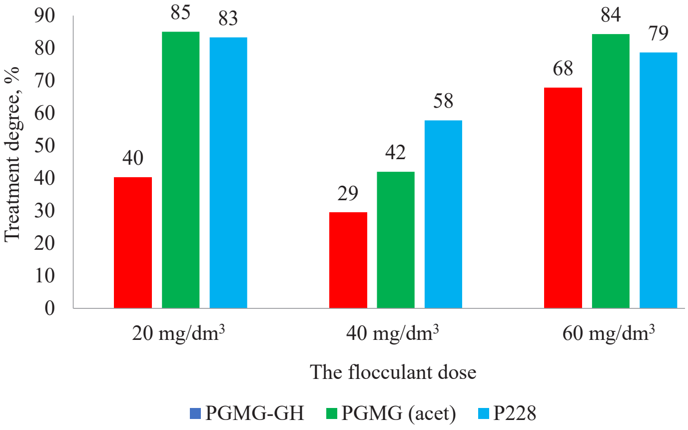


Fig. 6. Wastewater treatment degree depending on the type and dose of flocculant

4. Conclusions

The factors were analyzed and the intervals of variation of factors that have a significant impact on the process of coagulation treatment of wastewater from leather factories were determined.

An experiment plan was drawn up and implemented in order to study the effect of coagulants based on aluminum and ferrum, as well as flocculants on the wastewater treatment quality. The choice of quality indicators of the coagulation process was justified. A statistical analysis of the results of experimental studies was performed: the results containing gross errors were rejected, a correlation analysis of the interdependence of parameters and indicators of the quality of the coagulation process was performed.

Aluminum-based coagulant was found to be more effective than iron-based coagulant. The results of mathematical modeling were used to determine the optimal parameters of the coagulation process: pH 10, coagulant dose – 2.5 g/dm^3, flocculant dose – 100 mg/dm^3.

Conflict of interest

The authors declare that they have no conflict of interest in relation to this study, including financial, personal, authorship, or any other, that could affect the study and its results presented in this article.

Financing

The research was conducted without financial support on initiative topics with State registration numbers: 0124U001966, 0124U002058.

Data availability

Data will be provided upon reasonable request.

Use of artificial intelligence

The authors used artificial intelligence technologies within acceptable limits to provide their own verified data, which is described in the research methodology section.

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