

STUDY OF THE DECOLORIZATION PROCESS OF NATURAL WATERS USING THE METHOD OF OPTIMAL EXPERIMENTAL DESIGN

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Abstract

Color is one of the fundamental indicators of natural water quality, reflecting the presence of organic matter, colloidal systems, and other dissolved or suspended impurities. High color intensity not only deteriorates the organoleptic properties of water but also indicates the presence of humic substances capable of forming toxic compounds during disinfection. Therefore, the development of effective methods for decolorizing natural waters remains a relevant task in modern water treatment. This study investigates the process of natural water decolorization using the method of optimal experimental design. Mathematical modeling and statistical analysis were applied to develop predictive models describing the efficiency of water decolorization by coagulation with preliminary ozonation. The approach enables the evaluation of complex interrelations among technological parameters and the identification of optimal operating conditions. The article details the criteria for selecting factors and response functions, as well as the procedure for conducting experimental studies. The study defines criteria for selecting an experimental design that minimizes the number of trials while maintaining statistical reliability. Based on a three-factor rotatable design, second-order regression equations were obtained, representing experimental-statistical models of water decolorization for technical water supply purposes. A statistical analysis of the adequacy of the developed models was carried out, demonstrating their high predictive capability. The developed models make it possible to forecast the efficiency of the decolorization process and optimize the technological parameters of water treatment to achieve maximum effect with minimal reagent costs.

Keywords: water decolorization, optimal experimental design, coagulation, color, response surface, optimization of water treatment processes.

ИССЛЕДОВАНИЕ ПРОЦЕССА ОБЕСЦВЕЧИВАНИЯ ПРИРОДНЫХ ВОД С ПРИМЕНЕНИЕМ МЕТОДА ОПТИМАЛЬНОГО ПЛАНИРОВАНИЯ ЭКСПЕРИМЕНТА

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Реферат

Цветность является одним из ключевых показателей качества природных вод, свидетельствующим о наличии органических веществ, коллоидных систем и других загрязнений. Высокая цветность не только ухудшает органолептические свойства воды, но и указывает на присутствие гуминовых веществ, способных образовывать токсичные соединения при обеззараживании. В связи с этим разработка эффективных методов обезцвечивания природных вод остается актуальной задачей современной водоподготовки. В статье рассматривается применение методов математического моделирования и оптимального планирования эксперимента для исследования процесса обезцвечивания природных вод. На основе анализа современных публикаций и достижений в данной области показано, что использование математической теории позволяет создавать точные модели процессов водоподготовки, учитывающие влияние множества определяющих факторов. В качестве практического примера рассмотрено планирование многофакторного эксперимента на примере комбинированного метода обезцвечивания поверхностных вод коагулированием с предварительным озонированием. В работе представлены условия выбора методики планирования, позволяющие оптимизировать количество необходимых опытов при сохранении требуемой точности исследований. В статье подробно описаны критерии отбора факторов и функции отклика, а также процедура проведения экспериментальных исследований. В исследовании определены критерии выбора схемы эксперимента, которая минимизирует количество испытаний при сохранении статистической надежности. По результатам проведения трехфакторного ротатбельного эксперимента получены уравнения регрессии второго порядка, которые представляют собой экспериментально-статистические модели процессов подготовки воды для технического водоснабжения. Проведен статистический анализ адекватности разработанных моделей, показавший их высокую прогностическую способность. Разработанные модели позволяют прогнозировать эффективность процесса обезцвечивания и оптимизировать технологические параметры водоподготовки для достижения максимального эффекта при минимальных затратах реагентов.

Ключевые слова: обезцвечивание вод, оптимальное планирование эксперимента, коагулирование, цветность, поверхность отклика, оптимизация процессов водоподготовки.

Introduction

Solving most problems in chemistry and chemical engineering related to the treatment of natural waters traditionally requires extensive and resource-intensive experimental research. In this context, methods of optimal experimental design are of particular importance, as they allow the mathematical framework to be applied not only during the processing of experimental data but also at the stage of experiment planning and organization [1]. Mathematical modeling has become an essential component of modern environmental research. The environment, represented

by both natural and anthropogenic systems, is a complex set of material objects, processes, and interactions between them [2–4]. Traditional methods of water decolorization include coagulation, adsorption, membrane technologies, and chemical oxidation. Along with conventional coagulation-flocculation techniques employed at municipal water treatment plants (using aluminum and iron salts), oxidizing and sorption methods have been gaining wider application [5, 6]. The efficiency of each process is determined by numerous factors, including the coagulant dose, pH, alkalinity, temperature, and contact time. The empirical determination

of optimal operating conditions requires considerable time and material resources. Modern approaches based on the study of the mechanisms of complex physicochemical processes and the optimization of multicomponent systems make it possible to construct adequate models of water treatment technologies that take into account the combined influence of multiple variables [7, 8].

The objective of the present study was to investigate the main influencing factors and modeling conditions of the surface-water treatment process, as well as to develop experimental-statistical models capable of predicting the removal efficiency of pollutants responsible for color formation. The process under investigation involved the decolorization of surface water by coagulation preceded by ozonation.

Analysis of the Principles and Practical Implementation of Optimal Experimental Design in Water Treatment Modeling

Optimal experimental design is a branch of mathematical statistics aimed at maximizing the information gained from experiments while minimizing the number of trials required. The most widely used designs in water treatment research include:

- d-optimal designs, which minimize the variance of regression coefficients estimates in the model, an especially valuable feature when only a limited number of runs is feasible;
- i-optimal (integrated optimal) designs, which minimize the average variance of prediction throughout the experimental domain, making them particularly suitable for process optimization tasks;
- Response Surface Methodology (RSM), which provides a framework for constructing mathematical models that relate input factors to output responses and for determining optimal process conditions.

Multifactorial experimental design is extensively used in scientific and engineering studies as an effective tool for planning and analyzing experimental data [9–11]. The design procedure determines the required number of experiments and the conditions under which they must be carried out in order to achieve the desired accuracy and reliability of results. Variables that influence the studied process are referred to as factors. In relation to water purification processes, such factors typically include the coagulant or flocculant dosage, duration of the treatment stage, filtration rate, temperature, and other parameters. A key requirement for each factor is its controllability – the ability to maintain the prescribed level during the experiment. Additional criteria for selecting factors include their mutual independence, unambiguous influence on the response variable, and physical relevance to the studied process.

The efficiency of the technological process is assessed using one or more quantities called response functions. The effectiveness of a technological process is assessed using one or several quantities referred to as response functions. In water treatment, typical response functions include the degree of decolorization. Optimal experimental design methods make it possible to develop reliable mathematical representations of technological processes even when the underlying mechanisms are not fully known. Mathematical models obtained in this way are commonly referred to as experimental-statistical models.

Such models are often expressed in polynomial form: $Y = f(X_1, X_2, X_3, \dots, X_n)$, where Y is the response function (quantitatively describing process efficiency) and $X_1, X_2, X_3, \dots$ are the independent factors (process variables). The advantage of this mathematical representation lies in its ability to describe the influence of individual factors on the response function, to quantify the response under specific combinations of factor levels, and serve as a basis for process optimization and simulation [12].

A review of existing research on optimal experimental design in water treatment demonstrates its major advantages and outlines promising directions for further study. For example, the application of filtration process modeling has been used for optimizing existing treatment facilities and for evaluating the performance of local filtration materials, coagulants, and flocculants under surface water conditions [13]. Other studies have successfully applied mathematical modeling for the removal of iron compounds [14], and for multicomponent ion exchange processes on mixed cation exchangers (H^+ , Na^+) [15, 16]. The analysis confirms the high effectiveness of optimal experimental design methods for investigating and optimizing natural water decolorization processes. In particular, the response surface methodology (RSM) was employed to remove color and turbidity from surface waters and to reduce chemical oxygen demand

(COD) using a combined coagulant based on flaxseed and aluminum-potassium alum [17]. In those experiments, the coagulant dose (1.5–2.5 g/dm³), pH (3.5–7.0), and mixing time (38–40 minutes) were varied. Optimization yielded a chromaticity removal efficiency of 97.75 % at pH 7.0, a coagulant dose of 2.5 g/dm³, and a mixing time of 40 minutes.

Multifactorial Process of Decolorization of Natural Surface Waters

Surface water sources possess several advantages for use in technical water supply systems, primarily due to their relatively low mineralization and minimal hardness compared to groundwater. However, their effective utilization in industrial processes requires the development of optimal pretreatment methods aimed at removing organic impurities responsible for color formation. Among reagent-based treatment techniques, coagulation occupies a central position and remains one of the most widely applied methods in water treatment practice. The efficiency of coagulation during the treatment of highly colored but low-turbidity waters largely depends on the colloidal nature of the organic compounds present, which are predominantly of humic origin. These compounds, exhibiting weakly acidic properties, form stable, highly dispersed systems characterized by a negative surface charge. It should be noted that the decolorization process is strongly influenced by the qualitative composition of the organic matter. The presence of humic acids promotes higher removal efficiency, while the dominance of fulvic acids requires substantially greater reagent doses and complicates the overall technological process. A promising approach for enhancing coagulation efficiency involves the application of pre-oxidation methods [18]. The introduction of oxidizing agents facilitates destabilization of colloidal systems by destroying the hydration shells surrounding organic particles, thereby increasing the efficiency of subsequent coagulation. In addition, preliminary oxidation – particularly chlorination and ozonation – provides simultaneous removal of iron and manganese compounds, which are frequently present in surface waters. Contemporary studies confirm that combined treatment using oxidants followed by coagulation significantly improves color removal efficiency [19]. In this regard, the experimental investigation of optimal parameters for the combined application of oxidants and coagulants represents considerable practical interest for improving water treatment technologies intended for technical water supply systems.

The object of the present research was the Mukhovets River, a representative surface water body in the south-western region of Belarus. The study focused on evaluating the influence of ozone dose, coagulant dose, and flocculant dose on the removal efficiency of organic compounds determining the color and oxidizability of the water. The investigation employed a rotatable central composite experimental design, the main characteristics of which are presented in [7, 19].

The process of developing the mathematical model included the following stages: experimental design planning; execution of laboratory and field experiments on the selected water source; verification of experimental reproducibility; derivation of a mathematical model of the process with statistical testing of the regression coefficients for significance; evaluation of model adequacy using statistical criteria. To determine the optimal operating region, an exploratory experiment was first conducted, followed by a rotatable second-order design, in which the selected factors were varied at five levels. Reproducibility of the results was assessed using the Cochran criterion, the significance of the regression coefficients was determined by the Student's test, and the adequacy of the mathematical model was verified using the Fisher criterion at a 5 % significance level.

Investigation and optimization of the technological regime of surface water bleaching by coagulation with pre-ozonation

Preliminary experiments demonstrated that the introduction of ozone prior to coagulation increases the overall decolorization efficiency by an average of 10–15 %. The experimental work on water decolorization was conducted using surface water from the Mukhovets River, whose quality characteristics are representative of surface water bodies in the southern regions of the Republic of Belarus.

For the reagent treatment, analytically pure aluminum sulfate ($Al_2(SO_4)_3 \cdot H_2O$) was used as the coagulant, while polyacrylamide (PAA) served as the flocculant. The initial color value of the raw water was 55 degrees, which was taken as the baseline quality indicator [20]. The effectiveness of color removal was evaluated based on the results of

spectrophotometric measurements, which allowed quantification of the reduction in apparent color after treatment. The obtained data served as the basis for subsequent mathematical modeling and optimization of process parameters.

Design and Conduct of the Experiment at the Research Facility and Construction of the CCRD Matrix

To determine the optimal operating region, a preliminary experimental search was performed, followed by the implementation of a rotatable second-order central composite design (CCRD) within the identified range of factor variability. In this design, the factors

$$Y = b_0 + b_1 \cdot X_1 + b_2 \cdot X_2 + b_3 \cdot X_3 + b_{12} \cdot X_1 \cdot X_2 + b_{13} \cdot X_1 \cdot X_3 + b_{23} \cdot X_2 \cdot X_3 + b_{123} \cdot X_1 \cdot X_2 \cdot X_3 + b_{11} \cdot X_1^2 + b_{22} \cdot X_2^2 + b_{33} \cdot X_3^2; \quad (1)$$

where $b_0, b_1, b_2, b_3, b_{12}, b_{13}, b_{23}, b_{123}, b_{11}, b_{22}, b_{33}$ are the regression coefficients, and

X_1, X_2, X_3 are the coded variables associated with their respective physical quantities (x_1, x_2, x_3) through the following transformations

$$X_1 = \frac{x_1 - x_{01}}{\Delta x_1}; X_2 = \frac{x_2 - x_{02}}{\Delta x_2}; X_3 = \frac{x_3 - x_{03}}{\Delta x_3}, \quad (2)$$

where

x_{01} – coordinates of the center point of the experimental design;

Δx – the variation interval for each factor;

Y – the response function, representing the measured residual color of the treated water.

Calculation of Regression Coefficients and Corresponding Variance Estimates

Within the framework of the rotatable central composite design (CCRD), the regression coefficients and their corresponding variance estimates were calculated to determine their statistical significance [1, 2]. Table 1 presents the characteristics of the CCRD and the values of the design constants B, C, and A.

Table 1 – Characteristics of the CCRD and Design Constants

Number of factors n	3
Total number of experiments N	20
Number of central point experiments N_0	6
Constant B	0,8571429
Constant C	1,4285714
Constant A	0,4537037

Based on the results of the experimental studies, the summations and regression coefficients were calculated, as summarized in Table 2.

Table 2 – Summation Values and Calculated Regression Coefficients

Summation Term				Regression Coefficient			
S_0	150,40	S_{23}	-4,80	b_0	4,25	b_{23}	-0,57
S_1	-45,42	S_{11}	152,86	b_1	-3,24	b_{11}	3,22
S_2	6,55	S_{22}	104,32	b_2	0,47	b_{22}	0,33
S_3	-24,38	S_{33}	117,58	b_3	-1,74	b_{33}	1,12
S_{12}	4,40	S_{123}	1,40	b_{12}	0,52	b_{123}	0,03
S_{13}	-4,80			b_{13}	-0,57		

In accordance with the principles of rotatable experimental design, a coefficient b_i is considered statistically significant if its calculated value satisfies the condition: $|b_i| > s_{b_i} \cdot t$, where t is the critical value of Student's t-test [1, 2]. If this condition is not met, the coefficient is regarded as insignificant, and the corresponding term may be excluded from the regression model. Table 3 summarizes the variance estimates for determining the significance of the regression coefficients.

were varied at five levels corresponding to the coded variable values +1; -1; +1,68; -1,68 and 0, ensuring statistical rotatability and efficient estimation of quadratic effects.

Based on prior information, it was established that the residual color of treated water, which represents the efficiency of the decolorization process is primarily influenced by three key factors:

- the mass concentration of the coagulant, mg/dm³;
- the mass concentration of the flocculant, mg/dm³;
- the mass concentration of ozone, mg/dm³.

Accordingly, the mathematical description of the process was expressed in the form of a second-order regression equation

Table 3 – Variance Estimates for the Regression Coefficients

$S_{b_0}^2 =$	0,038	$S_{b_0}^2 \cdot t =$	0,499
$S_{b_1}^2 =$	0,014	$S_{b_1}^2 \cdot t =$	0,302
$S_{b_2}^2 =$	0,020	$S_{b_2}^2 \cdot t =$	0,361
$S_{b_3}^2 =$	0,013	$S_{b_3}^2 \cdot t =$	0,291

Table 4 presents the verification of the significance of the regression coefficients according to Student's t-test.

Table 4 – Verification of Regression Coefficient Significance

$ b_0 =$	4,25	>	0,499	Significant
$ b_1 =$	3,24	>	0,302	Significant
$ b_2 =$	0,47	>	0,302	Significant
$ b_3 =$	1,74	>	0,302	Significant
$ b_{12} =$	0,52	>	0,361	Significant
$ b_{13} =$	0,57	>	0,361	Significant
$ b_{23} =$	0,57	>	0,361	Significant
$ b_{11} =$	3,22	>	0,291	Significant
$ b_{22} =$	0,33	>	0,291	Significant
$ b_{33} =$	1,12	>	0,291	Significant
$ b_{123} =$	0,03	<	0,361	Not significant

Verification of Variance Homogeneity (Reproducibility of Experiments)

To verify the reproducibility of the experimental results, the Cochran criterion (G_p) was used, which is defined as the ratio of the maximum variance to the sum of all variances within a series of parallel experiments. For each series of parallel runs, the arithmetic mean value of the response function was calculated, followed by the variance estimate for that series.

The hypothesis of variance homogeneity is not rejected if the experimentally determined value of the Cochran criterion does not exceed its tabulated value [9]. The corresponding tabulated values of the Cochran criterion were adopted for a confidence probability of $P = 0,95$, under which the reproducibility hypothesis is considered valid. The quantity ($p = 1 - P$) represents the significance level. To determine the value of G , it is necessary to know the total number of variance estimates (N) and the degrees of freedom (f) associated with each of them, where $f = k - 1$, and k is the number of factors considered. Table 5 presents the experimental response function values (y_j^0) and the corresponding variance estimates (s_j^2) for reproducibility testing at the central points of the experimental plan.

Table 5 – Estimation of the variance of reproducibility of experiments in the center of the plan

Center-point experiments					Variance Estimate
Experiment No. j	y_j^3	$(y_j^3 - \bar{y})$	$(y_j^3 - \bar{y})^2$	s_j^2	
15	3,00	-0,72	0,51	0,257	$\max s_j^2$
16	4,00	0,28	0,08	0,040	
17	3,50	-0,22	0,05	0,023	
18	4,20	0,48	0,23	0,117	
19	3,60	-0,12	0,01	0,007	
20	4,00	0,28	0,08	0,040	
$\Sigma 6$	22,30	0,00	0,97	0,484	$\sum_{j=1}^N s_j^2$

The average value of the response function was calculated according to the expression

$$\bar{y} = \frac{1}{N_0} \cdot \sum_{j=1}^{N_0} y_j^3 = \frac{1}{6} \cdot 22,3 = 3,72. \quad (3)$$

where y_j^3 – experimental response function values; j – experiment number; N_0 – number of experiments at the center of the design.

The calculated value of the Kohren criterion G_p was determined by the formula

$$G_p = \frac{\max s_j^2}{\sum_{j=1}^N s_j^2} = \frac{0,257}{0,484} = 0,530. \quad (4)$$

The corresponding tabulated value of the Cochran criterion was taken from [9] for the following parameters: $P = 0,95$; $N = 6$; $f = k - 1 = 3 - 1 = 2$, where k is the number of selected factors. Thus, the tabulated value is: $G_t = 0,616$.

The condition $G_p \leq G_t$ is fulfilled: $0,530 < 0,616$, therefore, the experiments can be considered reproducible.

The variance estimate for a series of parallel experiments s_y^2 was calculated using the following formula

$$s_y^2 = \frac{1}{N_0 - 1} \cdot \sum_{j=1}^{N_0} (y_j^3 - \bar{y})^2 = \frac{1}{6 - 1} \cdot 0,97 = 0,194. \quad (5)$$

$$s_{ad}^2 = \frac{\sum_{j=1}^N (y_j^3 - y_j^p)^2 - s_y^2 \cdot (N_0 - 1)}{f_{ad}} = \frac{5,060 - 0,194 \cdot (6 - 1)}{5} = 0,818. \quad (6)$$

The number of degrees of freedom associated with this adequacy variance estimate was defined as

$$f_{ad} = N - \frac{(n+2) \cdot (n+1)}{2} - (N_0 - 1) = 20 - \frac{(3+2) \cdot (3+1)}{2} - (6 - 1) = 5, \quad (7)$$

where n is the number of factors.

The calculated value of the Fisher criterion was obtained as

$$F_p = \frac{\max(s_{ad}^2, s_y^2)}{\min(s_{ad}^2, s_y^2)} = \frac{0,818}{0,194} = 4,216. \quad (8)$$

$$Y = 4,25 - 3,24X_1 + 0,47X_2 - 1,74X_3 + 0,52X_1X_2 - 0,57X_1X_3 - 0,57X_2X_3 + 3,22X_1^2 + 0,33X_2^2 + 1,12X_3^2. \quad (9)$$

To convert the regression equation from coded variables to physical units, the following relationships (based on Eq. (2)) were applied:

$$X_1 = \frac{x_1 - x_{01}}{\Delta x_1} = \frac{k - 20}{10} = 0,1k - 2;$$

$$X_2 = \frac{x_2 - x_{02}}{\Delta x_2} = \frac{f - 1}{0,5} = 2f - 2;$$

This variance value was determined with the number of degrees of freedom corresponding to $f = N_0 - 1 = 6 - 1 = 5$.

Verification of the Adequacy of the Regression Equation

After obtaining the regression equation, it is necessary to verify its adequacy, i.e., its ability to accurately describe the response surface of the studied process. The adequacy of the regression model was verified using the Fisher criterion (F -test).

Table 6 – Variance Estimates for Determining the Adequacy of the Regression Equation

Experimental Response y_j^3	Calculated Response y_j^p	$(y_j^3 - y_j^p)$	$(y_j^3 - y_j^p)^2$
4,30	3,79	0,51	0,264
9,80	9,56	0,24	0,060
3,00	2,95	0,05	0,003
6,80	6,43	0,37	0,138
10,70	10,37	0,33	0,109
14,50	13,85	0,65	0,419
12,30	11,62	0,68	0,457
13,00	12,82	0,18	0,032
8,30	7,89	0,41	0,168
19,50	18,79	0,71	0,504
6,00	5,97	0,03	0,001
4,60	4,40	0,20	0,041
4,50	4,49	0,01	0,000
10,80	10,34	0,46	0,212
3,00	4,25	-1,25	1,554
4,00	4,25	-0,25	0,061
3,50	4,25	-0,75	0,557
4,20	4,25	-0,05	0,002
3,60	4,25	-0,65	0,418
4,00	4,25	-0,25	0,061
			5,060

The adequacy variance s_{ad}^2 was determined using the following expression

According to reference [9], the corresponding tabulated value of the Fisher criterion is: $F = 5,05$. Since the condition $F_p \leq F$ is satisfied ($4,216 < 5,05$), the obtained regression equation is considered adequate and provides a reliable description of the response surface.

$$X_3 = \frac{x_3 - x_{03}}{\Delta x_3} = \frac{g - 10}{5} = 0,2g - 2.$$

Substituting these expressions into the final regression equation yields the model in physical terms

$$C = f(k, f, g):$$

$$C = 29,50 - 1,6k - 1,5f - 0,788g + 0,104kf - 0,0114kg - 0,228fg + 0,0322k^2 + 1,32f^2 + 0,0448g^2, \quad (10)$$

where C – color intensity of water (color units), grad;
 k – coagulant dose D_k (mg/dm³);
 f – flocculant dose D_f (mg/dm³);
 g – ozone dose D_{oz} (mg/dm³).

Using this equation (10), it is possible to predict the performance efficiency of the water decolorization system for any given operational regime of coagulation treatment with preliminary ozonation. Figure 1 shows a graph of the residual color of water corresponding to the obtained model, depending on the ozone dose at different coagulant doses.

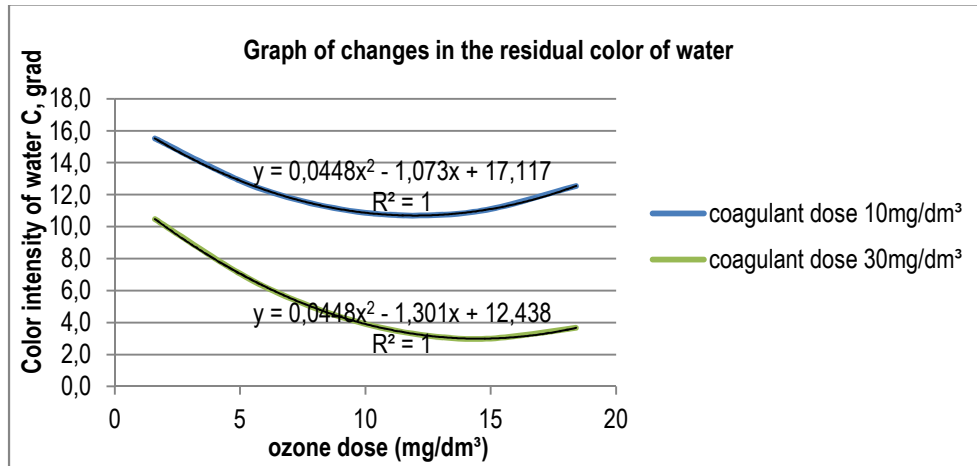


Figure 1 – Influence of ozone dose on the water decolorization process at different coagulant doses (flocculant dose = 0,75 mg/dm³)

Analysis of the regression model (Eq. 10) based on the determination of the extremum condition $C = f(k, f, g) \rightarrow \min$ with respect to the influencing factors D_k , D_f , and D_{oz} , showed that the minimum residual color of treated water is achieved under the following optimal conditions: coagulant dose – 26,12 mg/dm³, flocculant dose – 0,75 mg/dm³, ozone dose – 14 mg/dm³.

Mathematical processing of the experimental results indicates that all three investigated factors significantly influence the efficiency of water decolorization. However, their impact strength decreases in the following order: coagulant dose > ozone dose > flocculant dose.

Conclusions

Based on the conducted experimental studies simulating the natural water treatment process, the main influencing factors determining the efficiency of removing color-causing impurities from surface waters for technical water supply purposes were identified and analyzed. Using the method of optimal experimental design, an experimental-statistical model of the decolorization process by coagulation with preliminary ozonation was developed. The obtained regression equation describes the combined influence of ozone, coagulant, and flocculant doses on the residual color of treated water. The model enabled the determination of optimal process parameters at which the maximum decolorization efficiency is achieved under the given conditions.

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Material received 24.10.2025, approved 09.11.2025, accepted for publication 10.11.2025