LOW WASTE TECHNOLOGY FOR MINE WATERS TREATMENT USING LIME AND ALUMINUM COAGULANTS

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Abstract. In this paper the process of reagent desalination of mineralized mine waters was studied. The peculiarity of mine waters in many regions of Ukraine is that, along with hardness ions, they also contain sulphates in fairly high concentrations. Therefore, the task of desalination of mineralized waters consists in effective removal of sulphates along with softening of the solution. For effective purification of water from sulphates and hardness ions, 5/6 aluminum hydroxychloride ($Al_2(OH)_5Cl$) and sodium tetrahydroxoaluminate ($Na[Al(OH)_4]$) were used during liming. A significant increase in efficiency of the treatment process was achieved when the solution was acidified with carbon dioxide after treatment with reagents. The directions of processing of the formed sediments as part of building materials have been determined. Complex processing of the generated waste in the process of water treatment allows creating a low-waste technology for the purification of mineralized water.

Keywords: mine water, softening, aluminum coagulant, sulphate, hardness ion.

Introduction

Fresh water is the basis of human existence: its absence is incompatible with life and poor-quality water (salty or with toxic substances, or infected with pathogenic microorganisms) inhibits or completely excludes the normal development of countries and civilizations. Experts predict that in 2030, 47% will suffer from a lack of fresh water, and by 2050, this problem will affect 2/3 of world's population. Therefore, the necessary quantity and proper quality of fresh water resources determine the development of the economy and health of all countries without exception.

Pollution and irrational water use lead to a reduction of fresh water supplies. This is especially noticeable in industrial regions, where a large amount of water is used for production needs, and large volumes of wastewater are discharged into natural bodies [1,2]. The construction and operation of coal industry enterprises leads to a significant pollution of surface, underground and soil sources of water supply. During mining, water in general and mine water in particular is an integral part of almost all mining operations [3]. Wastewater discharged from various activities related to the coal industry degrades fresh water quality and creates the potential for surface and groundwater contamination. In the regions of Ukraine, up to 1 billion m^3 of mine water is pumped out of the mine and discharged into surface water bodies annually [4].

Mine water is both a renewable resource and a potential threat to the environment. Such water can be used for various needs, for example, for industrial purposes as technical water, or in agriculture for irrigation, and even for drinking purposes after appropriate purification [5]. Due to the appropriate methods of water resources management and the introduction of water purification technologies, it is possible to provide low-water regions with water for technical and drinking needs, which is expedient in both ecological and economic terms [6].

The constant transition of mining operations to deeper horizons leads to an increase in the volume and degree of contamination of mine waters with various substances. In addition to contamination with mechanical and organic impurities, mine waters are characterized by high mineralization; 60–75% of mine waters have mineralization over 1000 mg/L, up to 20% – 3000 mg/L or more [7,8]. The use of such waters in industry is impossible without proper treatment, and their discharge is a real danger of pollution for surface and underground waters [9].

Today, in Ukraine the most widespread technological schemes of existing and planned treatment facilities for mine waters mainly provide the removal of suspended substances, partially – organic pollutants, disinfection, reagent treatment and clarification of discharged water. The overall concentration of dissolved mineral compounds, especially hydrocarbonate, sulphate, and chloride ions, remains relatively stable during the treatment of mine waters. To remove the mineral content from such waters, demineralization technologies are essential. The quality standards for treated mine water, whether it's being discharged into water bodies or used elsewhere, require the extensive utilization of diverse purification methods and technologies. One of the most common methods for desalination of mineralized waters is distillation (evaporation). A significant disadvantage of using distillation facilities is the formation of scale on the heating elements, which affects the heat transfer and operation of the equipment. To prevent the scale formation, it is necessary to use additional reagents. Also, such equipment requires high energy consumption, which is not always economically beneficial [10].

In recent years, the reverse osmosis method has been widely used for the demineralization of mine waters. However, the application of this method for the purification of highly mineralized waters requires pretreatment of water that allows increasing the productivity of the process by minimizing the pollution, scale formation, degradation and destruction of membranes [11]. When using a complex system consisting of forward osmosis and reverse osmosis (RO), it is possible to achieve a purification efficiency of more than 80% with obtaining quality water suitable for discharge. It has been established that such system is an effective alternative to eliminate the existing traditional pretreatment process [12]. However, these technologies are quite expensive.

A promising direction in the processing and removal of excess salts from mine waters is the use of ion exchange. However, today, despite the great prospects of comprehensive processing of mineralized waters by this method, the technology has not been widely used. Ion exchange allows achieving a great depth of salt removal, but the cost of water desalination increases with increasing mineralization; at the same time, the depth of water desalination decreases. Thus, the ion-exchange resin (or sorbent) must be regenerated. Therefore, ion exchange desalination is used mainly for waters with a degree of mineralization of less than 2000 mg/L and a total

content of chlorides and sulphates of no more than 5 mg-eq/L [13,14].

With increasing depth, hydrogencarbonate waters become hydrogencarbonate-sulphate and sulphate-hydrogencarbonate. This is explained by the fact that the formation of the chemical composition of deep mines is influenced exclusively by oxidizing processes, which contribute to the accumulation of sulphate ions. Sulphate ions are not considered toxic; however, they contribute to the salinization of freshwater bodies and are therefore considered a negative environmental factor [15]. The presence of high concentrations of sulphate ions can lead to problems related to aggressiveness of such waters to concrete and corrosion of pipes [16]. Reducing corrosion processes requires the development of corrosion inhibitors, which further complicates and makes the technology more expensive. Because of this, the presence of sulphate ions in water is a serious environmental problem in many countries where mineral extraction takes place on a large scale [17].

The most common method of sulphate removal is electrocoagulation [18]. However, the electrocoagulation process is associated with high current consumption, which leads to higher electricity consumption and significant costs in economic terms. To eliminate this shortcoming, studies have recently been conducted on the possibility of operation of electrocoagulation installations using solar energy [19,20]. Although the new methods show their potential to significantly reduce sulphate ions, they need to be tested on an industrial scale to determine overall effectiveness. Reagent methods are quite cheap and simple, which contributes to their widespread implementation. It is important to select a reagent for effective water desalination [21,22].

The purpose of the work was to create low-waste water desalination technology and to determine the effectiveness of mine water demineralization processes by the liming method, depending on the type of aluminum coagulant, the ratio of reagents, and the process time.

Experimental
Materials

Water from Zahidno-Donbaska mine were used as the initial solution for investigation. The total hardness of the water was 31.0 mg-eq/L; sulphates content was 1210.0 mg/L; chlorides – 105.0 mg/L; nitrates – 10.6 mg/L; pH –7.27, as described in Instruments section. Calcium oxide (CaO) suspension was used for liming. Sodium tetrahydroxoaluminate and

aluminum hydroxychloride (AHC) were used to precipitate impurities.

Instruments

The water thermostat TW-2.03 Elmi was used to ensure proper water treatment conditions. Ashless cellulose filter paper – Blue Ribbon was used to separate the sediment from water. The sulphate and chloride ions concentration, hardness and alkalinity, as well as mineralization of the water were studied using a universal ionometer I-160 MI (chlorine-selective electrode ELIS-131Cl, sulphate-selective electrode XC-SO4-001, laboratory pH electrode ES-10601/4). Water hardness was determined by titration with Trilon B. Aluminum was determined by spectrophotometry (ULAB 108 UV Spectrophotometer). The normal density, hardening time, compressive strength of the cement were investigated using standard equipment according to generally accepted methods [23].

Water treatment procedure

The suspension of CaO was added to water samples and mixed thoroughly to completely dissolve the lime. Then the calculated volume of sodium tetrahydroxoaluminate or 5/6 AHC was added at constant stirring. The obtained suspension was kept in a thermostat at 40ºC during 2 h. The increase in temperature allows to accelerate the process. A longer time is required (3-4 hours) to conduct the process at 20ºC. After this, the suspension was kept at room temperature during 4 h for precipitation and was filtered. To reduce the hardness and alkalinity of the water, CO_2 gas was blown through it, resulting in the formation of precipitate. Then the treated water was filtered through a filter. The filtrate was collected and analyzed for the content of sulphate and chloride ions, hardness and alkalinity and mineralization (Figure 1).

The degree of sulphate anions removal (A_1) and the degree of softening (A_2) of water after the treatment with 5/6 AHC and sodium tetrahydroxoaluminate were calculated according to Eq.(1).

$$A_{1(2)} = \frac{c_{0(1,2)} - c_{f(1,2)}}{c_{0(1,2)}} \qquad (1)$$

where, C_o is concentration of hardness and sulphate anions in the initial water, mg-eq/L;

C_f is concentration of hardness and sulphate anions in the treated water, mg-eq/L.

Statistical analysis procedure

Water treatment experiments were performed according to a statistical 23-factorial design as shown in Table 1. The variables studied were the dose of AHC or sodium tetrahydroxoaluminate depends on the stoichiometry (X_1) and the dose of lime from stoichiometry temperature (X_2), and the removal of sulphates with the application of AHC and lime (Y_1), the removal of sulphates with the application of sodium tetrahydroxoaluminate and lime (Y_2), the effectiveness of softening of water during the application of AHC and lime (Y_3) and the effectiveness of softening of water during the application of sodium aluminate and lime (Y_4) were investigated. During the experiments the individual and combined effects of X_n were investigated. The model of the treatment process is presented as a second-order polynomial (Eq.(2)).

$$Y = a_0 + a_1X_1 + a_2X_2 + a_3X_1X_2 + a_4X_1^2 + a_5X_2^2 \qquad (2)$$

where, Y is response factor;
a_0-$a_{1,2,3}$ is regression coefficients.
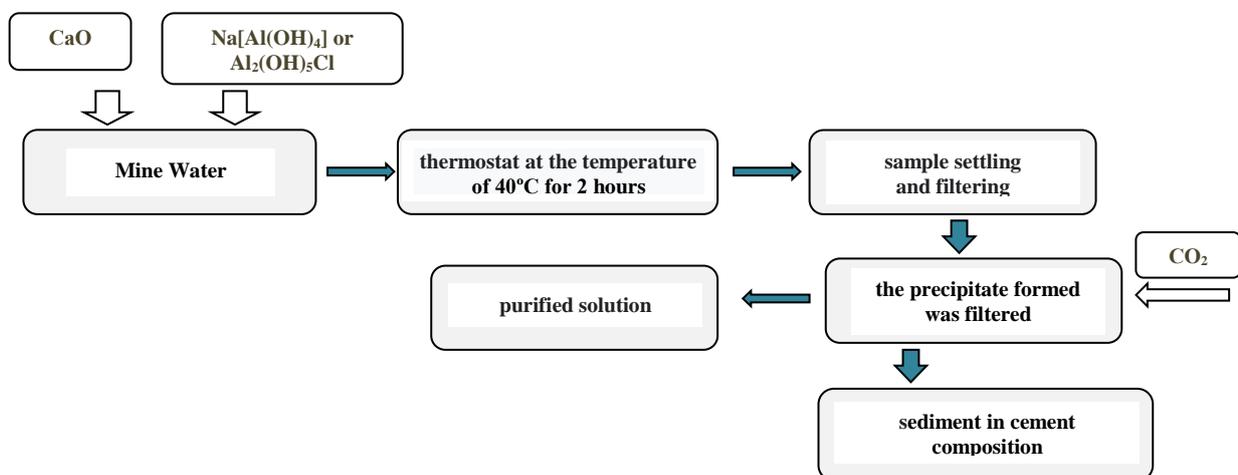
Figure 1. Process diagram mine waters treatment using lime and aluminum coagulants.

The coefficients of the second-order polynomial were calculated through the multiple regression analysis and the analysis of variance was performed using MINITAB 17 software. The resulting Eqs.(3-6) were used to visualize the response surfaces in MATLAB software.

$$Y_1 = + 43.78 + 4.438 \cdot X_1 - 21.975 \cdot X_2 + 48.75 \cdot X_1 \cdot X_2 - 14.233 \cdot X_1^2 + 0.16667 \cdot X_2^2 \qquad (3)$$

$$Y_2 = + 69.208 + 4.7 \cdot X_1 + 9.0833 \cdot X_2 - 1 \cdot X_1 \cdot X_2 + 0.5 \cdot X_1^2 + 0.05 \cdot X_2^2 \qquad (4)$$

$$Y_3 = + 258.27 - 52.295 \cdot X_1 - 288.34 \cdot X_2 + 74.324 \cdot X_1 \cdot X_2 - 3.49 \cdot X_1^2 + 87.25 \cdot X_2^2 \qquad (5)$$

$$Y_4 = + 37.691 + 41.5 \cdot X_1 - 7.083 \cdot X_2 + 19 \cdot X_1 \cdot X_2 - 16.1 \cdot X_1^2 - 0.01 \cdot X_2^2 \qquad (6)$$

Utilization of sediments in the composition of cement

To develop the possible ways for utilization of the formed sediment in the building industry, it was dried in a laboratory oven at 200ºC to a constant mass. Then, the influence of the formed sediment on Portland cement PC I-500 was investigated. The effect of sediment on the properties of cement was evaluated according to the following characteristics: normal density, hardening time, compressive strength. The cement was prepared and the mineralogical composition was the following, wt.%: C3S – 57.10, C2S – 21.27, C3A – 6.87, C4AF – 12.19. All characteristics were determined by standard techniques [24].

Results and discussion

Previous studies of reagent softening of water showed that each type of water requires a careful selection of reagents, depending on its composition [25,26]. Therefore, the efficiency of Zahidno-Donbaska's mine water desalination was determined in the work and math modeling was carried out for faster and effective selection of the dose of reagents, as well as methods of disposal of the sediments as part of building materials were considered.

The consumption of lime and aluminum coagulant was determined from the composition of the sediment given in reactions (Eqs.(7,8)), which show the formation of sediment $3CaO \cdot Al_2O_3 \cdot 3CaSO_4$. With a stoichiometric dose of lime, the sulphate content in water decreased from 1210 to 150 mg/L, for a 20% excess of lime, with an increase in the dose of sodium tetrahydroxoaluminate, the degree of removal of

sulphates from water increases from 84% to almost 90%. With the application of stoichiometric dose of lime and 20, 40, 60 and 80% excess sodium tetrahydroxoaluminate, the maximum degree of removal of sulphate ions is 87.7% and is observed at 80% excess of coagulant.

The efficiency of sulphates removal from water increases with an increase in the dose of lime and coagulant. The disadvantage of the process is that, under these conditions, a significant amount of alkali enters the water, which causes an increase in the alkalinity of the water.

$$3Na_2SO_4 + 6CaO + 2Na[Al(OH)_4] = 3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \downarrow + 8NaOH \qquad (7)$$

When treating water with carbon dioxide, hydrated alkalinity changes to total that generally does not contribute to the demineralization process. In addition to sodium tetrahydroxoaluminate, any other aluminum coagulant with high basicity can be used together with lime to precipitate sulphates. In the processes of water purification, the use of AHC has a number of advantages. With their high basicity, they slightly acidify the water, so a small amount of chlorides is added to the water. Since the content of chlorides is 105 mg/L, the use of 5/6 AHC in water purification processes is quite promising. The effectiveness of water purification from sulphates, as well as the effectiveness of its softening, depends both on the consumption of lime and coagulant, and on their ratio.

With a fixed dose of coagulant, with an increase in lime consumption, an increase in the efficiency of purification from sulphates is observed. Effective water softening was achieved with stoichiometric lime consumption and with an excess of up to 20% at all doses of coagulant. The residual alkalinity of the water increases with an increase in the consumption of lime, but decreases with an increase in the dose of 5/6 AHC, which contributes to water acidification. The sulphate removal processes are described by the Eq.(8).

$$6CaO + Al_2(OH)_5Cl + 3SO_4^{2-} = 3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \downarrow + Cl^- + 5OH^- \qquad (8)$$
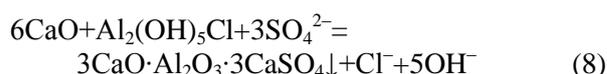
When using 5/6 AHC, it is possible to achieve a decrease in sulphate concentration to 165 mg/L with a residual water hardness of 1.8 mg-eq/L. It is advisable to reduce the alkalinity of water by acidifying it with carbon dioxide. A statistical 2^3-factorial design with

treatment conditions or factors is given in Table 1 and results of the processes are shown in Table 2.

It can be seen in Table 2 that increasing the dose of reagents led to an increase in efficiency of water treatment. The second-order polynomials for all normalized variables for factors of water treatment were calculated. In all cases the models for Y_1, Y_2, Y_3 and Y_4 correlated well with the experimental results as R^2-value for each model is close to 1. The resulting equations were used to predict responses. The results are shown in Figure 2.

When using sodium tetrahydroxoaluminate, it is possible to reduce the concentration of sulphates to 132 mg/L with a residual water hardness of 2.2 mg-eq/L. The maximum precipitation of sulphate occurred during the first 120 min during the precipitation of ettringite (Figure 3). Sulphate removal kinetics was similar for the 5/6 AHC and sodium tetrahydroxoaluminate treatments, but sodium tetrahydroxoaluminate water treatment resulted in slightly lower residual sulphate concentrations.

Table 1

The 2^3-factorial design for water treatment.

Factor	Low level (-1)	High level (1)
The dose of AHC or sodium tetrahydroxoaluminate depends on the stoichiometry (X_1)	1	2
The dose of lime from stoichiometry temperature X_2 (°C)	1	1.2

Table 2

Properties of reagents obtained under different conditions.

X_1	X_2	Y_1	Y_2	Y_3	Y_4
-1	-1	61.2	82.5	68.1	75.2
-1	0	63.6	83.3	69.1	76.2
-1	1	66.1	84.1	70.1	77.2
0	-1	69.3	85.0	89.5	84.8
0	0	74.9	85.7	911	87.3
0	1	80.5	86.5	92.7	89.8
1	-1	71.7	87.7	89.7	87.4
1	0	79.0	88.4	91.9	90.3
1	1	86.4	89.1	94.2	93.2
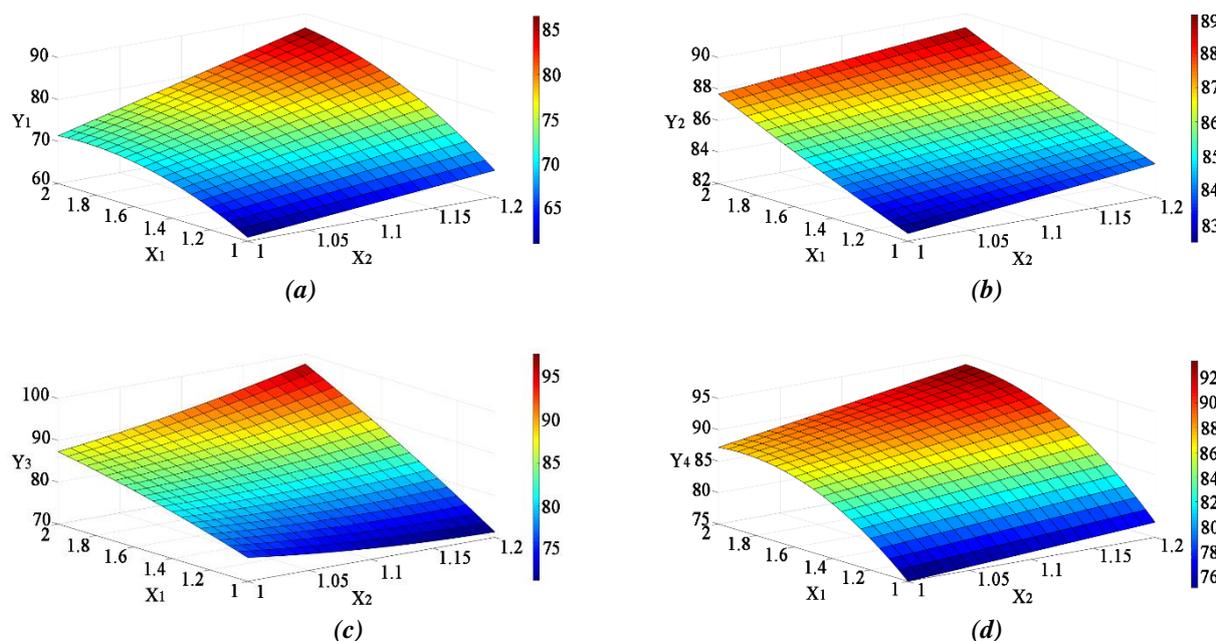
Figure 2. 3D-surface responses: the removal of sulphates with the application of AHC and lime *(a)*; the removal of sulphates with the application of sodium tetrahydroxoaluminate and lime *(b)*; the effectiveness of softening of water during the application of AHC and lime *(c)*; the effectiveness of softening of water during the application of sodium tetrahydroxoaluminate and lime *(d)*.

Physical and mechanical parameters of Portland cement PC I-500 with the use of sediment when using 5/6 AHC.

Composition, %		Normal density, %	Time of hardening, min		Compressive strength, MPa, aged, days		
cement	sediment		beginning	end	1	3	28
100	0.0	29.5	45	465	16.5	33.9	31.1
97.5	2.5	28.5	45	545	13.4	33.7	31.3
95.0	5.0	29.0	11	605	13.6	33.8	32.5
92.5	7.5	29.5	11	670	15.3	33.9	33.5

Physical and mechanical parameters of Portland cement PC I-500 with the use of sediment when using sodium tetrahydroxoaluminate.

Composition, %		Normal density, %	Time of hardening, min		Compressive strength, MPa, aged, days		
cement	sediment		beginning	end	1	3	28
100	0.0	29.5	45	465	16.5	33.9	31.1
97.5	2.5	29.0	44	520	12.9	33.6	31.2
95.0	5.0	29.0	12	630	13.1	33.7	33.0
92.5	7.5	29.5	10	655	14.9	33.9	34.1
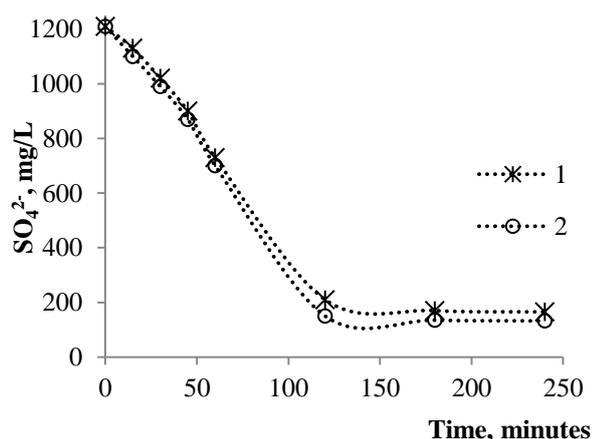
Figure 3. Dependence of the residual concentration of sulphates on time when using 5/6 AHC and sodium tetrahydroxoaluminate.

Maximum efficiency in water hardness reduction up to 2.2–4.7 mg-eq/L was achieved with the application of sodium tetrahydroxoaluminate and lime, and 2.1–3.9 mg-eq/L with the application of AHC and lime with a stoichiometric dose of lime. With the increase in the dose of lime by 20%, the residual hardness of water was 2.9–7.1 and 2.7–5.4 mg-eq/L, respectively. An increase in the dose of coagulants leads to an increase in the effectiveness of softening.

The application of sodium tetrahydroxoaluminate and AHC the residual concentrations of the reagents were insignificant (0.17–0.19 mg/L) and did not exceed the permissible content. According to DSanPiN

2.2.4-171-10, the permissible content of aluminum in drinking water is 0.2 mg/L [27].

The sediment contains a significant amount of calcium sulphate, so it is worth investigating its effect on the properties of Portland cement. Namely, how an increase in the total content of the sediment additive will affect the hardening time and strength. The results are shown in Tables 3 and 4. According to the data, the sediment additive is suitable for application in cement composition. On the 1st day, a slight decrease in strength is observed, but on the 3rd and 28th day no declines in strength are observed. The normal density of cement changes slightly as the sediment content increases. With a content of 5.0–7.5wt.%, the sediment behaves as an accelerator of the initial periods of hardening, and, on the contrary, slows down during the final periods. Thus, up to 7.5wt.% of Portland cement can be replaced with the sediment without deterioration of physical and mechanical properties of the material.

Conclusions
In this paper, a simple method for mine water treatment was developed. The influence of the consumption and ratio of lime and aluminum coagulants on the degree of removal of sulphates from water and the effectiveness of water softening was determined.

The efficiency of water desalination increases with the increase in the dose of coagulant when lime is used in stoichiometric amounts and when its excess is up to 20% at a

permissible chloride concentration. An effective removal of sulphates and softening of mineralized mine water using sodium tetrahydroxoaluminate and lime can be achieved by neutralizing the water with carbon dioxide. Under these conditions, the efficiency of the process increases by 5–10%. The clear dependence between the consumption and the ratio of lime and coagulant was established, which allows to determine the conditions of effective purification of water from sulphates at permissible concentrations of chlorides in water.

Sediments formed during water softening can be used in the construction industry as a partial substitute for cement. The optimal sediment content in Portland cement can be considered up to 7.5wt.%. At such sediment contents, the mechanical parameters of the cement remain within the normal range.

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