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ЕКОЛОГІЯ ТА РЕСУРСОЗБЕРЕЖЕННЯ

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PRODUCTION OF ACTIVE CHLORINE AND SODIUM HYPOCHLORITE IN A CLOSED ELECTROLYSER

Today, in the conditions of warfare, there is constant local pollution of the environment due to the use of explosives, fires, spillage of fuel and lubricants, etc. Every day, the danger increases when the water drainage systems of various enterprises are damaged, such as mining enterprises, facilities of the energy, metallurgy, chemical, petrochemical industries, sludge storage facilities for highly toxic waste. Next to this, there is a growing problem of highly efficient purification of water, primarily drinking water. As a result of the destruction of the Kakhovsky reservoir in 2023, the problem of disposal of mine waters, which are characterized by a high level of mineralization, a significant content of iron, manganese and other toxicants, has sharply worsened. In this case, there are no water resources for diluting mine waters before their discharge. Despite a significant number of developments in the field of water quality control and water purification technologies, a number of problems in this direction remain unresolved. Existing technologies are ineffective when the levels of mineralization and water hardness are exceeded. We are talking about the southern regions of our country and Donbas. In Mykolaiv, after the aggressor reduced the water intake on the Dnieper, the centralized water supply system supplies brackish water from the estuary. It is known that concentrated salt solutions, which are formed and are still present next to the problems described above, are practically not processed. In most cases, they are dumped into the surface reservoirs of the surrounding natural environment, which significantly worsens the state of water ecosystems of Ukraine. The situation is worsened by the fact that a significant part of mine water suitable for use, due to the problem of disposal of concentrates, is not used, but is discharged into the environment after dilution, or even without dilution and purification, since there is simply no resource for dilution in today's realities.

That is why, the problem of processing concentrates of baromembrane purification with obtaining secondary useful products was the aim of the research of this work.

Keywords*: water desalination, water treatment waste processing, active chlorine, sodium hypochlorite, electrolysis, catholyte, anolyte, ultrafiltration membrane, anion exchange membrane, current density, electrolyzer, absorber*

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Statement of the problem. The shortage of fresh water resources in Ukraine has recently been compensated by the usage of artesian water, surface water and mine water with high salinity. The use of such water in municipal services and industry is possible only after desalination. Reverse osmosis and nanofiltration are the main methods of desalination. The application of baromembrane methods is largely limited by the complexity of processing and utilisation of concentrates generated during water treatment.

Today, unfortunately, concentrated saline solutions are hardly ever recycled. In most cases, they are discharged into surface water bodies, which significantly worsens the state of Ukraine's aquatic ecosystems. The situation is aggravated by the fact that a significant part of mine water suitable for use is not used due to the problem of concentrate utilisation, but is discharged into the environment after dilution or even without dilution and treatment [1].

That is why the purpose of this study is the problem of processing baromembrane purification concentrates to obtain secondary useful products.

Analysis of previous studies. Taking into account the large volumes of sodium chloride solution waste in Ukraine and the shortage of reagents for water disinfection, disinfection of other media, bleaching of pulp, paper, and fabric, it is advisable to use this waste to produce active chlorine, sodium hypochlorite, etc.

It would seem that this is not difficult. After all, the technology for producing active chlorine and other oxidised chlorine compounds is well established. However, in the production of oxidised chlorine compounds, saturated, pure sodium chloride solutions with a concentration of > 100 g/dm^3 are used. Moreover, the production of oxidised chlorine compounds is not aimed at complete removal of chlorides from water [2-3]. As a rule, the process is carried out in order to achieve the maximum current yield of the obtained active chlorine or sodium hypochlorite with partial extraction of chlorides from water, maintaining the concentration of sodium chloride in the electrolyzer at a given level by dissolving the dry reagent in the reaction mixture.

While recycling waste solutions from water desalination processes, the task is to extract sodium chloride from water as completely as possible to obtain useful substances and therefore it is simply impossible to comply with the conditions of classical processes for the production of oxidised chlorine compounds. In addition, with a decrease in the chloride content in water, the current output of oxidised chlorine compounds decreases significantly. Therefore, we have studied the processes of chloride oxidation in a two-chamber electrolyzer using diffusion, anionic (MA-41) and cationic (MK-40) membranes. To prevent significant losses of chlorine in the form of Cl_2 and ClO_2 during degassing, a sealed electrolyzer was used. Oxidised chlorine compounds were removed from the anode chamber, and hydrogen was removed from the cathode chamber [4-5].

When using a cationic membrane, an alkali solution was produced in the cationic chamber in parallel with active chlorine in the anode chamber.

The aim of the study was to determine the conditions for the electrochemical production of active chlorine and sodium hypochlorite in a closed electrolyzer while ensuring the efficient removal of chlorides from water at a high current yield of oxidized chlorine products to produce concentrated sodium hypochlorite solutions.

To achieve this goal, we need to solve the following tasks:

1. Determine the dependence of the efficiency of producing oxidised chlorine compounds on the type of membranes used in a sealed electrolyzer;
2. Determine the dependence of the desalination efficiency of sodium chloride solutions on the electrolysis time;
3. To estimate the dependence of the current output of active chlorine from a sodium chloride solution on the electrolysis time and anode current density when using a closed electrolyzer.

Methods. Electrolysis was carried out using sodium chloride solutions with a concentration of 150-200 g/dm^3. An ultrafiltration membrane UMP-20 was used as a diffusion membrane. In this case, the cathode and anode chambers were filled with NaCl solution [6]. The hydrogen from the cathode chamber was drained into a measuring cylinder filled with water to determine the volume of hydrogen released during electrolysis. A schematic diagram of the device for capturing hydrogen and active chlorine is shown in Fig. 1. The same figure shows an absorber filled with an alkali solution for the capture of active chlorine and chlorine dioxide.

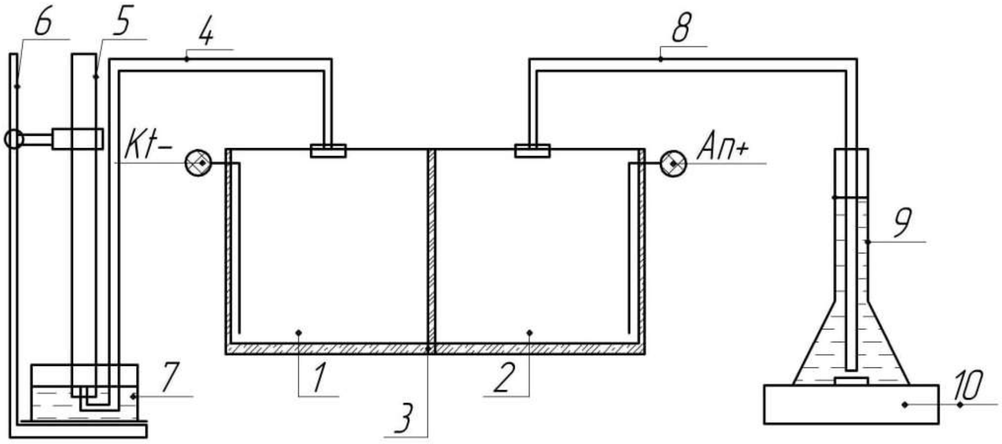
Electrolysis was carried out at a current density of 4.17-12.5 A/dm^2. Periodically, during the electrolysis, the concentration of chlorides and oxidised chlorine compounds was measured in the working solution, and the concentration of oxidised chlorine compounds in the absorber. The absorber was filled with 100 cm^3 of a 20 % NaOH solution. To improve absorption, the alkali solution was stirred with a magnetic stirrer. The efficiency of chloride removal from water was calculated using formula (1). The yield of oxidised chlorine compounds by current was calculated using formula (2) [7-9].

$$E = \frac{\text{Сп}-\text{Сз}}{\text{Сп}} \cdot 100, \% \qquad (1)$$

where $C_п$ is the initial concentration of sulphates in water, mg-eq/dm^3;
$C_з$ – residual sulphate concentration in treated water, mg-eq/dm^3;

$$B = \frac{qp}{q\text{т}} \cdot 100 = \frac{m\text{ф}}{m\text{т}} \cdot 100, \qquad (2)$$

q_p - the number of electricity for the substance actually transferred, or oxidised or reduced;
$q_т$ - theoretically calculated number of electricity;
$m_ф$ - - the number of substance actually transferred, g-eq;
m_T - theoretically possible number of transferred substance, g-eq.



1 – cathode chamber; 2 – anode chamber; 3 – membrane; 4 – flexible pipe for hydrogen removal; 5 – measuring cylinder for hydrogen capture; 6 – tripod; 7 – water tank; 8 – flexible tube for removing oxidised chlorine compounds; 9 – absorber (measuring flask) for capturing chlorine and ClO_2; 10 – magnetic stirrer; K_t – cathode; A_n – anode (titanium coated with ruthenium oxide)

Fig. 1 – Schematic diagram of a plant for the production of oxidised chlorine compounds from sodium chloride solutions

Electrolysis with MA-41 anionic membrane. When using an anionic membrane in a two-chamber closed electrolyzer, the anode chamber was filled with working solution, and the cathode chamber with 1 N alkali solution [10-11]. The concentration of sodium chloride was from 30 to 120 g/dm^3.

During the electrolysis, the alkalinity in the catholyte, pH, chloride content, active chlorine in the anolyte, and the concentration of active chlorine in the absorber were monitored.

The chloride extraction efficiency was calculated using formula (1), and the current output of active chlorine was calculated using formula (2).

Summary of the main material. The use of a membrane prevented the mixing of solutions with active chlorine with a catholyte saturated with hydrogen. At the same time, both the cathode and anode chambers were filled with sodium chloride solutions with a concentration of 150 and 200 g/dm^3. Hydrogen was drained from the cathode chamber into a container, which accumulated, displacing water from the measuring cylinder. The active chlorine was absorbed by a 20 % NaOH solution (100 cm^3) placed in a measuring flask (absorber). The concentration of active chlorine in the absorber and anolyte, as well as the chloride content in the electrolyte, was periodically measured. The results are shown in Fig. 2.

As can be seen from Figure 2, active chlorine accumulates in the electrolyte and the absorber. With a current density of 4.17 A/dm^2, the concentration of active chlorine in the electrolyte reached 340 mg-eq/dm^3 in 6 hours, and 224 mg-eq/dm^3 in the absorber. The current output of active chlorine generally reached ~70 %. The chloride concentration decreased from 2480 mg-eq/dm^3 to 1870 mg-eq/dm^3. At the same time, the total number of chlorides decreased by 159 mg-eq/dm^3. The number of synthesised active chlorine reached 110 mg-eq/dm^3. Obviously, a significant number of active chlorine is released with hydrogen from the catholyte, which leads to significant losses.

Similar results were obtained using a sodium chloride solution with a concentration of 200 mg-eq/dm^3 (Fig. 3). The anodic current density in this case reached 12.5 A/dm^2. At the same time, the concentration of active chlorine in the electrolyte reached 4350 mg-eq/dm in 10 hours^3, in the absorber – 410 mg-eq/dm^3. The output of active chlorine by current reached 73-74 %.

If we recalculate the concentration of active chlorine taking into account its conversion into sodium hypochlorite, the mass of hypochlorite reached 162 g/dm^3 or ~16%. In the absorber, the concentration of sodium hypochlorite reached 16.6 g/dm^3 or 1.6 %. The formation of hypochlorite in the electrolyte proceeded according to the following scheme: chlorine was released at the anode. Hydroxide anion was formed at the cathode.

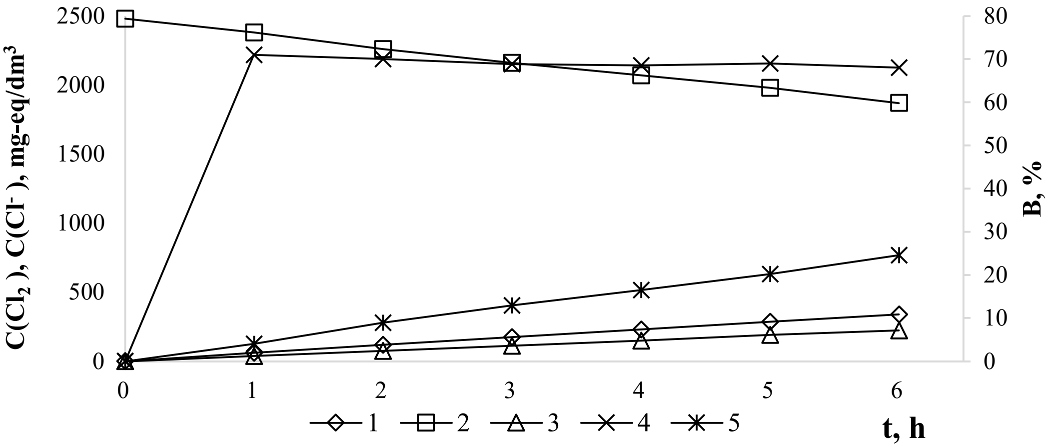


Fig. 2 – Dependence of the concentration of active chlorine (1), chlorides (2) in the anolyte, active chlorine in the absorber (3), current output of active chlorine (4), degree of chloride extraction (5) on the electrolysis time of sodium chloride solution (C(NaCl) = 150 g/dm^3) in a two-chamber electrolyzer (UPM-20 membrane) at a current density of j = 4.17 A/dm^2 (pH = 7.0)

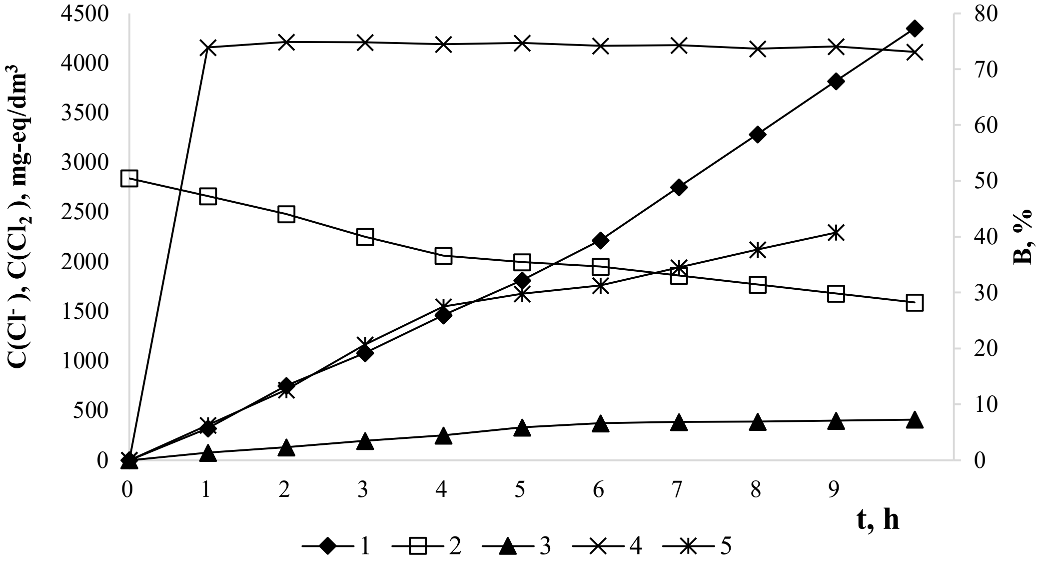


Fig. 3 – Changes in the concentration of active chlorine (1), chlorides (2) in the anolyte, active chlorine in the absorber (3), and active chlorine current output (4), degree of chloride extraction (5) with the time of electrolysis of sodium chloride solution (C(NaCl) = 200 g/dm^3) in a two-chamber electrolyzer (UPM-20 membrane) at a current density j = 12.5 A/dm^2 (pH = 6.0–7.0)

Alkali was formed with sodium ions, which interacted with active chlorine. But a significant number of active chlorine was removed from the electrolyte into the absorber during degassing with hydrogen. This should lead to alkalinisation of the solution. After all, hydroxide anions can be oxidised at the anode, which leads to the neutralisation of the solution:

With the total number of active chlorine produced at 1172 mg-eq, the number of chloride decreased by 1250 mg-eq. This indicates that ~78 mg-eq of Cl_2 was lost due to its degassing from the cathode region. The current yield of the generated hydrogen was close to 100%.

The disadvantage of the process is the degassing of chlorine from the catholite, which can be captured with an alkali solution, and the high mineralisation of the resulting sodium hypochlorite due to the significant residual sodium chloride content, which reaches ~93 g/dm^3.

When using an anionic membrane in a two-chamber electrolyzer, the anode chamber was filled with a solution of sodium chloride, and the cathode chamber with 1 N NaOH. Volatile products of chloride oxidation were removed from the anode chamber and hydrogen was removed from the cathode chamber. The content of active chlorine in the catholyte and absorber and the content of chlorides in the anolyte were periodically monitored. As can be seen from Figure 4, during electrolysis at a NaCl concentration of 120 g/dm^3, a significant number of active chlorine was removed from the anolyte and sorbed in the absorber.

The bulk of the active chlorine was accumulated in the anolyte. In total, 127 mg-eq of active chlorine (sodium hypochlorite) was formed and remained there. The absorber accumulated 9 mg-eq of sodium hypochlorite, and a total of 136.5 mg-eq of active chlorine was formed. The concentration of chlorides decreased by 70 mg-eq. The average pH in the anode area was 7.0-7.5.

No significant accumulation of alkali in the anolyte occurred due to the oxidation of hydroxide anions at the anode. The current yield of active chlorine, as in the previous case, reached ~ 73 %. Thus, the number of active chlorine formed in 5 hours at a current density of 8.33 A/dm^2 reached 136.5 mg-eq, and the decrease in the number of chloride reached 70.2 mg-eq.

Similar results were obtained during the electrolysis of a NaCl solution with a concentration of 120 g/dm^3 at a current density of 12.5 A/dm^2 (Fig. 5). In this case, in 5 hours, the concentration of active chlorine in the anolyte increased to 1445 mg-eq/dm^3, and in the absorber to 165 mg-eq/dm^3. In total, 204.35 mg-eq of active chlorine was obtained. The decrease in chloride concentration reached only 70 mg-eq.

Obviously, at high anode current densities, chloride oxidation does not stop at the production of Cl_2. Hypochlorite is oxidised to ClO_2^- , and then in the presence of Cl_2, ClO_2 is formed, which is released from the anolyte and dissolved in NaOH solution in the absorber, or in the anolyte to form sodium chlorate and chlorite.

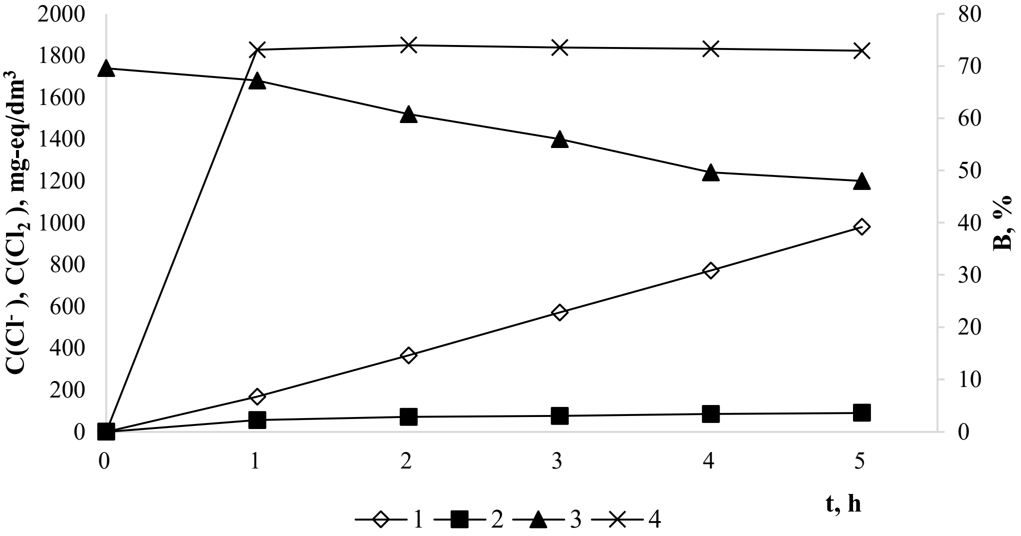


Fig. 4 – Dependence of the concentration of active chlorine in the anolyte (1) and absorber (2), the concentration of chlorides (3) and the current output of active chlorine (4) on the electrolysis time of sodium chloride solution (C(NaCl) = 120 g/dm^3) in a two-chamber electrolyzer (MA-41 membrane) at a current density of j = 8.33 A/dm^2, (catholyte 1H NaOH), (pH = 7.0–7.5)

Thus, oxidised chlorine compounds with oxidation degrees of Cl^{3+} and Cl^{5+} accumulate mainly in the anolyte and partially in the absorber.

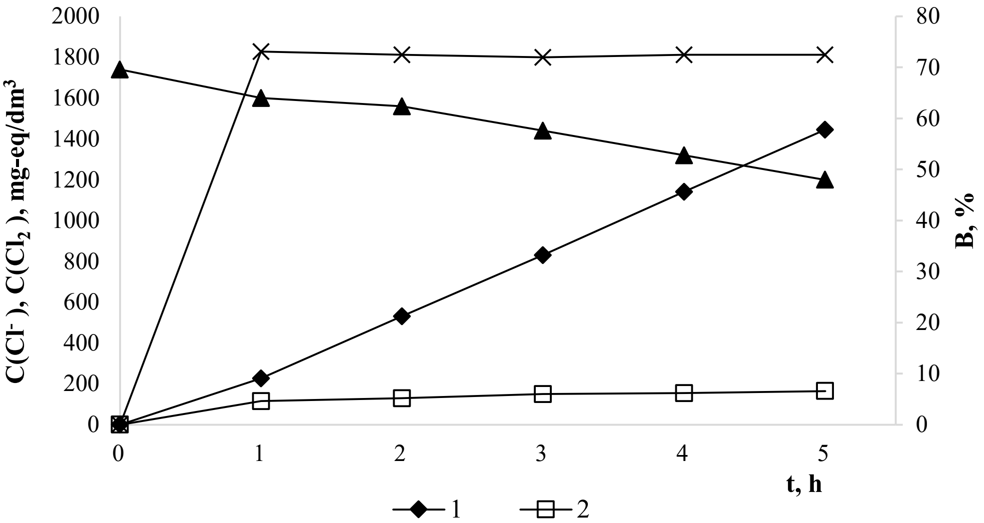


Fig. 5 – Dependence of the concentration of active chlorine in the anolyte (1) and absorber (2), the concentration of chlorides (3) and the current output of active chlorine (4) on the electrolysis time of sodium chloride solution (C(NaCl) = 120 g/dm^3) in a two-chamber electrolyzer (MA-41 membrane) at a current density j = 12.5 A/dm^2, (catholyte 1H NaOH), (pH = 6.5–7.0)

With a decrease in the concentration of sodium chloride in the anolyte to 30-45 g/dm^3, the yield of active chlorine decreased at a current density (j) of 4.167 A/dm^2 to 56-65 %, at a current density of 8.33 A/dm^2 to 43-46 %, and at a current density of 12.5 A/dm^2, the yield of active chlorine by current decreased to 32-40 % (Figs. 6-8).

At a current density of 4.167 A/dm^2 (Fig. 6), the concentration of active chlorine in the anolyte reached 350 mg-eq/dm^3 after 5 hours of electrolysis, and in the absorber – 75 mg-eq/dm^3. The total number of active chlorine obtained reached 53 mg-eq/dm^3. At the same time, the decrease in chloride concentration was only 19.5 mg-eq/dm^3. It is obvious that in this case, along with hypochlorite, sodium chlorite and chlorate were obtained.

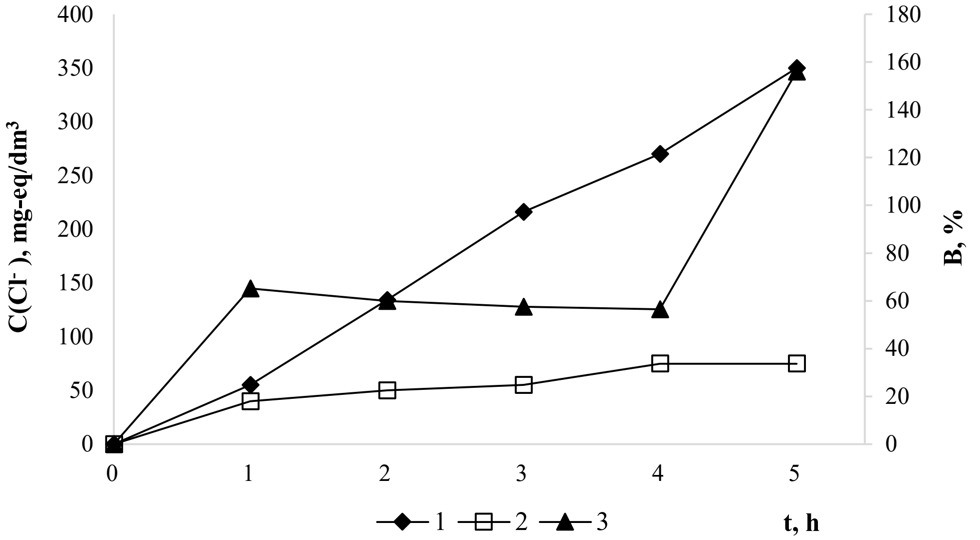


Fig. 6 – Dependence of the concentration of active chlorine in the anolyte (1) and in the absorber (2), and active chlorine yield by current (3) on the time of electrolysis of NaCl solution (C(NaCl) = 780 mg-eq/dm^3) in a two-chamber electrolyzer (MA-41 membrane) at a current density j = 4.167 A/dm^2, (catholyte 1H NaOH), (residual Cl$^-$ concentration= 630 mg-eq/dm)^3

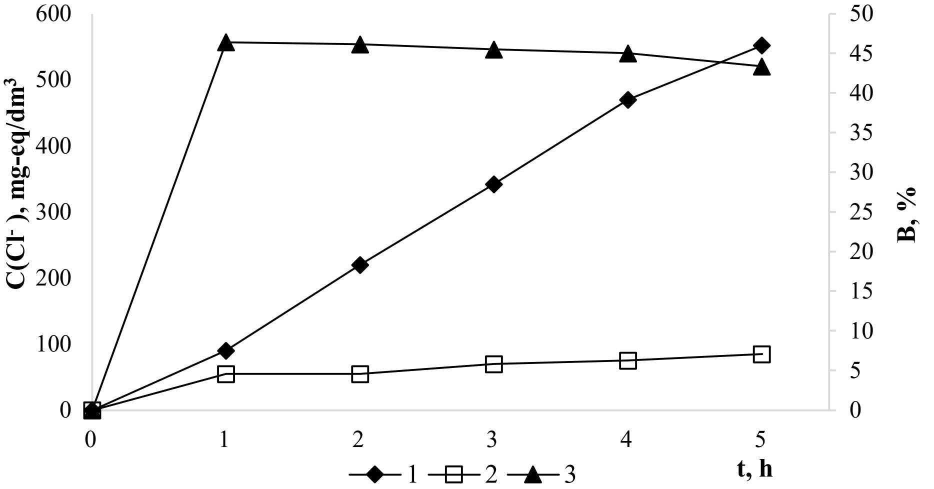


Fig. 7 – Dependence of the concentration of active chlorine in the anolyte (1), in the absorber (2), the output of active chlorine by current (3) on the electrolysis time of NaCl solution (C(NaCl) = 36.9 g/dm^3) in a two-chamber electrolyzer (MA-41 membrane) at a current density of j = 8.33 A/dm^2, (residual Cl$^-$ concentration = 320 mg-eq/dm^3), pH = 7.0–7.2

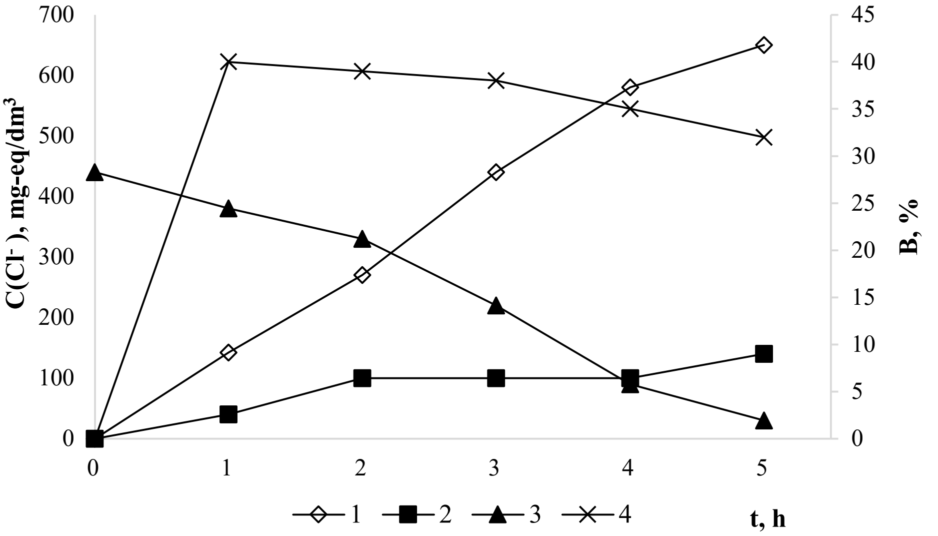


Fig. 8 – Dependence of the concentration of active chlorine in the anolyte (1) and absorber (2), chlorides (3) and active chlorine yield by current (4) on the time of electrolysis of NaCl solution (C(NaCl) = 30 g/dm^3) in a two-chamber electrolyzer (MA-41 membrane) at a current density j = 12.5 A/dm^2, (residual Cl$^-$ concentration = 30 mg-eq/dm^3), pH = 7.0–7.2

At a current density of 8.33 A/dm^2 (Fig. 7), the concentration of active chlorine in the anolyte reached 552 mg/dm^3, and in the absorber – 85 mg-eq/dm^3. In just 5 hours, 72 mg-eq were formed in the anolyte and 8.5 mg-eq in the absorber, a total of 80 mg-eq of active chlorine. At the same time, the chloride content decreased by 40 mg-eq, which confirms the possibility of chlorite and chlorate formation. The yield of active chlorine by current was in the range of 43-46 %.

With an increase in current density to 12.5 A/dm^2 at a NaCl concentration of 30 g/dm^3, the concentration of active chlorine in the anolyte reached 650 mg-eq/dm^3, and in the absorber – 140 mg-eq/dm^3. In just 5 hours, 98.5 mg-eq of active chlorine was obtained, with a decrease in chloride concentration by 53.3 mg-eq.

Obviously, in this case, chlorites and chlorates were formed and a significant number of current was consumed for the electrolysis of water. The output of active chlorine by current reached only 32-40 %.

Conclusions

1. As a result of experimental studies using the UPM diffusion membrane, it was determined that active chlorine accumulates in the electrolyte and the absorber. It was found that the current output of active chlorine generally reached ~ 70 %. At the same time, the total number of chloride decreased. The number of synthesised active chlorine reached 110 mg-eq/dm^3. Obviously, a significant number of active chlorine is released with hydrogen from the catholyte, which leads to significant losses. When using a higher concentration of sodium chloride solution, the current output of active chlorine reached 73-74 %.

2. During the electrolysis of NaCl solution, a significant number of active chlorine was removed from the anolyte and sorbed in the absorber. The bulk of the active chlorine was accumulated in the anolyte. No significant accumulation of alkali in the anolyte occurred due to the oxidation of hydroxide anions at the anode. The current yield of active chlorine, as in the previous case, reached ~ 73 %.

Prospects for further research

In the course of the experimental study, it can be said that when using two-chamber electrolyzers with diffusion and anionic membranes, the bulk of oxidised chlorine compounds was concentrated in the anolyte. Therefore, the use of a two-chamber electrolyzer with a cationic membrane is more promising.

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ОТРИМАННЯ АКТИВНОГО ХЛОРУ ТА ГІПОХЛОРИТУ НАТРІЮ В ЗАКРИТОМУ ЕЛЕКТРОЛІЗЕРІ

Сьогодні умовах ведення війни постійно відбувається локальне забруднення довкілля внаслідок використання вибухових речовин, пожеж, розливу паливно-мастильних матеріалів тощо. Щодня, небезпека збільшується при пошкодженні систем водовідведення різноманітних підприємств, таких як видобувні підприємства, об'єкти енергетики, металургії, хімічної, нафтохімічної промисловості, шламосховищ високотоксичних відходів. Поруч із цим існує і масштабується проблема високоефективного очищення води, насамперед питної. Внаслідок знищення Каховського водосховища у 2023 році різко загострилась проблема утилізації шахтних вод, які характеризуються високим рівнем мінералізації, значним вмістом заліза, марганцю та інших токсикантів. В даному випадку немає водних ресурсів для розведення шахтних вод перед їх скидом. Незважаючи на значну кількість розробок в галузі контролю якості води, технологій водоочищення, ряд проблем в даному напрямку залишаються невирішеними. Існуючі технології неефективні при перевищенні рівнів мінералізації та жорсткості води. Мова йде про південні області нашої країни та Донбас. У Миколаєві після зниження агресором водозабору на Дніпрі, в централізованій системі водопостачання подають солонувату воду з лиману. Відомо, що концентровані сольові розчини, котрі утворюються і все таки присутні поруч із проблемами, описаними вище, практично не переробляються. Їх скидають у більшості випадків у поверхневі водойми навколишнього природнього середовища, що суттєво погіршує стан водних екосистем України. Ситуація погіршується тим, що значна частина шахтних вод придатних для використання, через проблему утилізації концентратів не використовується, а скидається у довкілля після розведення, або, навіть, без розведення та очищення, так як ресурсу на розведення просто немає в реаліях сьогодення. Саме тому, проблема переробки концентратів баромембранного очищення з отриманням вторинних корисних продуктів була метою досліджень даної роботи. Дефіцит ресурсів прісних вод в Україні останнім часом компенсується використанням артезіанських вод, поверхневих вод та шахтних вод із підвищеною мінералізацією. Використання таких вод в комунальних господарствах, промисловості можливе лише після її опріснення. Головним методом опріснення таких вод є зворотній осмос та нанофільтрування. Застосування баромембранних методів в значній мірі обмежується складністю переробки та утилізації концентратів, що утворюються при очищенні води. Враховуючи великі об'єми відходів розчинів хлориду натрію в Україні та дефіцит реагентів для знезараження води, дезінфекції інших середовищ, відбілювання целюлози, паперу, тканини, доцільним є використання даних відходів для отримання активного хлору, гіпохлориту натрію тощо. Здавалось би, це не складно. Адже, технології отримання активного хлору, інших окислених сполук хлору досконало відпрацьовано. Однак, при виробництві окислених сполук хлору використовуються насичені, чисті розчини хлориду натрію з концентрацією > 100 г/дм^3. Більше того, при отриманні окислених сполук хлору не стоїть задача повного вилучення хлоридів з води. Як правило процес ведуть з метою досягнення максимального виходу за струмом отриманого активного хлору або гіпохлориту *натрію з частковим вилученням хлоридів з води, підтримуючи в електролізері концентрацію хлориду натрію на заданому рівні за рахунок розчинення сухого реагенту у реакційній суміші. При переробці відпрацьованих розчинів процесів знесолення води, насамперед, стоїть задача максимально повного вилучення хлориду натрію з води з отриманням корисних речовин і тому дотримуватись умов класичних процесів отримання сполук окисленого хлору просто неможливо. Крім того, при зниженні вмісту хлоридів у воді, вихід за струмом окислених сполук хлору суттєво знижується. Тому, нами були вивчені процеси окислення хлоридів у двокамерному електролізері з використанням дифузійної, аніонної (МА-41) та катіонної (МК-40) мембран. Для запобігання значним втратам хлору у вигляді* Cl_2 *та* ClO_2 *при дегазації використовували герметичний електролізер. З анодної камери відводили окислені сполуки хлору, з катодної камери відводили водень. При використанні катіонітової мембрани паралельно із активним хлором в анодній камері отримували розчин лугу в катіонній камері.*

Ключові слова: *знесолення води, переробка відходів водоочищення, активний хлор, гіпохлорит натрію, електроліз, католіт, аноліт, ультрафільтраційна мембрана, аніонообмінна мембрана, щільність струму, електролізер, абсорбер*

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