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THE MECHANISM OF ACCUMULATION OF FERRUM COMPOUNDS IN HIGHLY MINERALIZED MINE AND QUARRY WATERS AND THEIR PURIFICATION BY ELECTROLYSIS.

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The mechanism of accumulation of dissolved iron compounds in groundwater and brines under the influence of sulfate reduction and the resulting decrease in the redox potential is considered. The mechanism under consideration has been experimentally confirmed. A method of extraction of sulfides and ferrum-containing compounds by electrochemical oxidation using an inert anode is proposed. The technology for obtaining an inert anode based on manganese and lead oxides, stable in most natural waters and brines, which does not contain noble metals and their compounds, is described. The specified technology involves the deposition of manganese dioxide on a titanium base by thermal decomposition of manganese nitrate. After that, the base is covered with a thin layer of PbO_2 by electrodeposition from an alkaline complex electrolyte, and then with a thick layer of the same oxide by electrodeposition from a nitrate electrolyte. It has been established that the contact of an alkaline complex electrolyte with the active surface of metallic lead can significantly reduce the formation of bottom deposits during electrodeposition. It is shown how interruption of the current during the electrodeposition of such an anode can reduce its porosity and increase its stability. The process of iron removal and sulfide extraction from highly mineralized mine and quarry waters has been studied. It has been theoretically calculated and experimentally confirmed that the electricity consumption for the iron removal process of such waters does not exceed 1 kW*h/m^3. An example of the application of the method of electrochemical iron removal and sulfide extraction from highly mineralized brines with their subsequent resource-saving processing and obtaining water for power supply of energy facilities and commodity mineral salts is given.

Keywords: Sulfate reduction, ferrum removal, sulfide removal, electrolysis, mine waters and brines.

INTRODUCTION. Almost all mining and industrial regions of Ukraine are water-deficient. For the needs of industry, in particular, thermal power engineering, a significant amount of drinking water is used in these regions. At the same time, large amounts of water are pumped out of mines, including closed ones, and quarries. But these waters are contaminated with mineral impurities and cannot be used for technical needs without deep purification, in particular demineralization. The calculation of possible environmental damage from the discharge of only brines from quarries and tailings ponds of the Kalush mining and industrial region into surface water bodies, carried out according to the methodology (Methodology, 2009), shows that such damage exceeds 3 billion UAH.

There is a method for purifying highly mineralized waters and brines with the production of impurities in the form of marketable by-products (Tarelin A.O., 2018). However, in most cases, such waters contain quantitatively small, but quite dangerous impurities of soluble iron and sulfide compounds. When attempting to subject these waters to membrane demineralization without prior removal of iron and sulfides, the latter are deposited on the surface of the membranes, irreversibly disabling them. When attempting to demineralize such waters thermally, sulfides and iron (III) hydroxide are deposited on the heating surfaces in the form of a rocky scale, which can only be removed mechanically. Therefore, preliminary deep extraction of these compounds is necessary.

The mechanism of accumulation of soluble ferrum compounds and sulfides in groundwater and brines is currently not sufficiently studied. There are separate publications describing the mechanism of dissolution of pyrite and chalcopyrite when groundwater flows through restored rocks (Xinfeng Wang, 2021). However, insufficient attention is paid to the processes of dissolution of ferrum compounds from oxidized rocks. These rocks make up the majority of the surface of the earth's crust. They contain trivalent ferrum oxide, which is almost insoluble at pH above 5.0. At the same time, it is in the waters flowing through such rocks that the concentrations of the specified element are relatively high (Table 1).

Table 1.

Main indicators of highly mineralized waters and brines.

Name of water	Indicators, mg/dm^3		
	Ferrous soluble	Sulfides	Total mineralization
Water from the Batkivshchyna mine of the Kryvyi Rih iron ore basin	30,3	17,2	83920
Brine from the Dombrovsky quarry	30,7	15,6	350000
Formation water from oil production wells of the Poltava region	270	18,0	220000

The mechanism of accumulation of soluble ferrum compounds in low-mineralized groundwater in the Netherlands flowing through oxidized clays has been described (van Beek, G.G.E.M., 2021). However, the main focus of this article is on the dissolution and accumulation of $FeCO_3$ siderite in waters under the influence of low pH, and insufficient emphasis is placed on the role of redox processes.

The aim of the work is the theoretical and experimental study of the causes and mechanism of accumulation of ferrum compounds in mine waters and brines, as well as the development of methods and equipment for their removal.

EXPERIMENT AND DISCUSSION OF RESULTS. Calculations of the potentials of redox reactions that occur in salt solutions under anaerobic conditions were carried out using the well-known Nernst formula. The materials for the research were brine taken from the Dombrovsky quarry at a depth of 42 m, a water sample from the Rodina mine of the Kryvyi Rih iron ore basin, and model solutions of a composition similar to the brine composition and groundwater of the Buchach horizon (mineralization 2.5 g/dm^3). The concentrations of the solution components were measured using standard analytical methods.

Manganese nitrate and known nitrate and alkaline complex electrolytes for lead dioxide electrodeposition were used to manufacture inert anode samples (Mykhailenko V.G., 2022).

As a result of the vital activity of bacteria of the genus Desulfovibrio and Desulfotomaculum (de Rezende J.R, 2013, Osturk M, 2021), in sulfate-containing solutions under anaerobic conditions, in the presence of organic substances, microbiological sulfate reduction occurs and hydrogen sulfide and free carbonic acid accumulate (Basen M, 2011):

$$SO_4^{2-} + 10H^+ + 8e = H_2S + 4H_2O;$$
$$C_3H_6O_3 + 3H_2O - 12e = 3CO_2 + 12H^+.$$

At the same time, a significant decrease in pH and Eh of solutions occurs. Since in a weakly acidic environment hydrogen sulfide is extremely poorly dissociated, the content of S2- ions is small, the solubility product of FeS and FeS_2 is not achieved, and the indicated sparingly soluble compounds cannot be formed (Bozo-Hurtado L, 2013)

Therefore, it was necessary to establish and experimentally substantiate the mechanism of accumulation of iron compounds and sulfides in highly mineralized waters, and then develop a method for extracting these compounds, which, when present together, interfere with the low-waste processing of such waters to obtain purified water for the energy industry and related commercial products. Known technologies for purifying water from such compounds by interaction with air oxygen or ozone (Duranceau S J, 2010 Leszczyński J., 2019) do not work at high mineralization due to poor solubility of gases. There is a known method for extracting iron compounds and sulfides from highly mineralized formation waters by electrocoagulation using iron anodes (Shamsiyev Sh.D, 2019). However, this method is associated with high consumption of anodes, which are products from commercial metal sheets. In addition, it does not allow for deep extraction of dissolved iron compounds, and is generally unsuitable for brines with high mineralization.

Simultaneous removal of sulfides and ferrum compounds from highly mineralized

waters is possible using electrotreatment with inert anodes, but at present this method is too expensive, since it is necessary to use noble metals or titanium coated with a layer of such metals or their compounds as such anodes (Liu L., 2024). The use of graphite as an inert anode for the purification of highly mineralized waters is impractical, since this material is unstable during anodic polarization in the O2 release mode (Sanches-Sanches T J., 2020). Thus, the next task of the research was to develop a stable inert anode that would not contain noble metals and their compounds. When implementing it, the authors paid attention to low-wear anodes based on manganese and lead dioxides.

Titanium-based anodes coated with a layer of manganese dioxide (MnO_2) do not require a noble metal sublayer (Mohammadi M.). During the thermal decomposition of manganese nitrate, a metallothermal reaction occurs on the titanium surface, and the metallic manganese released as a result dissolves in titanium, forming a penetration solution. Therefore, manganese dioxide obtained by thermal decomposition of manganese nitrate has good adhesion to the titanium base and, at the same time, creates a relief necessary to improve the adhesion to the base of other coatings. Therefore, before applying such a coating to titanium, it is possible not to apply a relief by mechanical treatment, as in other cases. However, manganese dioxide itself is not sufficiently stable during anodic polarization in chloride-sulfate solutions and gradually collapses. Eventually, the active coating becomes porous, the titanium base begins to be covered with a semiconducting film of titanium dioxide, and after that, electrolysis stops due to a significant increase in the anode voltage.

A more stable anode material is lead dioxide (Mukimin A., 2013). However, when it is applied directly to a titanium substrate, during anodic polarization, a relatively rapid diffusion of atomic oxygen to the titanium surface occurs and the substrate is subsequently passivated by a TiO2 film with increasing voltage and termination of electrolysis (Olesia B., 2017). To significantly increase the service life of such anodes, a thin coating of platinum, iridium or other noble metals and their compounds is applied under the layer of the main oxide coating (Devilliers D., 2003).

Solutions for electrodeposition of lead dioxide of various compositions are known. The most common is an acidic nitrate electrolyte, which is characterized by high stability, allows high current densities (up to 1000 A/m2) and is easy to prepare and use (Mykhailenko V.H., 2022).

The second group of electrolytes is alkaline plumbite (Mukimin A., 2013) and complex (Mykhailenko V.H., 2022) solutions, which give compact shiny deposits, but only at low current densities (100–200 A/m^2). In addition, these electrolytes are kinetically unstable, since after passing a certain amount of electricity (4–5 $A*h/dm^3$ for plumbite electrolyte and 5–6 $A*h/dm^3$ for complex) brick-red bottom deposits fall out of them. Thus, it was necessary to develop a method for obtaining inert anodes and, on their basis, develop a technology for extracting iron compounds and sulfides from highly mineralized wastewater.

The selected water sample from the Rodina mine has a redox potential relative to the hydrogen electrode of 230 mV and pH 5.5, the brine of the Dombrovsky quarry – 270 mV and 5.7, respectively, and the formation water sample – 250 mV and 5.4. At this pH, trivalent

ferric compounds will not dissolve. However, the analysis shows that significant concentrations of divalent ferric compounds in ionic form are present in the solution. According to the Pourbe diagram for ferric compounds, under these conditions, divalent ferric ions are stable.

The following mechanism for the dissolution of oxidized iron-containing rocks was proposed.

When a solution with a low redox potential comes into contact with iron-containing rocks, hydration of trivalent ferric oxide occurs and its reduction occurs according to the reaction:

$$Fe_2O_3 + 3H_2O + 2e = 2Fe(OH)_2 + 2OH^-.$$

The equilibrium potential of this reaction is +581 mV. At the same time, hydrogen sulfide is oxidized by the reaction:

$$H_2S + 3H_2O - 6e = SO_3^{2-} + 8H^+.$$

This reaction has a potential of (-500) mV. Thus, the potential difference between the oxidation and reduction reactions exceeds 1 V, which is quite sufficient for their spontaneous occurrence.

The formed sulfite ion SO_3^{2-} is used by sulfate-reducing bacteria to oxidize a new amount of organic matter

The formed iron (II) hydroxide interacts with carbon dioxide to form soluble iron bicarbonate $Fe(HCO_3)_2$. This mechanism is similar to that described in the literature (van Beek, G.G.E.M., 2021), however, it clearly links the accumulation of ferrum bicarbonate (and not siderite, which is poorly soluble) not only with pH, but also with the low redox potential of the system.

The described mechanism of dissolution of Fe_2O_3 was experimentally confirmed during laboratory studies.

The experiments were performed on three model solutions, the composition of which simulated brackish water with a mineralization of 2.5 g/dm^3 (solutions 1 and 2) and brine with a mineralization of 305 g/dm^3 (solution 3). Sodium sulfide and sodium bicarbonate were added to solutions 2 and 3 and then neutralized with hydrochloric acid to pH 5.7, thus reproducing the conditions in groundwater. Solution 1 was treated in the same way, but instead of sodium sulfide, a similar amount of sodium bicarbonate was added. The solutions were brought into contact with an ferrum-containing substance, in this experiment with freshly deposited trivalent ferric hydroxide. The results of the studies (Fig. 1) show that in the absence of a reducing medium, the accumulation of iron compounds in the solution does not occur. If a strong reducing agent (H2S) appears in the solution, dissolution of iron compounds and their accumulation occur, and in a highly mineralized environment this process proceeds much slower than in low-mineralized solutions.

The dissolution of trivalent iron compounds occurs approximately ten times slower upon contact of the solution with ground Kryvyi Rih red hematite (Fig. 2). After 5 days of contact with the iron-containing substance, the concentration of soluble iron compounds increased from 0 to 0.4 mg/dm^3, which, however, is a sufficient rate for geological processes. Thus, the mechanism of accumulation of iron compounds in natural waters and brines with the participation of the sulfate reduction process has been experimentally proven.

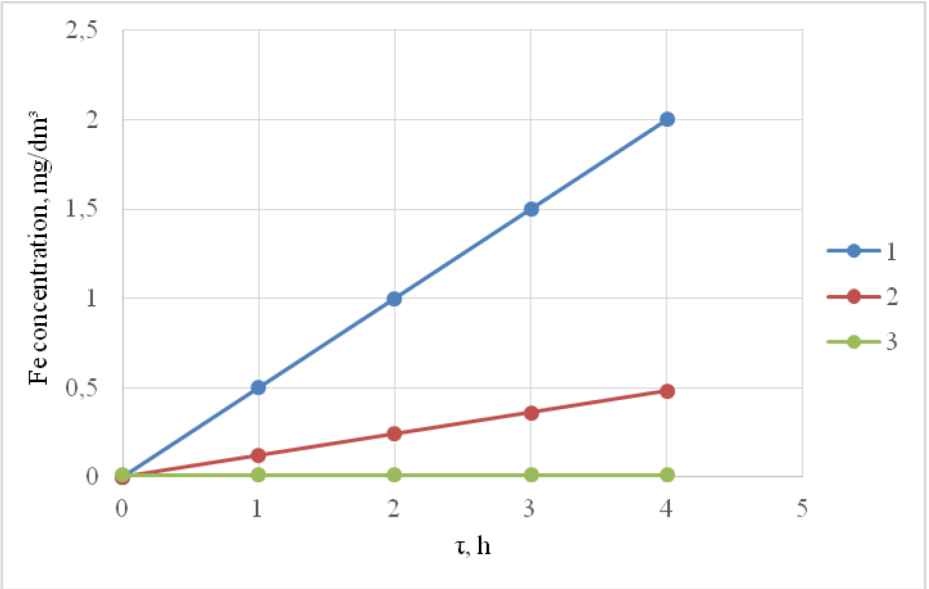

Fig. 1. Accumulation of ferrum compounds in model solutions upon contact with freshly deposited Fe(OH)3. 1 – low-mineralized water with an admixture of hydrogen sulfide 15 mg/l; 2 – high-mineralized water with an admixture of hydrogen sulfide 15 mg/l; 3 – low-mineralized water without the addition of sulfides.

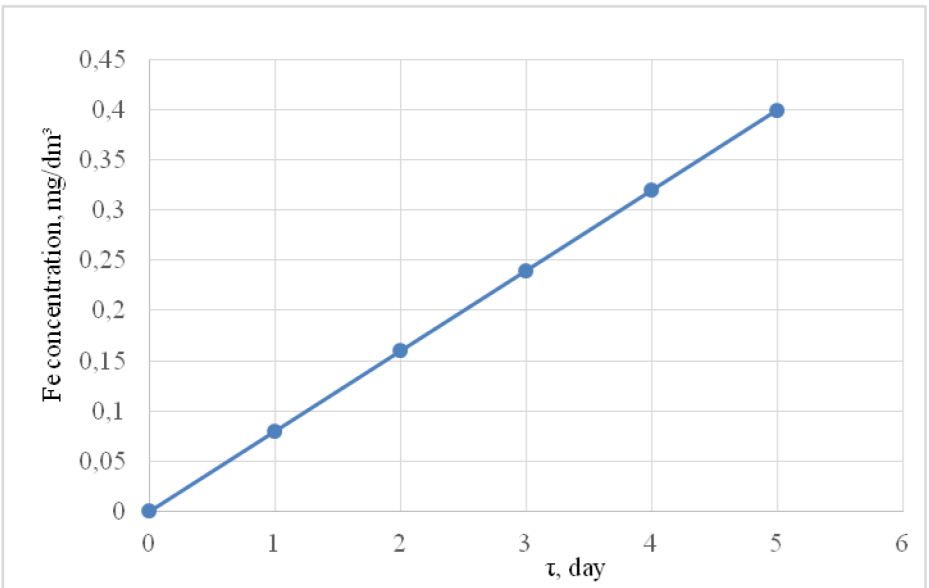

Fig. 2. Accumulation of ferrum compounds in a highly mineralized solution containing 15 mg/l hydrogen sulfide upon contact with hematite.

The task of the next experiment was to determine the rate of accumulation of soluble ferrum compounds upon contact of sulfide-containing solutions with brown clay (Fig. 3).

From the data presented, it can be seen that upon contact with clay, the dissolution of ferrum compounds occurs even more slowly, but in geological time it is still quite fast.

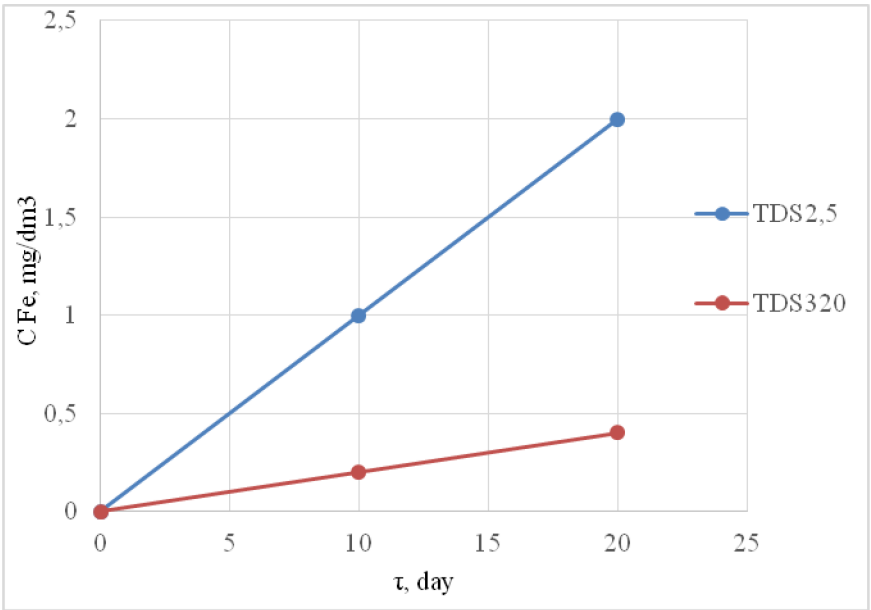

Fig. 3. Accumulation of iron compounds in solutions upon contact with brown clay.
1 – concentration of iron compounds in low-mineralized water;
2 – the same in highly mineralized brine.

The next task was to develop a technology for producing an inert metal oxide anode that would be stable in alkaline, neutral and acidic environments and, at the same time, would not contain noble metals and their compounds. The method for producing such anodes is based on the idea of combining the advantages of MnO_2 and PbO_2. It was necessary to apply a thin layer of MnO_2 to the titanium base, the purpose of which was to prevent passivation of the anode by titanium dioxide, and then cover the electrode with a thick layer of active PbO_2 coating.

Since alkaline electrolytes for electrodeposition of PbO_2 are kinetically unstable, a nitrate electrolyte was taken as the basic option. However, it was found that PbO_2 deposition does not occur on the MnO_2 surface even at high current densities. This is explained by the greater positivity of the electrode potential of the PbO_2 deposition process (+1.288 V) compared to the oxygen evolution potential (+1.053 V). Instead, PbO_2 begins to be released from the alkaline complex electrolyte before reaching the oxygen evolution potential (+0.22 V and +0.41 V, respectively). Thus, at low current densities, PbO_2 is deposited on the manganese dioxide surface without oxygen evolution. Therefore, it is necessary to cover the manganese dioxide surface with a lead dioxide deposit deposited

from the alkaline complex solution, and then apply a base layer of PbO_2 coating from the nitrate electrolyte.

The reasons for the kinetic instability of alkaline electrolytes were established. In an alkaline complex electrolyte containing a high concentration of the complexing agent EDTA, during the oxidation of plumbite ions to $HPbO^{3-}$ plumbate at the anode, the latter partially dissolves in the electrolyte. At the same time, the concentration of plumbates in the solution increases, and when it reaches saturation, which we experimentally established at 75×10^{-5} mol/dm^3, plumbates interact with plumbites according to the reactions:

$$2HPbO_2^- + HPbO_3^- = Pb_3O_4\downarrow + 3OH^-;$$
$$HPbO_2^- + HPbO_3^- = Pb_2O_3\downarrow + 2OH^-,$$

forming bottom sediments.

The study of the kinetics of plumbates accumulation during electrolysis in a conventional cell and in a cell with a catholyte separated from the main solution by a cation-exchange membrane showed that upon contact with the cathode and cathode lead (which is released during electrolysis), the accumulation of plumbates in the electrolyte occurs several times slower. The process of plumbates recovery upon contact of the electrolyte with the active surface of metallic lead was investigated (Fig. 4), the experimental results were processed according to the kinetic equation of the first order and the criterion of kinetic stability of the alkaline complex electrolyte during electrodeposition of PbO_2 was derived:

$$\frac{S_{Pb}}{S_A} = \frac{K_i}{K'C}, \qquad (1)$$

where and is the electrolysis current density, A/m^2,

C is the concentration of plumbates in the electrolyte, mol/dm^3.

This criterion is actually the ratio of the areas of the anode and the metallic lead immersed in the solution, and under normal deposition conditions (i = 100 A/m^2, C = $37.5*10^{-5}$ mol/dm^3) it is equal to 5.99. Therefore, if the surface of the metallic lead immersed in the electrolyte during the electrodeposition of PbO^2 is 6 times larger than the area of the anode, the formation of bottom deposits will not occur.

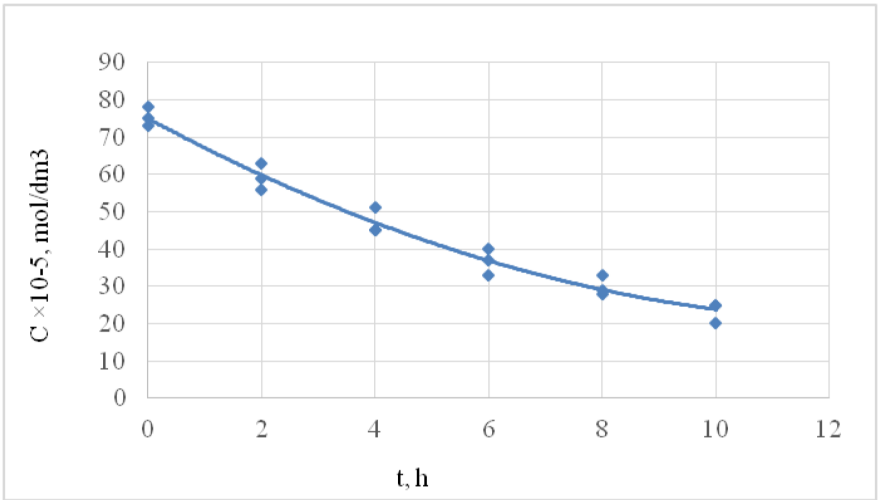

Fig. 4. Kinetics of plumbates reduction on the active surface of metallic lead.

After the MnO_2 sublayer deposited on titanium by thermal decomposition of manganese nitrate is completely covered with a thin PbO_2 layer, further lead dioxide deposition can be carried out at high current densities from a nitrate electrolyte.

Interruption of the PbO_2 electrodeposition process for more than 10 minutes leads to passivation of the oxide surface. Therefore, the next electrodeposition cycle begins with the appearance of new nuclei of lead dioxide crystals, and thus, the pores of the first coating layer are partially covered by another layer. This increases the stability of the anode and its service life.

Samples of bench anodes with an area of 2 dm^2 were obtained, and the extraction of sulfides and iron compounds from the brine of the Dombrovsky quarry was performed on these anodes (Fig. 4). The electrolysis voltage was 4.5 V, at a current density of 1000 A/m^2. The indicated graphs show that during electrolysis, sulfides are first oxidized to sulfates and the pH of the brine decreases from 5.8 to 5.3. After that, when more positive potentials are reached, the ferric compounds are oxidized to form trivalent ferric hydroxide in the form of flakes, which are easily separated from the brine when it is clarified by filtration. The estimated electricity consumption for the oxidation of ferric and sulfides in the brine with a mineralization of 350 g/dm^3 does not exceed 1 kWh/m^3.

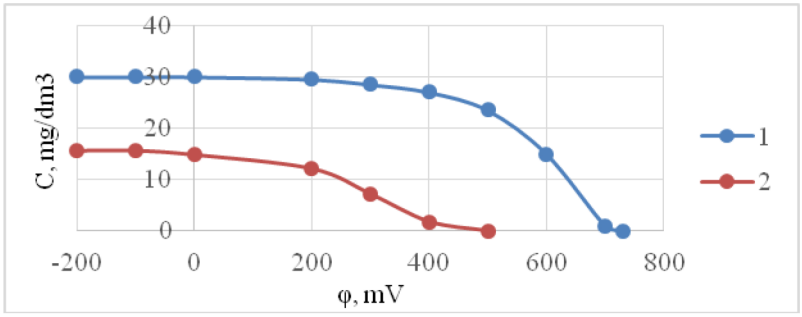

Fig. 5. Dependence of the residual concentration of ferrum compounds (row 1) and sulfides (row 2) on the redox potential of the solution during the electrolysis of the brine of the Dombrovsky quarry.

After the extraction of ferrum compounds and sulfides, the brine was subjected to galurgic separation, during which technical sodium chloride, valuable potassium fertilizer, crystalline magnesium chloride and purified water with an electrical conductivity of 3–5 μS/cm (residual mineralization 1.5–2.5 mg/dm^3) were obtained, which after further purification is suitable for use as feed water for power boilers. The further purification of water by electrodeionization on a laboratory electrodeionizer allowed to obtain deionized water with a residual electrical conductivity of 0.2 μS/cm at an additional electricity consumption of 0.1 kWh/m^3.

CONCLUSIONS. A mechanism for the accumulation of soluble iron compounds during the flow of water and brines through oxidized iron-bearing rocks has been proposed and experimentally proven. Based on this mechanism, a method for the extraction of iron compounds

from brines and highly mineralized waters by electrolysis with an inert anode has been developed. A technology for obtaining inert metal oxide anodes that do not contain noble metals or their compounds has been developed. Such anodes can also be used for water treatment and wastewater treatment, including in the production of composite fuels, for the removal of sulfides from municipal wastewater, which will improve their further treatment, the creation of closed technological cycles by hydrolysis of salt solutions and other needs. These anodes are used to remove sulfides and iron compounds from highly mineralized waters before their demineralization to obtain purified water for the needs of the energy industry (electrical conductivity 2.5–5 μS/cm) and salt commodity products. The use of electrodeionization allows the obtained water to be further purified to a residual mineralization of 0.2 μS/cm, which meets the standards for feed water for power boilers. The additional electricity consumption in this case does not exceed 0.1 kWh/m^3.

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МЕХАНІЗМ НАКОПИЧЕННЯ СПОЛУК ФЕРУМУ У ВИСОКОМІНЕРАЛІЗОВАНИХ ШАХТНИХ ТА КАР'ЄРНИХ ВОДАХ ТА ОЧИЩЕННЯ ЇХ ЕЛЕКТРОЛІЗОМ

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Розглянуто механізм накопичення у підземних водах та розсолах розчинених сполук феруму під впливом сульфатредукції та викликаного нею зниження окисно-відновного потенціалу. Розглянутий механізм експериментально підтверджено. Запропоновано спосіб вилучення сульфідів та ферумвмісних сполук електрохімічним окисненням із використанням інертного аноду. Описано технологію одержання інертного аноду на основі оксидів мангану та плюмбуму, стійкого у більшості природних вод та розсолів, який не містить благородних металів та їхніх сполук. Зазначена технологія включає осадження на титанову основу манган діоксиду шляхом термічного розкладання манган нітрату. Після цього основа покривається тонким шаром PbO_2 шляхом електроосадження з лужного комплексного електроліту, а потім – товстим шаром того ж оксиду шляхом електроосадження з нітратного електроліту. Встановлено, що контакт лужного комплексного електроліту з активною поверхнею металевого плюмбуму дозволяє істотно зменшити утворення донних відкладень при електроосадженні. Показано, як переривання струму при електроосадженні такого аноду може зменшити його пористість та збільшити стійкість. Досліджено процес знезалізнення та вилучення сульфідів із високомінералізованих шахтних та кар'єрних вод. Теоретич-

но розраховано та експериментально підтверджено, що витрати електроенергії на процес знезалізнення таких вод не перевищують 1 кВт*год/м^3. Наведено приклад застосування способу електрохімічного знезалізнення та вилучення сульфідів із високомінералізованих розсолів із подальшим їхнім ресурсозберігаючим переробленням та одержанням води для живлення об'єктів енергетики та товарних мінеральних солей.

Ключові слова: сульфатредукція, знезалізнення, очищення від сульфідів, електроліз, шахтні води та розсоли.

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