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STUDYING THE PROCESSES OF SULPHATES AND CHLORIDES EXTRACTION FROM WATER AT LOW-WASTE WATER DEMINERALIZATION TECHNOLOGY

Introduction. The coal mining companies are pumping and discharging into environment the untreated mine-pit and quarry waters that results in a negative impact on natural water reservoirs, and respectively involves a continued reduction in water resources' stocks and quality. The only one mine's untreated water discharge annually produces the environment contamination with more than 26 tons of iron, 1,300 tons of sulphate, 876 tons of chlorides, 175 tons of suspended substances. This problem is particularly acute for the eastern and southern Ukraine regions, where local resources are insufficient to cover the need for quality water. Therefore, the situation implies involving the green technology solutions for mine water treatment problem.

Until recently, the most common water treatment technology was ion-exchange one, but it has an important drawback, namely the use of a large reactants' number for regeneration, followed by discharge of mineralized drain water into surface waters. Consequently, this technology application is rational only under condition of effective regeneration techniques development and reduced volume of regeneration solutions used, the third contributive factor being those solutions' subsequent processing.

The currently prevailing water treatment technology is reverse osmosis technique, as its application practically excludes the use of reactants, essentially reducing the amount of salt discharged into surface waters; the reverse osmosis is very effective due to high selectivity membranes. But to prevent water resources pollution necessary is to develop solutions for mineral concentrates treatment.

Therefore the problem of mine water demineralization and high mineralization index concentrates' treatment is an urgent one.

Literature review. The treatment of baromembrane water demineralization concentrates containing only sulphates, hydrocarbons and hardness ions can be reduced to reactant-aided precipitation of sulfate ions and hardness ions in the form of calcium carbonate, magnesium hydroxide and calcium hydroxyl-alumosulphate. However, in the presence of chlorides such demineralization may not be possible because the chlorides in such case shall not be separated of the water. Electrolyzing the solutions containing chlorides and sulphates we can obtain the sulfuric acid and active chlorine [1]. But at that the sulfuric acid contains chlorides (HCl) and active chlorine admixtures. Besides, the active chlorine catching and treatment is a complex process. The concentrates resulting from sulphates' separation at natural and waste water demineralization, can contain apart of hardness ions the sodium cations, chlorides and bicarbonates. The solution containing only chloride anions, carbonates and bicarbonates gives such electrodialysis products as alkali and hydrochloric acid or sodium hypochlorite [2, 3]. In such a way, the separation of chloride and sulfate represents a complex issue and needs to be addressed through new efficient technologies development.

Aim of the Research. Investigation of ion exchange separation of sulfate and chloride with high-basic anionite Cl^- species at regeneration solutions efficient treatment for their reiterative use, followed

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with solutions' desalination at reverse osmosis plant, and resulting concentrates' demineralization thus obtaining chlorine-containing compounds for water disinfection.

Main Body. To remove sulphates from the model solution, chemically similar to the content of St. Isaac water reservoir in Alchevsk city: Hardness = 8,8 mg-eq/dm³, [Ca²⁺] = 4,3 mg-eq/dm³, [Mg²⁺] = 4,5 mg-eq/dm³, Alkaline hardness = 3,45 mg-eq/dm³, pH = 8,17, [SO₄²⁻] = 643,2 mg/dm³, [Cl⁻] = 110,05 mg/dm³, the anionite AV-17-8 Cl⁻ species has been used, volume applied: 20 cm³. The solution flow at sorption process amounted to 10...15 cm³/min. Every sample taken for further analysis of chlorides and sulphates content had 100 cm³ volume. The exchange dynamic capacity prior to break-through (EDC_{b,b}) and the total dynamic exchange capacity of the ion exchanger resin (TDEC) will be, based on the mass of anionite-adsorbed ions:

$$EDC_{b,b} = \frac{\sum_{i=1}^m (C_{init.} - C_i) V_{sample}}{V_i},$$

$$TDEC = \frac{\sum_{i=1}^n (C_{init.} - C_i) V_{sample}}{V_i},$$

where $C_{init.}$ — solution initial ions concentration, mg-eq/dm³;

C_i — ions concentration at i^{th} sample after sorption, mg-eq/dm³;

V_{sample} — sample volume, cm³;

V_i — ionite volume, cm³;

m — number of samples taken before sulphates' break-through;

n — number of samples taken before ionite capacity exhausted.

To demineralize the obtained solution a reverse osmosis membrane Filmtec TW30-1812-50 has been chosen. After passing through reverse osmosis filter (permeate section class 93,75 %) the obtained concentrate had the characteristics: Hardness = 66 mg-eq/dm³, [Ca²⁺] = 24 mg-eq/dm³, [Mg²⁺] = 42 mg-eq/dm³, [Cl⁻] = 110 mg-eq/dm³ and pH = 8,8.

The reverse osmosis water desalination process has been performed using 16 dm³ of model solution. The water was pumped into a cassette equipped with reverse osmosis membrane. Concentrate being drained back in the original solution container the permeate has been taken into in a separate container. The system pressure was supported by control valve regulating the sampled concentrate dosage. After each dm³ of demineralized solution extraction, both the permeate and the concentrate were analyzed for content of chlorides, sulphates, hardness ions and pH. The permeate extraction stages varied from 6,25 to 93,75%.

At the sulphates' content assessment the photometric method was applied and as to chlorides the Mohr's method has been used. In assessing the hardness and calcium content parameters, we followed the standard methods.

So, the membrane selectivity by components:

$$R = \frac{C_0 - C_{perm.}}{C_0} \cdot 100,$$

where C_0 , $C_{perm.}$ — concentration, respectively, at the initial solution and the permeate.

Every subsequent to the first one sample taking, we considered the increase in processed fluid's component concentration at the expense of concentrate recirculation.

The membrane productivity (transmembrane flow velocity) is:

$$J = \frac{\Delta V}{S \Delta t},$$

where ΔV — volume (dm³) of permeate, passed through membrane having area S , m² during extraction time Δt , hours.

When high-hardness and high chlorides content water baromembrane separation, the concentrates are characterized by high contents in chlorides as well as calcium and magnesium ions. Therefore, for these solutions' electrochemical treatment designed is a bi-chamber electrolyzer with anionic membrane MA-41.

In quality of aqueous media treated by electrodialysis, method we selected the reverse osmosis concentrate resulting from desalination procedure and the concentrate pre-softened using reactant-involving methods.

Here the cathode represents a plate of stainless steel 12X18H10T; the anode is a titanium plate coated with ruthenium oxide. The electrodes' area $S_C = S_A = 0,16 \text{ dm}^2$. Electrolysis performed at 0,5 A current:

— The processed solution has been placed at the cathode area; the anode chamber contained the 0,01-normal NaCl solution. While electrolysis the chlorides content in the cathode zone and the active chlorine level in the anode zone were controlled at regular intervals;

— At unprocessed concentrate placing in the anode area, the 1,0-normal solution of NaOH has been placed in the cathode area. While electrolysis the alkalinity in the cathode zone, chlorides content, active chlorine and hardness in the anode zone were controlled at regular intervals.

The current yield was calculated as the ratio of the chlorides and sulphates actually extracted from solution to the index theoretically calculated by Faraday's law [4].

Results. The effectiveness of chlorides and sulphates ion-exchange separation using the anionite AV-17-8 (Cl⁻ type) can be assessed considering the initial sorption curves, as shown at Fig. 1. As we can see, at initial solution concentration in sulphates of 13,4 mg-eq/dm³, the amount of solution passed before the sulphates' break-through was 1,6 dm³, at that the EDC_{b,b.} = 1,07 g-eq/dm³ and TDEC = 1,61 g-eq/dm³. After the model solution anionitic filtration (Cl⁻ type) the chlorides' concentration increased from 3,1 to 16,5 mg-eq/dm³.

When regeneration solutions treatment with calcium chloride reactant the sulphates are deposited in the form of gypsum, which after drying and sintering can be used at building materials manufacturing. The processes of multiple reiterative use of anionite AV-17-8 (Cl⁻ type) restored regeneration solutions are confirmed experimentally. Advisable is to clean the water containing chlorides and hydrocarbonates, hardness ions and sodium cations with reverse osmosis technique.

Results for demineralization by reverse osmosis using membrane filter Filmtec TW30-1812-50 are shown at Fig. 2, 3. As Fig. 2 represents, hardness ions are effectively removed from water. By the extraction level of 87 % the permeate hardness is below 1 mg-eq/dm³. Only reaching the permeate extraction level of 94 % its hardness index augments to 5,4 mg-eq/dm³ at concentrate hardness of 66 mg-eq/dm³. The effectiveness of chlorides' extraction from water is satisfactory up to extraction level of 87 %. Summarizing, (Fig. 3), the increase in permeate extraction degree is concurring to decrease in membrane efficiency that reduced in respect of both hardness ions and chlorides. This is due to the concentrate augmented mineralization when permeate extraction increases.

Since the efficiency of water treatment depends not only on the remaining, but also on the initial ions concentration, when water treatment efficiency assessing we should consider the membrane selectivity. As Fig. 2 represents, in model solution treatment the membrane selectivity as to hardness and calcium ions reaches ~99 % at permeate extraction level 87 % and only the permeate extraction level exceeding 90% it drops up to 91...92 %. The selectivity in respect of chlorides is much lower — 90...92 %, dropping to 78 % with the permeate extraction level reaching 94 %.

The extraction level increased during water treatment, the permeate's pH raises from 7,15 to 8,3 and the concentrate's pH respectively, from 8,2 to 8,8. Increase in concentrate pH from 8,17 to 8,20 (initial solution) and then to 8,8 in the concentrate can be attributed to that the membrane selectivity to

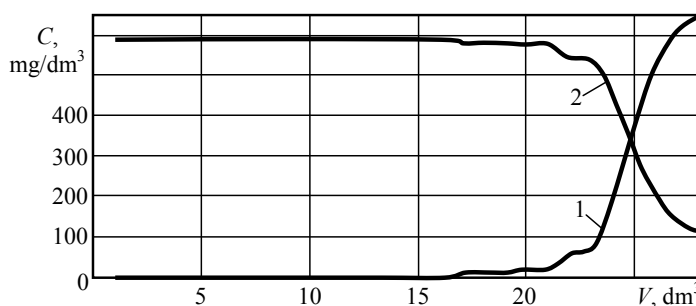


Fig. 1. Dependence between sulphates' and chlorides' concentration and the volume of solution passed through the AV-17-8 anionite (Cl⁻ type) ($V = 20 \text{ dm}^3$): $[\text{SO}_4^{2-}]$ (1), $[\text{Cl}^-]$ (2)

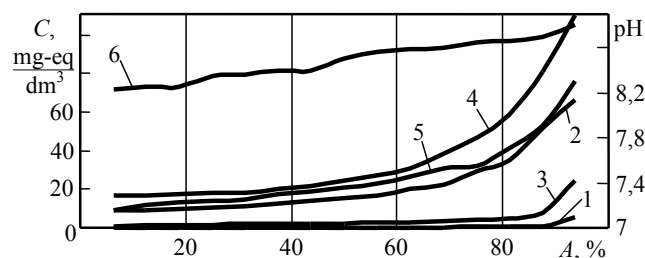


Fig. 2. Changing of permeate's and concentrate's characteristics upon the permeate extraction level when water RP treatment: Hardness (1), (2); $[Cl^-]$ (3), (4); pH (5), (6); permeate (1), (3), (5); concentrate (2), (4), (6)

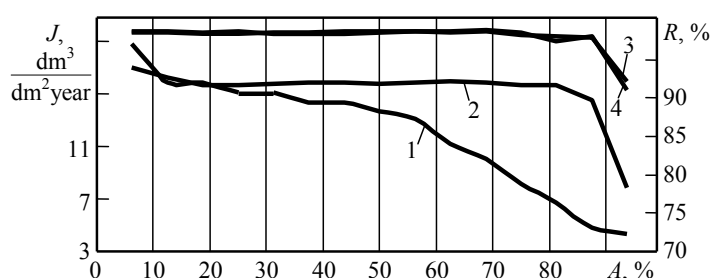


Fig. 3. The reverse osmosis membrane's efficiency and selectivity dependence upon the permeate extraction level when water RP treatment: efficiency (1); selectivity: $[Cl^-]$ (2); hardness (3); $[Ca^{2+}]$ (4)

hardness ions exceed the selectivity to hydrocarbonates [5], that involves increased hardness ion concentration in the concentrate compared to hydrocarbon concentration. Due to the larger transmembrane hydrocarbon passage capacity when compared to hardness ions, the pH decreases. Further, with concentrate pH increase the permeate pH augments concurrently, but it is always lower than the concentrate's pH.

As a result of water treatment at the permeate extraction level 94% we obtained the concentrate which volume is only 6% of the initial water volume, having hardness index of 66 mg-eq/dm³ and chlorides content of 110 mg-eq/dm³. These solutions' evaporation will be uneconomical. To process these concentrates with high chlorides' content, at the absence of sulphates and nitrates we can apply the electrodialysis methods obtaining alkali and hydrochloric acid or a mixture of oxidized chlorine compounds for water sterilization [2, 3].

However, when high solutions' hardness, this process is difficult to implement because of hardness ions poisoning cationic membranes or involving low-solubility cathode deposit formation, that significantly slows the electrolysis process and results in electricity significant overconsumption [6, 7]. Therefore, the solutions undergo preliminary softened using reactant method. As we observe from tables 1 and 2, the softening is more efficient when sodium carbonate and alkali adding, rather than lime and sodium carbonate. The softening effectiveness augments with increasing NaOH doses at stoichiometric amount of sodium carbonate.

Obviously, the solutions with hardness index of 0,25...0,95 mg-eq/dm³ can be treated in a four-chambers electrolyzer obtaining hydrochloric acid and alkali [6, 7] as resulting products.

Apart of hydrochloric acid separation, these concentrates can be processed to obtain oxidized chlorine compounds, including active chlorine, sodium hypochlorite, sodium chlorite and chlorate.

Results for chlorine oxidation in two-chamber electrolyzer (anionic membrane MA-41) when softened concentrate placed at cathode area are shown below (Fig. 4). The oxidation occurs at the anode chamber, filled initially with of 0,01-normal sodium chloride solution. In the cathode chamber takes place alkali separation process concurrently to reduction in chlorides concentration due to their diffusion into anode area. Experiment running, the processed solution (catholyte) content in chlorides dropped to 7 mg-eq/dm³. The anode area meanwhile accumulates oxidized chlorine compounds that can be monitored by the iodine displacement from potassium iodide solution. The maximum content of oxidized chlorine reaches 112 mg-eq/dm³. But after 75 minutes of electrolysis, its concentration decreases to 84 and further to 38 mg-eq/dm³. This can be explained by the gaseous chlorine compounds evolving and release from the anode chamber as described by reactions

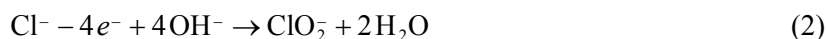


Table 1

Water softening efficiency dependence onto amounts of sodium carbonate and alkali added

№	Dose of Na ₂ CO ₃ , mg-eq/dm ³	Dose of NaOH, mg-eq/dm ³	Hardness, mg-eq/dm ³	Ca ²⁺ , mg-eq/dm ³	Alkalinity, mg-eq/dm ³	pH	Softening degree, Z, %
1	24,0	42,0	2,50	1,10	4,29	11,27	96,21
2	24,0	44,1	1,75	0,80	5,56	11,02	97,35
3	24,0	46,2	0,95	0,67	8,58	11,76	98,56
4	24,0	50,4	0,70	0,40	11,93	12,16	98,94
5	24,0	54,6	0,38	0,31	14,16	12,36	99,43
6	24,0	58,8	0,38	0,27	18,88	12,45	99,43
7	24,0	63,0	0,25	0,23	24,30	12,55	99,62
8	25,2	42,0	4,40	2,00	9,00	10,98	93,33
9	26,4	42,0	4,40	1,40	9,50	11,1	93,33
10	28,8	42,0	5,02	1,5	9,67	11,05	92,39
11	31,2	42,0	4,00	1,25	9,33	11,06	93,94
12	33,6	42,0	3,00	0,65	10,5	11,2	95,45
13	36,0	42,0	2,20	0,55	10,25	11,38	96,67

Table 2

Water softening efficiency dependence onto amounts of sodium carbonate and lime added

№	Dose of CaO, mg-eq/dm ³	Dose of Na ₂ CO ₃ , mg-eq/dm ³	Hardness, mg-eq/dm ³	Ca ²⁺ , mg-eq/dm ³	Alkalinity, mg-eq/dm ³	pH	Softening degree, Z, %
1	42,00	66,00	6,80	6,40	5,60	12,2	89,70
2	42,00	69,30	3,40	3,40	4,60	12,10	94,85
3	42,00	72,60	1,10	1,10	5,80	12,06	98,33
4	42,00	75,90	0,80	0,60	7,40	12,16	98,79
5	42,00	79,20	0,23	0,60	10,75	12,17	99,65
6	44,10	66,00	4,76	7,70	8,06	12,32	92,79
7	46,20	66,00	8,20	9,80	7,70	12,36	87,58
8	48,30	66,00	9,20	9,40	8,30	12,38	86,06
9	50,4	66,00	9,00	9,00	7,70	12,34	86,36

Generally these results are not significantly high when oxidizing solution for water sterilisation obtaining. The current yield on chlorides' diffusion and active chlorine production is low.

When placing the treated solution (softened concentrate) in the anode chamber and filling the working chamber with 1-normal alkali solution maintaining the same parameters electrolysis the obtained concentration of oxidized chlorine compounds makes 120...198 mg-eq/dm³ (Fig. 5), that exceeds the initial solution chlorides concentration as the chlorine

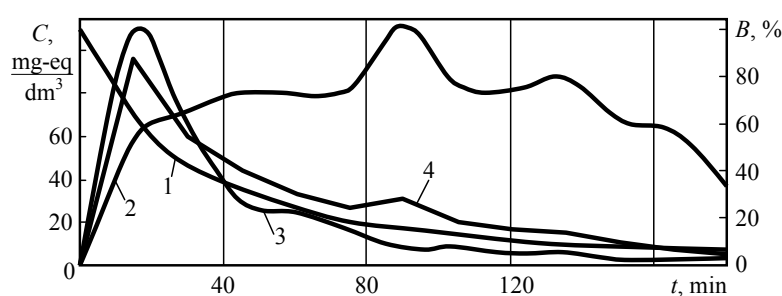


Fig. 4. Dependency of cathode area chlorides' concentration, anode area active chlorine (oxidized chlorine compounds) content, current yield when chlorides' passage from cathode zone and active chlorine current yield upon the electrolysis time of softened concentrate (Hardness = 1,0 mg-eq/dm³, Alkalinity = 8,58 mg-eq/dm³, [Cl⁻] = 110 mg-eq/dm³ and pH = 11,76) placed at the cathode zone of two-chambers electrolyser (membrane MA-41, anode zone filled with 0,1-n NaCl, I = 0,5 A): [Cl⁻] (1); [Cl₂] (2); [B_{Cl⁻}] (3); [B_{Cl₂}] (4)}}

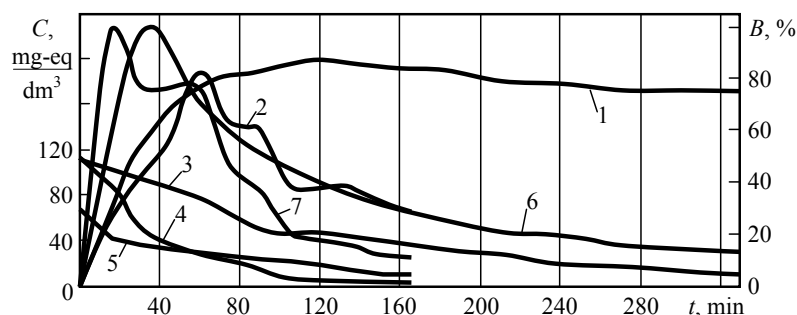


Fig. 5. Dependency of concentration in active chlorine, chlorides, current yield as to active chlorine at the concentrate (2), (4), (5), (7) and the softened concentrate (1), (3), (6) onto the duration of electrolysis at current density $3,17 \text{ A/dm}^2$ ($I = 0,5 \text{ A}$): $[\text{Cl}_2]$ (1), (2); $[\text{Cl}^-]$ (3), (4); hardness (5); $[B_{\text{Cl}_2}]$ (6), (7);

oxidizes not only up to active chlorine specie, but reaching higher degrees of oxidation compounds ClO^- , ClO_2^- and, perhaps, ClO_3^- . The resulting solution may represent a promising one for water disinfection.

It is interesting to note that similar results have been obtained when used in the anode zone the concentrate without softening reactant (Fig. 5). As the operated solution is separated from the cathode region with anionic membrane the hardness ions have no influence on the

cathodic process. In this case, there is a significant reduction in the concentrate's chlorides content, and the concentration of oxidized chlorine compounds reaches $\sim 188 \text{ mg-eq/dm}^3$. The current yield is also high while time-dependent dropping from 97 to 11 %, due to a decrease in solution chlorides concentration, and to the loss of chlorine as active chlorine and chlorine dioxide: reaction (1)...(3). As solution pH increases from 8,8 to 10,9, we observe the decrease in its hardness.

Therefore we conclude that the ion-exchange chlorides and sulphates separation at the stage of water pre-treatment before reverse osmosis application does simplify the technology of concentrates utilization.

Conclusions. Identified and studied are the conditions of ion-exchange water desulphatization and efficient separation of sulphates and chlorides at the preliminary water treatment stage. It is shown that concentrates resulting from the reverse osmosis desalination of previously desulphatized water are effectively softened with reactants that allow further electro dialysis to produce demineralised water and hydrochloric acid and alkali. Identified and assessed are the parameters of softened and high hardness chlorine-containing concentrates treatment by electro dialysis with obtaining solutions of oxidized chlorine compounds suitable for water disinfection.

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АНОТАЦІЯ / АННОТАЦИЯ / ABSTRACT

І.М. Трус, І.М. Макаренко, Т.О. Шаблій. **Вивчення процесів вилучення з води сульфатів та хлоридів для маловідходної технології знесолення води.** Для вирішення проблеми утилізації рідких відходів з підвищеним солесмістом, які утворюються при знесоленні води, досліджено процеси іонообмінного розділення хлоридів та сульфатів з вилученням сульфатів із технологічного циклу у вигляді сульфату кальцію. Показано, що очищену від сульфатів воду можна ефективно опріснювати на зворотньоосмотичних фільтрах, включаючи і мембрани низького тиску Filmtec TW30-1812-50. Отримані в якості рідких відходів концентрати містять хлориди, іони натрію та іони жорсткості. Встановлено, що при обробці таких концентратів вапном та содою або лугом та содою відбувається їх пом'якшення при зниженні жорсткості до 0,25...0,95 мг-екв/дм³, що дозволяє в подальшому переробляти дані розчини електролізом з отриманням луку та соляної кислоти. При безпосередньому електролізі отриманих при зворотньоосмотичному опрісненні води концентратів в двохкамерних електролізерах з аніонною мембраною отримано дезинфікуючий розчин, що містить окислені сполуки хлору (активний хлор, гіпохлорит, хлорит та хлорат натрію) і є ефективним при знезараженні води. Отриманий розчин добре зберігається і є перспективним для знезараження природних та стічних вод.

Ключові слова: зворотній осмос, іонний обмін, електроліз, пом'якшення, знезараження води, сульфати, хлориди.

І.Н. Трус, І.Н. Макаренко, Т.А. Шаблій. **Изучение процессов извлечения из воды сульфатов и хлоридов для малоотходной технологии обессоливания воды.** Для решения проблемы утилизации жидких отходов с повышенным солесодержанием, которые образуются при обессоливания воды, изучены процессы ионообменного разделения хлоридов и сульфатов с изъятием сульфатов из технологического цикла в виде сульфата кальция. Показано, что очищенную от сульфатов воду можно эффективно опреснить на обратноосмотических фильтрах, включая и мембраны низкого давления Filmtec TW30-1812-50. Полученные в качестве жидких отходов концентраты содержат хлориды, ионы натрия и ионы жесткости. Установлено, что при обработке таких концентратов известью и содой или щелочью и содой происходит их умягчение при снижении жесткости до 0,25...0,95 мг-экв/дм³, что позволяет в дальнейшем перерабатывать данные растворы электролизом с получением щелочи и соляной кислоты. При непосредственном электролизе полученных при обратноосмотическом опреснении воды концентратов в двухкамерных электролизерах с анионной мембраной получен дезинфицирующий раствор, содержащий окисленные соединения хлора (активный хлор, гипохлорит, хлорит и хлорат натрия), являющийся эффективным при обеззараживании воды. Полученный раствор хорошо сохраняется и является перспективным для обеззараживания природных и сточных вод.

Ключевые слова: обратный осмос, ионный обмен, электролиз, умягчение, обеззараживание воды, сульфаты, хлориды.

I.M. Trus, I.M. Makarenko, T.O. Shabliy. Studying the processes of sulphates and chlorides extraction from water at low-waste water demineralization technology. To solve the disposal problem of high-salinity liquid wastes resulting from the water demineralization, researched are the processes of chlorides' and sulphates' ion-exchange separation with further sulphates (in the form of calcium sulphate) removal from the technological cycle. It is shown that the desulphatized water can be effectively desalinated by reverse osmosis filters, including low-pressure membranes Filmtec TW30-1812-50. The liquid waste obtained in form of concentrates, does contain chlorides, sodium ions and hardness ions. Established is that at these concentrates processing by lime and sodium carbonate or alkali and sodium carbonate they are softened with hardness decrease up to 0,25...0,95 mg-eq/dm³, which allows these solutions' further electrolysis to obtain alkali and hydrochloric acid. Through direct electrolysis of concentrates, obtained by reverse osmosis water desalination at anionic membrane two-chamber electrolyzers, we obtained a disinfectant solution containing chlorine oxigenates (active chlorine, hypochlorite, chlorite and sodium chlorate) and duly effective in water sterilization. The resulting solution well keeps its properties and is promising for disinfection of natural and waste waters.

Keywords: reverse osmosis, ion exchange, electrolysis, water demineralizing, water sterilization, sulphates, chlorides.

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