

Cleaning Returnable Wastewater from Dysh Deposit Located in Krasnodar Territory

Savenok Olga Vadimovna

*Associate Professor, Department of Oil and Gas Engineering,
Kuban State Technological University, Krasnodarskiy kray, Russia.
¹Orcid: 0000-0003-1312-4312*

Arutyunyan Ashot Straevich

*Candidate of Technical Sciences, Associate Professor of Applied Mathematicians Department,
Kuban State Technological University, Krasnodarskiy kray, Russia.*

Likhacheva Olga Nikolaevna

*Candidate of Philological Sciences, Associate Professor of Foreign Languages Chair,
Kuban State Technological University, Krasnodarskiy kray, Russia.*

Orcid: 0000-0002-1274-3368

Barambonye Solange

*2nd-year masters' student, Department code 21.04.01 «Oil and Gas engineering»
Institute of Oil, Gas, and Energy, Kuban state technological university, Krasnodarskiy kray, Russia.*

Kusov Gennady Vladimirovich

PhD Student, North-Caucasian Federal University, Stavropol Krai, Russia

Abstract

This article describes the research methods and apparatus for cleaning returnable sewage of Dysh oilfield: determination of water hardness by the complex-metric method, by atomic spectrometry and measuring pH by the potentiometric method, determining a total mineralization of formation water gravimetrically and Defining the content of dry residue. The process of electrocoagulation treatment of wastewater from the Dysh deposit has been studied. A technological scheme for purification and demineralization of wastewater at the Dysh deposit has been developed.

Keywords: determination of water hardness through complexometric method, determination of water hardness by atomic spectrometry, pH measurement by the potentiometric method, determination of the total mineralization of formation waters, electrocoagulation treatment of wastewater, technological scheme of sewage water treatment magnitude of prevented environmental damage.

Problems of environmental protection are especially acute in industries related to subsoil use. Among major environmental

problems, a significant role is played by the pollution of water and land resources as a result of the ingress of oil production wastes into the environment.

Despite various types of environmental protection activities, the state of the environment continues to deteriorate. For many years, negative trends have been developing which negatively affect not only the efficiency of field development (deterioration in the quality of residual oil reserves, growth in the share of hard-to-recover reserves, the late stage of development of most fields, etc.) but also the state of the environment [1]. Some wells contain toxic and corrosive components, and these objects are potentially dangerous. Estimating the state of surface water and groundwater in the areas of oil and gas production operations by the results of processing hydrochemical materials, it can be stated that 40 % of chemical analyzes indicate significant techno-genic impacts. In aqueous objects excess of chloride concentration is recorded, total hardness and total mineralization is twice higher than the norms for the investigated (studied) objects. One of the reasons for the current situation is the injection of a huge amount of chemical reagents into the underground horizons, used by the oil industry to increase oil recovery deposit. Together with

that, the problem of utilization of associated formation waters remains unresolved, mixed with oil.

According to the Central Dispatching Department (CDD) with the ministry of energy, average product abduction (water cut) oil production extracted six tons of water.

In accordance with the foregoing, the purpose of this study was formulated: development of technological scheme for purification and demineralization of wastewater at the deposit of Dysh in Krasnodar region.

Scientists from Kuban state technological university in cooperation with researchers from Kuban state university [2, 3] would conduct following studies.

METHODS AND INSTRUMENTS OF RESEARCH

In carrying out these studies, electrocoagulation purification of model and reservoir waters of the Dysh deposit was used in the installation described in the works of Shokhina K.A. and Maksimovich V.G [4-6], as well as the standard of physical and physicochemical methods: gravimetry, thermal analysis, complexometry, potentiometry, etc.

Weight measurements were carried out on analytical scales VLA-200G-M with an error of 0,1mg, the temperature was maintained on the thermostat ShS-80-01SPU with an error of 0,1 Celsius degree, the volumes of formation water and other liquid reagents were measured with an accuracy of 0, 05 ml.

Photomicrographs of sections of the Dysh core samples submitted for the study were obtained with a scanning electron microscope JEOL 7500F scientific and educational center «Diagnostics of structure and properties of nanomaterials» of Kuban state university.

The determination of the cation and anionic composition of the formation water was carried out in the analytical laboratory of Kuban state university.

The methods used in the work that allowed deviations from standard methods are set out below.

Determination of water hardness by complexometric method

The sample is taken with a volume of at least 400 cm³ in a container made of polymer materials or glass. The shelf life of the water sample is not more than 24 hours. The method is based on the formation of complex compounds of Trilon-B with ions of alkaline-earth elements. The determination is carried out by titrating the sample with a solution of Trilon-B at pH=10 in the presence of an indicator. The least determined hardness of water is 0,1 H.

In a conical flask with a capacity of 250 cm³, add 10,0 cm³ of a solution of magnesium ions add 90 cm³ of distilled water, 5 cm³ of buffer solution, from 5 to 7 drops of the indicator solu-

tion or from 0.05 to 0.1 g of dry indicator mixture and immediately titrate with a solution of Trilon-B until the color changes at the equivalent point from the wine-red (red-violet) to the blue (with greenish tinge) when using the indicator eriochrome black T, and when using the indicator chromic dark blue-acid to blue (blue-violet).

The solution of Trilon-B at the beginning of the titration is added fairly quickly with constant stirring. Then, when the color of the solution begins to change, Trilon-B solution was added slowly. Equivalent point is reached when the staining changes when the color of the solution ceases to change when adding drops of Trilon-B solution.

Titration is carried out against the background of a titrated control sample. As a control sample, a slightly oversaturated test sample can be used. For the result, the mean arithmetic value of the results of at least two definitions is taken. The coefficient of correction K to the concentration of a solution of Trilon-B [7, 8] is calculated by the formula:

$$K = \frac{10}{V}, \quad (1)$$

where V – a volume of a solution of Trilon-B, consumed in the titration, cm³; 10 – a volume of magnesium ion solution cm³.

Determination of water hardness by atomic spectrometry

The method is based on measurement of resonance absorption of light by the sabbatical atoms of the chemical elements of magnesium and calcium when light passes through the atomic vapor sample being studied, formed in flame. Lanthanum chloride or cesium chloride is added to the aliquot of the sample to eliminate the fading effects.

In volumetric flasks with capacity of 100 cm³, 10 cm³ of lanthanum chloride solution are introduced, if an air-acetylene flame is used, or 10 cm³ of cesium chloride solution, if nitrous oxide is used acetylene, then add an aliquot sample of water (as generally not more than 10 cm³) and bring to the mark with a solution hydrochloric acid.

If the measured content of calcium or magnesium in the test sample higher than the maximum values established when the spectrometer calibrated, then reduced analyzed sample is used for the determination. In accordance with the instruction manual for the operation of the spectrometer, the solutions to be analyzed are introduced into it, and in the intervals between them a solution hydrochloric acid. Determine the absorption of each element at an analytical wavelength.

Simultaneously, a blank test is carried out using the same reagents in the same quantities, replacing the volume of the analyzed sample with bi-distilled water.

By calibration curve, including using a software spectrometer, determine the mass concentration of calcium and magnesium

in the solutions and in the diluted solution and calculate the calcium and magnesium content of the sample, taking into account dilution of sample value, obtained in a blank solution.

The hardness of water H is calculated from the formula:

$$H = \left(\frac{C_i}{C_{ie}} \right) \cdot F \cdot \left(\frac{V_f}{V_{ws}} \right), \quad (2)$$

where C_i – a mass element concentration in the water sample, determined according to the calibration curve, minus the result of the analysis of the blank solution, mg/dm³; C_{ie} – a mass concentration of element, mg/dm³, numerically equal to 1/2 mol; F – a dilution factor of the initial water sample during preservation (usually $F = 1$); V_f – capacity of the flask in which the sample was prepared, cm³; V_{ws} – water sample taken for analysis, cm³.

pH measurement by potentiometric method

Method for determination of the pH of water samples based on the measurement of the EMF (electron motor force) of the electrode system consisting of a glass electrode, which potential is determined by the activity of hydrogen ions, and the auxiliary reference electrode with a known potential.

The test sample with a volume of 30 cm³ is placed in a 50 cm³ beaker. The electrodes are rinsed with distilled water, washed with water under investigation, immersed in a beaker with the analyzed sample. In this case, the glass measuring electrode ball must be completely immersed in the solution, and the salt contact of the auxiliary electrode must be immersed to a depth of 5-6 mm. Simultaneously, a thermo-compensator is immersed in the beaker.

The pH value is calculated on the instrument scale when the instrument reading does not change by more than 0.2 pH units for one minute, after a minute the measurement is repeated if the pH values differ by no more than 0.2, then the analysis result is taken by the average arithmetical value.

After measurement, the electrodes are rinsed with distilled water and wiped with filter paper or a soft cloth.

If it becomes necessary to decrease the electrode, then it is wiped with a soft cloth moistened with ethyl alcohol and then several times rinsed with distilled water and wiped with a soft cloth.

If necessary, the electrode is regenerated by immersion for 2 hours in a 2 % solution of hydrochloric acid and then thoroughly washed with distilled water [9].

Determination of total mineralization of formation water by weight method of determination of the dry residue content

The value of the dry residue characterizes the total content of dissolved in water non-volatile mineral and partially organic compounds.

250-500 cm³ of filtered water is evaporated in a porcelain cup previously dried to a constant mass. Evaporation is carried out in a water bath with distilled water. Then a plate with a dry residue is placed in a thermostat at 110 °C and dried to constant weight.

The dry residue X (mg/dm³) is calculated by the formula:

$$X = \frac{(m - m_1) \cdot 1000}{V}, \quad (3)$$

where m – a mass of a cup with a dry residue, mg; m_1 – a mass of empty cup, mg; V – the volume of water taken to determine, cm³.

This method of determining the dry residue gives somewhat exaggerated results due to the hydrolysis and hygroscopicity of magnesium and calcium chlorides and the difficult return of water of crystallization with sulfates of calcium and magnesium. These disadvantages are eliminated by adding chemically pure sodium carbonate to the evaporated water. In this case, chlorides, calcium, and magnesium sulfates are converted to anhydrous carbonates, and from sodium salts, only sodium sulfate has crystallization water, but it is completely removed by drying the dry residue at 150-180 °C.

All measurement results are given in the corresponding tables and graphs.

Study of the process of electrocoagulation cleaning wastewater from the Dysh deposit

The reservoir waters were investigated for general mineralization, pH, Ca²⁺, Mg²⁺, SO₄²⁻, Cl⁻, HCO₃⁻ and other. A thermal treatment of the dry residue as a result of the conducted studies, it was found that with a fairly close composition of samples of formation water of these wells, significant differences are observed between them, apparently related to the specific features of these wells. Thus, the results of analyses showed that the total mineralization of samples of formation water is 10.7-11.1 g/l, which allows them to be classified by their degree of salinity to saline reservoirs. The hydrogen pH for the samples under study varies between 8.30-8.45.

The results of the study of formation water for the content of anions are given in Table 1. The sample numbers correspond to the conditionally assigned number of the well.

Table 1: Results of the study of formation waters for the content of some anions, mg/l

Sample №	SO ₄ ²⁻	Cl ⁻	HCO ₃ ⁻	Br ⁻	I ⁻
1	7.4	4076	3843	71.3	20.7
2	6.6	4254	3904	69.5	24.0
3	7.4	4431	3843	70.5	23.8
4	8.2	4254	3660	68.7	23.5

As can be seen from the data in Table 1, the main salts of the adjacent water from the Dysh deposit are chlorides and hydrogen carbonates. To determine the type of these salts, an atomic-adsorption analysis of the formation water on the cation content was carried out on the basis of the analytical center of Kuban state university. The results of the study of formation waters for the content of some cations are given in Table 2.

Table 2

Sample №	Mg ²⁺	Fe ²⁺	Ca ²⁺	K ⁺	Na ⁺	Li ⁺	B ³⁺
1	68.4	0.08	86	33	3419	1.3	40.8
2	72.0	0.12	100	35	3781	1.2	43.0
3	72.0	0.21	100	34	3437	1.2	42.5
4	66.0	0.19	70	33	3439	1.2	41.7

Analysis of the data in Tables 1 and 2 allows us to consider that the main components of the mineral composition of the samples of formation water presented for study are chlorides and bicarbonates of sodium, calcium, and magnesium. For the experimental confirmation of this assumption, a thermal analysis of dry residues of evaporated formation water was carried out. The graphs of the change in the mass of the dry residue of evaporated formation water with increasing temperature are shown in Figure 1.

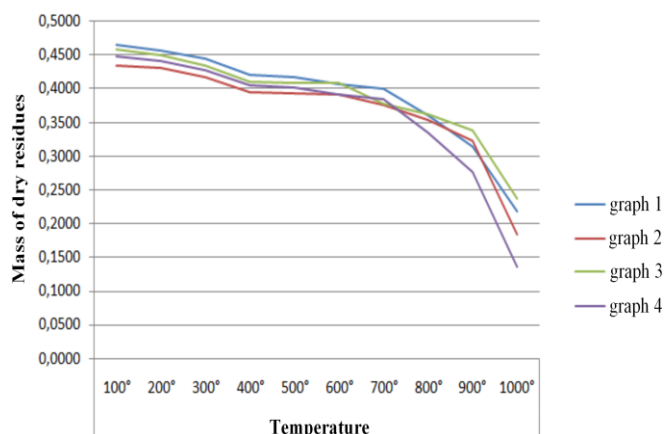


Figure 1: Diagram of the change in the mass of the dry residue of strata water

The graphs of the change in the mass of the dry residue of evaporated strata water (Figure 1) confirm the presence of bicarbonate ions (their decomposition into CO₂ and H₂O at temperatures above 700 °C). Overcoming the mineralization of formation water can serve as a decrease in carbonate hardness.

To reduce the mineralization, we used the method of electrocoagulant wastewater treatment. The method of electrocoagulation purification is based on the formation, under the influence of a passing electrical current, of highly active aluminum and/or iron hydroxides immediately reacting with polluting water impurities, followed by a rapid transition of bound impurities and unreacted reagents into insoluble, chemically inert, easily separable sludge [10]. While this method is usually used to purify waters from colloidal and fine impurities, it is obvious that the side effect of the formation of flakes of aluminum or iron hydroxides will be a change in the ionic composition of the mixture due to the numerous interactions between the components of the system and sorption on the colloids of the hydroxides of not only neutral molecules oil and other molecules, but also ions (cations and anions) alternately.

It is also important that, in comparison with reagent coagulation during electrocoagulation, sulfate and chloride do not additionally enrich the purified water with anions and other salts, which facilitates the control of the final ionic composition of the mixture and allows regulating the process. Electrocoagulation purification was carried out on a laboratory unit developed at the Department of General, Inorganic Chemistry and Information and Computing Technologies in Chemistry of Kuban state university and described earlier in the works of Shokhina K.A. and Maksimovich V.G. [4-6]. Schematic diagram of the elementary electrocoagulation cell used by us in this work is shown in Figure 2.

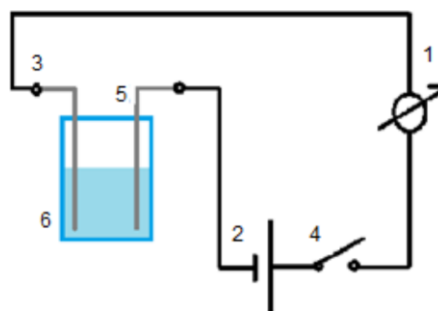


Figure 2: Diagram of a laboratory electrocoagulation unit:

- 1 – galvanometer (ammeter); 2 – source of direct current (potentiostat);
- 3 – wires and clamps; 4 – the key; 5 – electrodes; 6 – chemical beaker

Aluminum electrodes are usually used for electrocoagulation treatment of wastewater from galvanic industries, however, it was shown in [4-6] that it is more convenient to use iron electrodes when cleaning oil sludge. First, iron electrodes are more stable in alkaline solutions than aluminum electrodes, and secondly, because of the greater equivalence of iron, they

are more economical, and the cost of iron is an order of magnitude lower than the cost of aluminum. Finally, as our experiments have shown, iron electrodes are less prone to passivation during electrocoagulation than aluminum electrodes. Thus, during the experiment, the current strength in the solution increased even slightly over time (Figure 3).

Explain this fact by simply dissolving the iron anode and increasing the conductivity of the solution due to an increase in the concentration of electrolytes is impossible, since the initial concentrations of ions of the mineral salts of the formation water already provide sufficient conductivity for electrolysis. We attribute this effect to the fact that when using iron electrodes, the process of their anodic dissolution leads to the formation of divalent iron ions (Fe^{2+}), which are immediately oxidized to the trivalent state (Fe^{3+}) and form a gel of hydrox-

ide $Fe(OH)_3$. Oxidation of Fe^{2+} to Fe^{3+} occurs due to oxygen dissolved in water and overvoltage on the electrodes, and the subsequent formation of iron (III) hydroxide occurs due to the binding of the formed Fe^{3+} ions to the hydroxyl ions, preventing their depletion with passivation of the anode. A small increase in the current intensity we associate with the growth (development) of the electrode surface during electrolysis.

At the same time, as our studies have shown, the iron (III) hydroxide gel that is formed clears the formation water well enough not only from oil and other molecular impurities (surfactants, for example) but also binds calcium and magnesium ions quite well. Analysis of the composition of the formation water after electrocoagulation cleaning showed that the concentration of Ca^{2+} and Mg^{2+} ions in all samples of formation water significantly decreased (Tables 3-6).

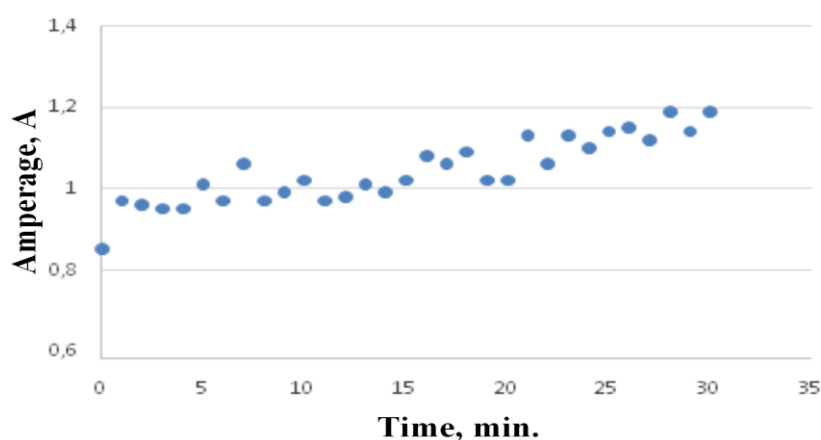


Figure 3: Variation of current strength in solution over time

Table 3: Sample formation water № 1

Rigidity, mg-eq/l		The calcium content, mg-eq/l		The magnesium content, mg-eq/l	
initial	after electrocoagulation	initial	after electrocoagulation	initial	after electrocoagulation
10.00	2.10	4.30	0.55	5.70	1.55

Table 4: Sample formation water № 2

Rigidity, mg-eq/l		The calcium content, mg-eq/l		The magnesium content, mg-eq/l	
initial	after electrocoagulation	initial	after electrocoagulation	initial	after electrocoagulation
11.00	3.00	5.00	0.75	6.00	2.25

Table 5: Sample formation water № 3

Rigidity, mg-eq/l		The calcium content, mg-eq/l		The magnesium content, mg-eq/l	
initial	after electrocoagulation	initial	after electrocoagulation	initial	after electrocoagulation
11.00	1.20	5.00	0.60	6.00	0.60

Table 6: Sample formation water № 4

Rigidity, mg-eq/l		The calcium content, mg-eq/l		The magnesium content, mg-eq/l	
initial	after electrocoagulation	initial	after electrocoagulation	initial	after electrocoagulation
9.00	2.00	3.50	0.50	5.50	1.50

The diagram of the change in the content of Ca^{2+} and Mg^{2+} ions in the samples of wastewater before and after the electrocoagulation cleaning is shown in Figure 4.

Analysis of the obtained results allows to conclude that the decrease in the Ca^{2+} ion content in the initial period of electrocoagulation purification is more intense than the decrease

in Mg^{2+} ions. Thus, the concentration of Ca^{2+} ions decreased by 86.36 % on average and the concentration of Mg^{2+} ions by 69.64 %. This effect can be explained within the framework of Pearson's theory by a greater affinity for iron (III) hydroxide of calcium ions than the more rigid magnesium ions.

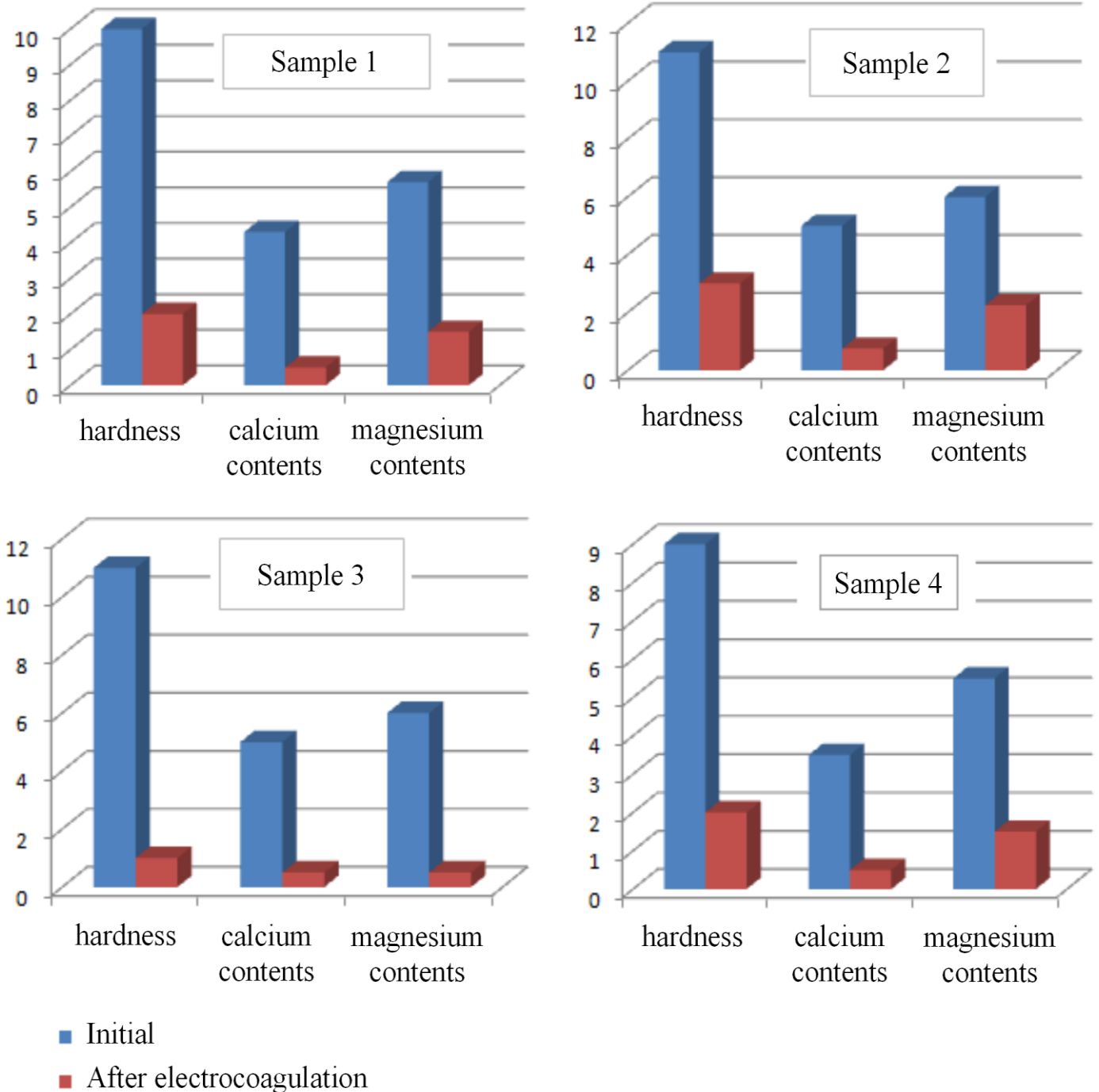


Figure 4: Diagram of changes in the content of ions Ca^{2+} and Mg^{2+} (mg-eq/l) in water that has been electrocoagulation cleared

According to the results of our study, the precipitation of hardness metal cations (Mg^{2+} , Ca^{2+} , Fe^{2+} , Fe^{3+} , etc.) occurs both on the surface of the electrodes, which was confirmed by X-ray luminescence analysis and on the flocs of the formed coagulant $Fe(OH)_3$. The results of X-ray-luminescent analysis of the surface of electrodes after electrocoagulation cleaning are given in Table 7.

Table 7: Content of Ca^{2+} and Mg^{2+} ions on the surface of electrodes after electrocoagulation cleaning

Name of ions	The ion content in the test samples, %			
	Sample № 1	Sample № 2	Sample № 3	Sample № 4
Ca^{2+}	0.156	0.108	0.215	0.232
Mg^{2+}	0.145	0.086	0.144	0.175

The data cited to confirm the hypothesis that the concentration of the ions under consideration can be reduced by electrocoagulation. To determine the possibility of regulating the content of hardness metal ions in the solution of the treated wastewater during electrocoagulation cleaning, the influence of the current level change during electrocoagulation cleaning on the reduction of mineralization of wastewater was investigated.

Electrocoagulation was carried out sequentially on three identical samples of formation water with voltages set at a constant current source: 5, 10 and 17 V. As in the previous experiment, the current strength in the solution changed somewhat over time. Table 8 shows the «averaged» current strength with experiment duration of 30 minutes.

Table 8: Current intensity value with voltage change

Voltage value, V	Amperage, A
5	0.81
10	1.46
17	0.01

As can be seen, when the current is passed through a voltage of 17 V, the value of the current strength is significantly reduced, which can be explained by excessive polarization («burning out») of the electrodes at a voltage greater than 15 V. The determination of calcium and magnesium ions was carried out by the titrimetric method. The method is based on the complex-metric titration of the water under investigation using a Trilon-B reagent (sodium salt of ethylene diamine tetra-acetic acid) in the presence of black eriochrome indicator T.

In the presence of Ca^{2+} and Mg^{2+} ions at $pH = 7-11$, the indicator due to the formation of complexes with these ions acquires a characteristic lilac-red color. When Trilon-B is added, these complexes decompose, and Ca^{2+} and Mg^{2+} ions bind to a colorless, more stable complex with Trilon-B, and the solution

acquires a pale blue color of the indicator. Change in color occurs at the time when all the ions of Ca^{2+} and Mg^{2+} are consumed. In this case, the amount of Trilon-B reacted is equivalent to the number of Ca^{2+} and Mg^{2+} ions.

Calcium and Magnesium are calculated by formulas:

$$C_{Ca} = \frac{a \cdot N \cdot 20,0}{V} \cdot 1000; \quad (5)$$

$$C_{Mg} = \frac{b \cdot N \cdot 12,6}{V} \cdot 1000, \quad (6)$$

where a and b – the consumption of Trilon-B (titrant), which went to titration of calcium and magnesium ions, ml; N – the normality of solution Trilon-B (0.1 mol/dm^3); V – the volume of the sample taken for analysis, ml; 20.0 and 12.6 – gram equivalents of calcium and magnesium.

The data obtained in the course of the experiment for determining the concentration of Ca^{2+} and Mg^{2+} ions are given in Tables 9 and 10.

Table 9: Results of the experiment in determining the concentration of Ca^{2+} ions

Voltage, V	Volume of H_2O , ml	The volume of solution Trilon-B, which has used in titration, ml
5	10	0.5
10	10	0.4
17	10	0.2

Table 10: Results of the experiment in determining the concentration of Mg^{2+} ions

Voltage, V	Volume of H_2O , ml	The volume of solution Trilon-B, which has used in titration, ml
5	10	0.5
10	10	0.2
17	10	0.1

The results of the calculations are given in Tables 11 and 12.

Table 11: Calculated concentrations of Ca^{2+} ions

Voltage, V	Calcium concentration, mg/dm^3
5	100
10	80
17	40

Table 12: The calculated concentrations of Mg^{2+} ions

Voltage, V	Magnesium concentration, mg/dm^3
5	60
10	30
17	10

The graphs of the dependence of the final concentration of Ca^{2+} and Mg^{2+} ions on the voltage are shown in Figures 5 and 6.

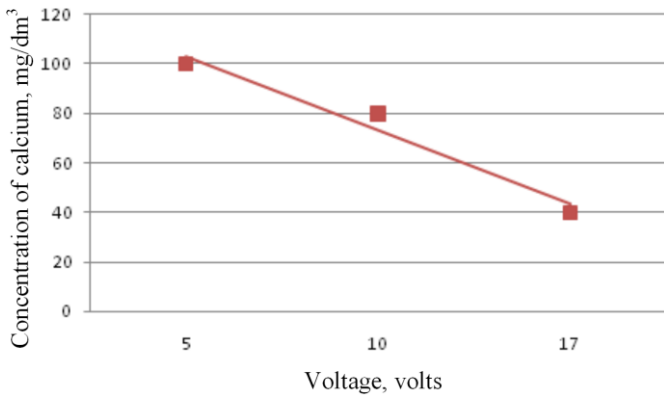


Figure 5: Graph of changes in the concentration of ions Ca^{2+}

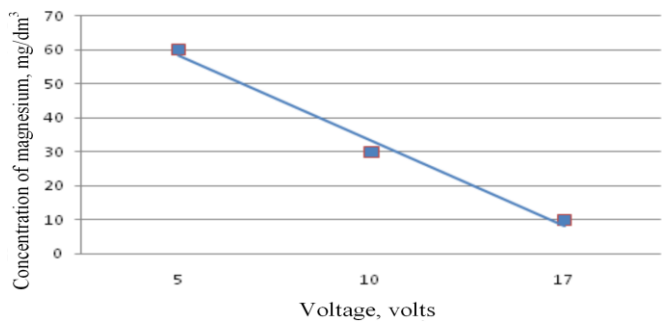


Figure 6: Graph of changes in the concentration of ions Mg^{2+}

Analyzing the obtained results, it can be concluded that with increasing potentiostat voltage of the electrocoagulation installation, the efficiency of purification from Ca^{2+} and Mg^{2+} ions increases. However, at a voltage of more than 15 V, long-term maintenance of the process is impossible due to the polarization of the electrodes and a sharp decrease in the current in the solution. Therefore, the recommended voltage for conducting electrocoagulation cleaning is 10-13 V.

Moreover, judging by the results obtained, by selecting the current-voltage characteristics, it is possible to regulate the content of calcium and magnesium ions in the cleaned reservoir waters.

Development of the technological scheme of sewage water treatment of the Dysh deposit

For complex and more complete treatment of associated formation water, the following purification scheme is proposed (Figure 7):

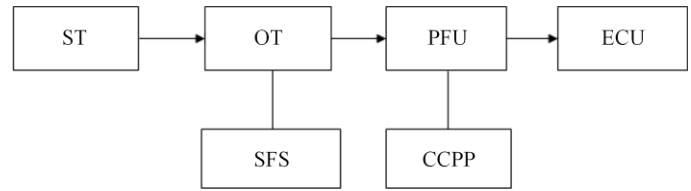


Figure 7: Scheme of cleaning associated reservoir waters:
 ST – sand trap; OT – an oil trap; SFS – site for sediment;
 CCPP – a collection of captured petroleum products;
 PFU – pressure flotation unit;
 ECU – electrocoagulation unit

The formation water flow is fed to the oil trap, where the first stage of purification takes place, which is based on the separation in the field of gravitational forces in a quiescent or slowly moving fluid flow. In this case, suspended substances with a density greater than the density of water, precipitate, substances with a lower density – float. The size of the retained globules of oil is more than 80 microns. Based on the operating data of recent years, the residual oil content after oil traps is 80-450 mg/l. The efficiency factor for cleaning oil traps is about 55 %.

To ensure the normal operation of the main sedimentation facilities (oil traps, etc.), it is advisable to remove the heavy mineral impurities from the sewage beforehand. They reduce the mobility of the sediment, increase the load on the scraper mechanisms, are deposited in pipelines, trays, water distribution devices, entrance chambers, cause premature wear of the equipment. At sewage treatment plants, this work is carried out by auxiliary settling facilities, called sand catchers. Depending on the amount of oil delivered to the tank farms, pumping stations, and liquid filling stations, the volume of wastewater can vary significantly. The amount of mechanical impurities in wastewater usually ranges from 500-1000 mg/l. In the normal operation of the pits, only 10-15 % of coarse particles are retained from the total amount of mechanical impurities.

Further, the stream of purified formation water enters the flotation plant. For the purification of wastewater after the main oil recovery unit, as indicated, flotation is also used. The flotation method is that air bubbles adhering to oil particles lift them to the surface of the water in the form of a foam. The latter is rapidly destroyed, and the oil layer is removed by the oil collection device. As a result, produced water with a content of pollutants of 20-30 mg/l was obtained at the outlet. Further, the reservoir water is fed to the purification by electrocoagulation [11].

CONCLUSIONS

The following results were obtained during the research:

- 1) the technological scheme of sewage water treatment was developed;

- 2) the possibility of lowering the mineralization of formation water in the process of electrocoagulation cleaning has been investigated.

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