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NEW APPROACHES TO COMPREHENSIVE ELECTROCHEMICAL PROCESSING OF SULFATE-CHLORIDE HIGH-MINERALIZED WASTEWATER TREATMENT RESIDUES

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Abstract

The results of electrochemical processing of spent acidic, neutral, and alkaline sulfate-chloride-containing regenerative solutions in two- and three-chamber electrolyzers are presented. It has been determined that the highest current efficiency for the products of electrodialysis can be achieved in the presence of hardness ions when processing acidic sulfate-containing solutions using three-chamber electrolyzers. It has been established that during electrodialysis of alkaline solutions after regeneration of anionites containing chloride ions, accumulation of alkali occurs in the cathodic region, and in the anodic region, chloride ions accumulate due to diffusion through the anion exchange membrane during the first stage, followed by preferential oxidation of chloride ions with liberation of free chlorine during the second stage. It has been shown that electrodialysis can effectively solve the problem of comprehensive processing of neutral, alkaline, and acidic regenerative sulfate-chloride-containing solutions, with the production of alkali and acid at concentrations suitable for reuse in regeneration processes.

Keywords: **Electrochemical processing; Electrodialysis; Spent regenerant solutions; Sulfate-chloride solutions; Three-chamber electrolyzers; Anion membrane; Cation membrane.**

1. INTRODUCTION

The water ecosystems of Ukraine, despite a decrease in production due to the crisis, are constantly experiencing growing anthropogenic pressure. The ecological well-being of water ecosystems is largely determined by sustainable water management aimed at creating and developing closed-loop water consumption systems, preventing the discharge of pollutants into water bodies, and creating resource-saving technologies. The use of natural water without prior conditioning in cooling systems leads to premature failure of heat exchangers, condensers, and pipelines due to scaling and sediment deposition. At the same time, a significant amount of water is discharged into water bodies during system blowdown, leading to their thermal pollution, and a sharp increase in natural water intake. Rational water use in industry and energy largely depends on the efficiency of water treatment. However, the processing of highly mineralized wastewater remains an important and challenging issue to be addressed.

2. ANALYSIS OF PREVIOUS STUDIES

To address the challenges of water softening and desalination in the industrial and energy sectors, membrane-based methods are widely proposed [1, 2]. However, without prior chemical or ion exchange treatment, these membranes quickly lose their permeability due to salt deposition on their surface. Additionally, pretreating the water with aluminium hydroxychloride [3, 4] or sodium hydroxyaluminate [5] results in the formation of precipitates that require disposal. While membrane regeneration is an effective solution, it is often prohibitively expensive, leading to the decision to replace the membranes entirely and resulting in a significant increase in the cost of water treatment. In the case of reverse osmosis and nanofiltration, high concentrations of sulfate and sodium chloride are present in the concentrates, requiring additional processing solutions [6].

Ion exchange is one of the most accessible, widespread, and effective methods for water softening and desalination, as it allows for maintaining the salt concentration in water at a desired level [7]. The use of softened water enables closed cooling systems, reducing the intake of natural water and avoiding water discharge during water recycling. However, the main problem that arises during the implementation of the method is the difficulty in processing and disposing of solutions formed during the regeneration of ion-exchange resins. These solutions contain excess reagents used during regeneration (acids, alkalis, sodium sulfate, and chloride), which necessitates their chemical treatment before discharge. One way to process concentrates is through the precipitation of gypsum and ettringite, but in this case, the question of purifying filtrates by membrane methods arises again [8].

To extract monovalent cations and anions (chlorides and sulfates) from regeneration solutions, electrochemical methods are the most promising due to the absence of additional chemical reagents [9]. Electrodialysis is used for processing spent regeneration solutions in two-chamber electrolyzers to obtain acids [10] and alkalis [11] with a current density of 10–30 A/dm^2 from sulfate-containing salt solutions. However, acidic salt solutions are formed during the regeneration of cation-exchange resins, while basic salt solutions are formed during the regeneration of anion-exchange resins. These solutions may contain not only sodium cations but also calcium and magnesium cations or chloride anions along with sulfate anions, which significantly complicates the electrodialysis process. Furthermore, the high current density required for electrodialysis requires more electrical energy, thereby increasing the cost of producing acids and alkalis.

Therefore, there is interest in studying the processing of sulfuric acid regeneration solutions that contain calcium and magnesium ions. It would also be interesting to evaluate the efficiency of electrolysis of acidic and alkaline regeneration solutions containing chlorides, compared to neutral solutions. Another question that will be addressed in the research is the comparison of the efficiency of electrolysis in two- and three-chamber electrolyzers at low current densities to increase the energy efficiency of the process.

3. MATERIALS AND METHODS

For electrolysis, two-chamber and three-chamber electrolyzers were used. Each chamber had a volume of 100 cm^3. A stainless steel plate of 12X18H10T was used as the cathode, and a titanium plate coated with ruthenium oxide was used as the anode. The electrode area was $S_K = S_A = 0.12$ dm^2. The electrolysis was carried out at a current density of 3–7 A/dm^2. The reagents for model solutions were provided by LLC “KhimLaborReaktiv” (Ukraine, Brovary city). Model solutions similar in composition to regenerative solutions were used as working solutions.

For investigating electrodialysis in acidic solutions, model solutions and mixtures were used, including

sodium sulfate solution with a concentration of 32 g/dm^3; sodium sulfate and sulfuric acid with concentrations of 324 mg-eq/dm^3 and 590–625 mg-eq/dm^3, respectively; sulfuric acid, magnesium sulfate, and sodium sulfate with concentrations of 408 mg-eq/dm^3, 667 mg-eq/dm3, and 363 mg-eq/dm^3, respectively.

To investigate electrodialysis in alkaline solutions, model solutions and mixtures of sodium sulfate and caustic soda were used with concentrations of 450 and 53 mg-eq/dm^3, respectively; sodium sulfate, sodium chloride, and caustic soda with concentrations of 250, 517, and 86 mg-eq/dm^3, respectively; sodium sulfate and sodium chloride with concentrations of 250–300 mg-eq/dm^3.

To study electrodialysis in alkaline solutions, model solutions and mixtures of sodium sulfate and alkali were used with concentrations of 450 and 53 mg-eq/dm^3, respectively; sodium sulfate, sodium chloride, and alkali were used with concentrations of 250, 517, and 86 mg-eq/dm^3, respectively; and sodium sulfate and sodium chloride were used with concentrations of 250–300 mg-eq/dm^3.

To ensure electrical conductivity in the cathodic region, a solution of alkali with a concentration of 9 mg-eq/dm^3 was used, and in the anodic region, a solution of sulfuric acid with a concentration of 1 mg-eq/dm^3 was used.

Cation exchange membrane MC-40 and anion exchange membrane MA-41, produced by JSC "Shchokinoazot" (Russia, Shchokino), were used as heterogeneous ion-exchange membranes. These membranes were obtained by pressing a mixture of ionite powder and polyethylene, using synthetic ion-exchange resins KU-2 and AV-17, respectively [12]. To ensure mechanical strength, the surface of the membranes was reinforced with nylon mesh. The mentioned heterogeneous ion-exchange membranes exhibit chemical resistance and can be used for an extended period, even in concentrated solutions of acids and alkalis [13].

When conducting research in a two-chamber electrolyzer for acidic solutions, the MC-40 cationic membrane was used, with the working solution placed in the anodic region and the alkali solution placed in the cathodic region. When using the MA-41 anionic membrane, the working solution was placed in the cathodic region and the sulfuric acid solution was placed in the anodic region. In the case of a three-chamber electrolyzer, the working solution was placed in the middle chamber, the alkali solution was placed in the cathodic chamber, and the sulfuric acid solution was placed in the anodic chamber.

In the case of alkaline solutions in a three-chamber electrolyzer, the working solution was placed in the middle chamber, and in a two-chamber electrolyzer, the working solution was placed in the cathodic zone, with the MA-41 anion exchange membrane used as the membrane.

During the electrolysis process, the acidity and alkalinity in the anodic and cathodic chambers, the alkalinity or acidity, and the sulfate content in the working chamber, as well as the sulfate and chloride anion content in the anodic region, were monitored hourly. The alkalinity and acidity were determined using the classical titrimetric method with 0.1 M HCl and 0.1 M NaOH, and indicators such as phenolphthalein and methyl orange were employed accordingly [14]. The sulfate anion content was determined using a photometric method [14]. The chloride anion content was determined using the Mohr method [15].

The degree of purification of solutions from sulfates was calculated using the following formula:

$$Z = \frac{(C_{in} - C_{res})}{C_{in}} \times 100\,\% \qquad (1)$$

where C_{in} – initial ion concentration;

C_{res} – residual ion concentration.

The current efficiency of electrochemical systems was calculated as the ratio of the actual amount of electricity spent on the transfer of equivalent substance (q_a) to the theoretical amount of electricity spent on the transfer of substance (q_T):

$$B = \frac{q_a}{q_T} \times 100\% = \frac{m_a}{m_T} \times 100\% \qquad (2)$$

where q_a – the amount of electricity actually spent on the transfer of equivalent substance;

q_T – the theoretical amount of electricity spent on the transfer of substance;

m_a – the actual amount of transferred substance;

m_T – the theoretically determined amount of transferred substance.

The actual amount of transferred substance is determined by the change in its concentration in the total volume of the solution.

The theoretical amount of transferred substance was determined according to Faraday's law:

$$m_T = Ke \times I \times t \quad (3)$$

(3)

where K_e – the electrochemical equivalent;

I – the current, A;

t – the time, hours.

The total duration of the electrolysis process in two- and three-chamber electrolyzers was regulated by the ability to obtain acid and alkali solutions of the required concentration suitable for the regeneration of ionites for reuse.

4. RESULTS AND DISCUSSION

The results of the electrolysis of an acidic solution, which models regenerative solutions formed after regeneration of cationic filters that adsorbed monovalent cations, are presented in Figure 1, Figure 2, and Figure 3. In this case, electrolysis was carried out in a solution containing sodium sulfate and sulfuric acid.

When using three-chamber electrolyzers (Figure 1 and Figure 2), a decrease in acidity and sodium sulfate content in the working zone, formation of an alkaline solution in the cathodic area, and an acidic solution in the anodic area were observed. The average electrochemical efficiency for acid production at a current density of 6.62 A/dm^2 reached 57.9% and at a current density of 3.97 A/dm^2 – 55.2%. This corresponds to known data [16]. The current efficiency for alkali production was 18.2% and 17.3% respectively. Obviously, such a significant decrease in alkali current efficiency compared to acid is related to the high acidity of the working solution. In this case, a considerable amount of current is used for hydrogen reduction at the cathode, and due to the high concentration of hydrogen, the transmembrane transport of sodium ions to the cathodic area is reduced. The acidity in the working area in this case decreases to 4–10 mg-eq/dm^3.

In the case of using a two-chamber electrolyzer with a cationic membrane MC-40, the working solution is placed in the anodic area. In this case, only sodium cations and protons migrate from the anodic to the cathodic area. The latter process is undesirable.

As evident from Figure 3, when replacing a three-chamber electrolyzer with a two-chamber one, the current efficiency of acid production decreases to 13%, and alkali production decreases to 13.4%. Overall, the advantage of this process is that only acid and alkali solutions are obtained as a result of electrolysis, whereas in the case of a three-chamber electrolyzer, there is still a working solution that requires a significant amount of electrical energy for complete desalination. However, a drawback of the process is a significant reduction in acid current efficiency from 55% to 13%. The current efficiency of alkali also decreases from 17% to 13% in this case. Overall, this indicates that the diffusion of sodium cations through the cationic membrane at an acidity level of around 500 mg-eq/dm3 does not change significantly with a further increase in acidity up to 1000 mg-eq/dm^3.

One of the options for increasing the alkali current efficiency during electrolysis of acidic regenerative

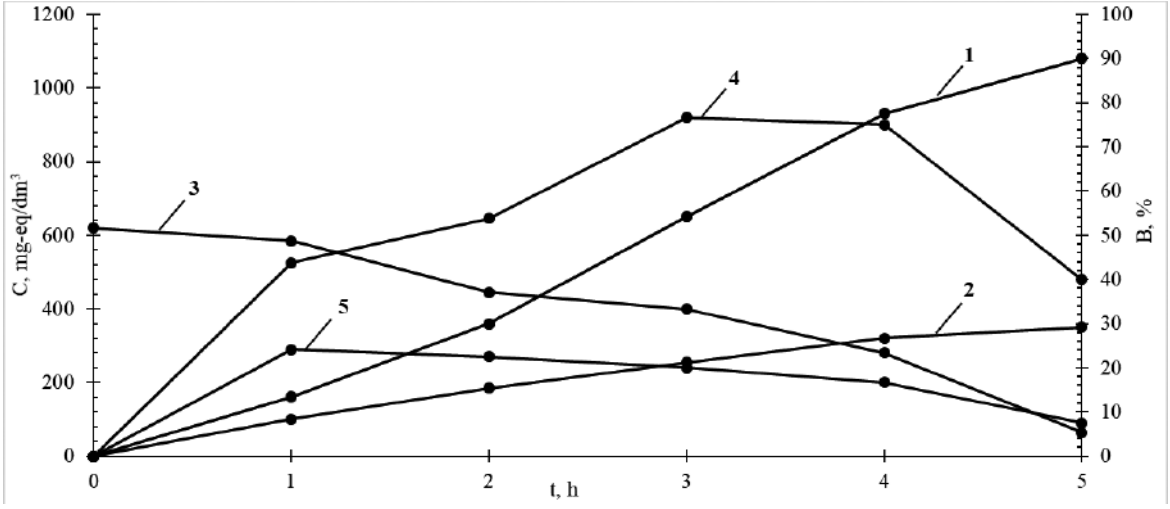


Figure 1.
Dependence of acidity, alkalinity, and current efficiency on electrolysis time of sodium sulfate (324 mg-eq/dm^3) and sulfuric acid (625 mg-eq/dm^3) solutions in a three-chamber electrolyzer (cation membrane MC-40; anion membrane MA-41) at a current density of 6.62 A/dm^2 (ACEmid = 57.9%; ALEmid = 18.2%)

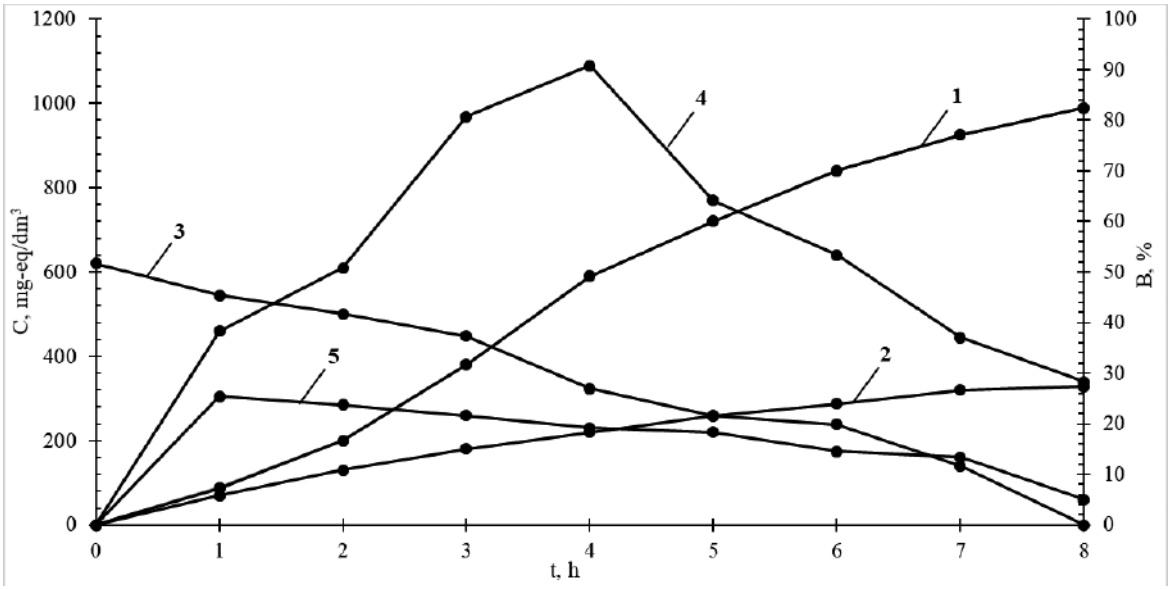


Figure 2.
Dependence of acidity, alkalinity, and current efficiency on time of electrolysis of sodium sulfate (324 mg-eq/dm^3) and sulfuric acid (590 mg-eq/dm^3) solution in a three-chamber electrolyzer (cationic membrane MC-40; anionic membrane MA-41) at a current density of 3.97 A/dm^2 (ACEmid=55.2%; ALEmid=17.3%)%)

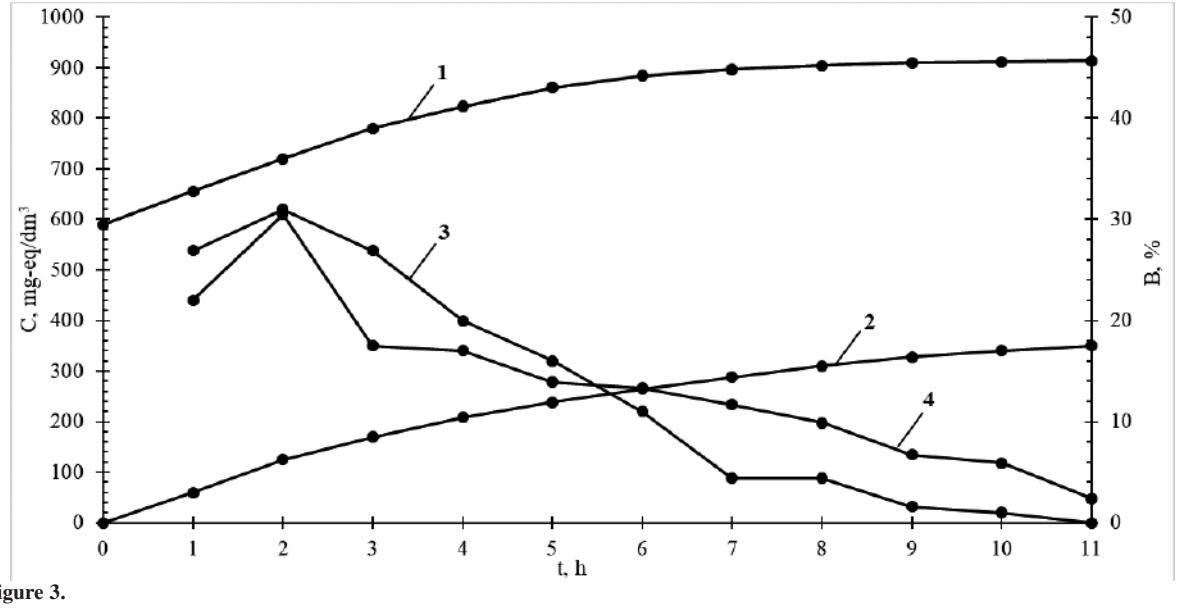


Figure 3.
Dependence of acidity, alkalinity, and current efficiency on time of electrolysis of sodium sulfate (324 mg-eq/dm^3) and sulfuric acid (590 mg-eq/dm^3) solutions in a two-chamber electrolyzer (cationic membrane MC-40) at a current density of 3.97 A/dm^2 (ACEmid = 13.0%; ALEmid = 13.4%)

solutions is their pre-neutralization with alkali or spent alkaline solutions. Therefore, sodium sulfate solution was used as a model in further investigations. The efficiency of electrolysis of neutral sodium sulfate solutions can be assessed in Table 1.

As observed from Table 1, when neutral solutions are used, the alkaline output increases significantly compared to acidic solutions, while the acid output decreases slightly, especially with an increase in acidity in the anodic region.

In two-chamber electrolyzers, a decrease in current output was noted when the working solution was placed in the cathodic zone (MC-40 membrane) for both acid and alkali, with a reduction of 26% and

Table 1.
Dependence of acidity (alkalinity) of anodic, cathodic, and working solutions on the time of electrolysis of sodium sulfate solution (C=32 g/dm^3), current density, and type of electrolyzer

Electrolysis time, t, hours	Number of chambers	I, A/dm^2	Membrane		Cathode zone			Anode zone			Working zone, concentration (C), mg-eq/dm^3	
			Cationic	Anionic	Acidity, mg-eq/dm^3		B, %	Alkalinity, mg-eq/dm^3		B, %		
					I	II		I	II		I	II
3.5	3	3.97	MC-40	MA-41	1	437	55.6	1	426	54.2	-	4
4.0	3	3.97			437	828	43.6	426	715	32.4	4	0
3.5	3	3.97			828	1096	34.2	715	987	34.7	0	2
4.5	3	3.97			1096	1448	34.9	987	1301	31.2	2	0
4.0	3	3.97			1448	1751	33.8	1301	1571	30.1	0	6
5.0*	3	6.62			9	1012	53.6	1	824	44.1	0	586
6.0	2	3.97	-		9	346	25.7	1	468	34.7	-	-
7.0	2	3.97	MC-40	-	9	400	24.9	1	446	28.4	-	-

I - Before electrolysis;
II - After electrolysis;
* Concentration of Na_2SO_4 – 150 g/dm^3

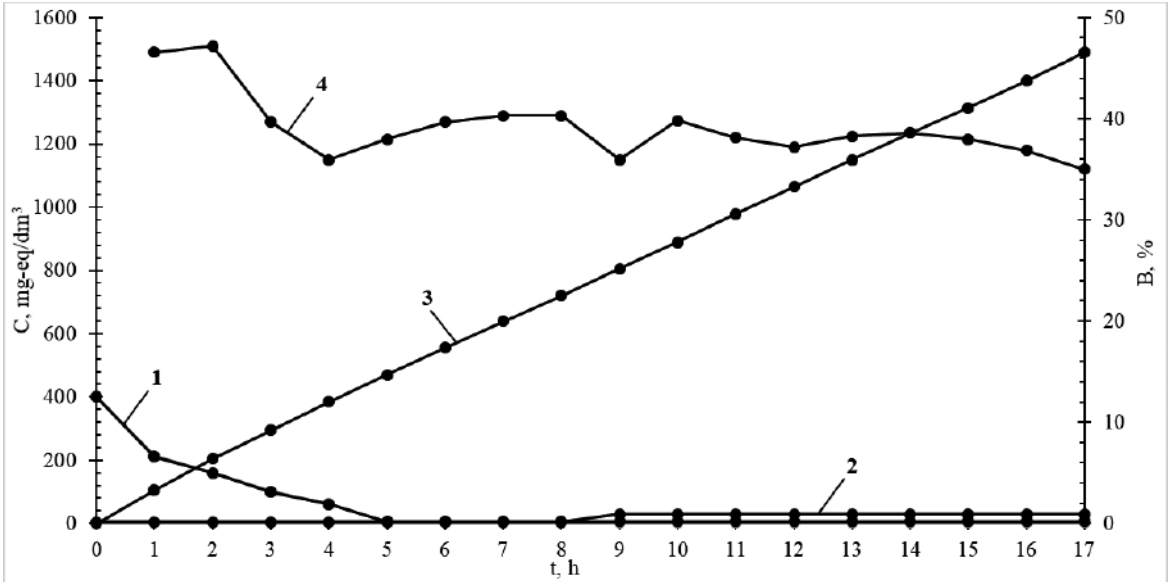


Figure 4.
Dependence of acidity, alkalinity, and acid current efficiency on the electrolysis time of sodium sulfate (408 mg-eq/dm^3), magnesium sulfate (667 mg-eq/dm^3), and sodium sulfate (363 mg-eq/dm^3) solutions in a two-compartment electrolyzer (MA-41 membrane) at a current density of 3.97 A/dm^2 (ACEmid = 39.0%)

30% respectively. When the working solution was placed in the anodic region (MA-41 membrane), the current output decreased by 20% for acid and 29% for alkali.

During the electrolysis of concentrated sodium sulfate solution in a three-chamber electrolyzer for 3 hours, the concentration of alkali in the cathodic region reached 1012 mg-eq/dm^3, while the concentration of acid was only 824 mg-eq/dm^3. At the same time, the acidity in the working zone reached 586 mg-eq/dm^3. This indicates that hydroxyl ions diffuse better through the MA-41 membrane in the presence of sulfate ions, compared to protons through the cationic MC-40 membrane in the presence of sodium ions. This is supported by the fact that regardless of the pH of the working solution, over time, acidification of the solution always occurs in the working zone of the three-chamber electrolyzer.

From Table 1, it can be seen that with repeated use of acidic solutions in the anodic zone and alkaline solutions in the cathodic zone, after several consecutive processes, the acidity in the anodic region can reach 1500 mg-eq/dm^3 (concentration of acid 7.5%) and the alkalinity in the cathodic region can reach

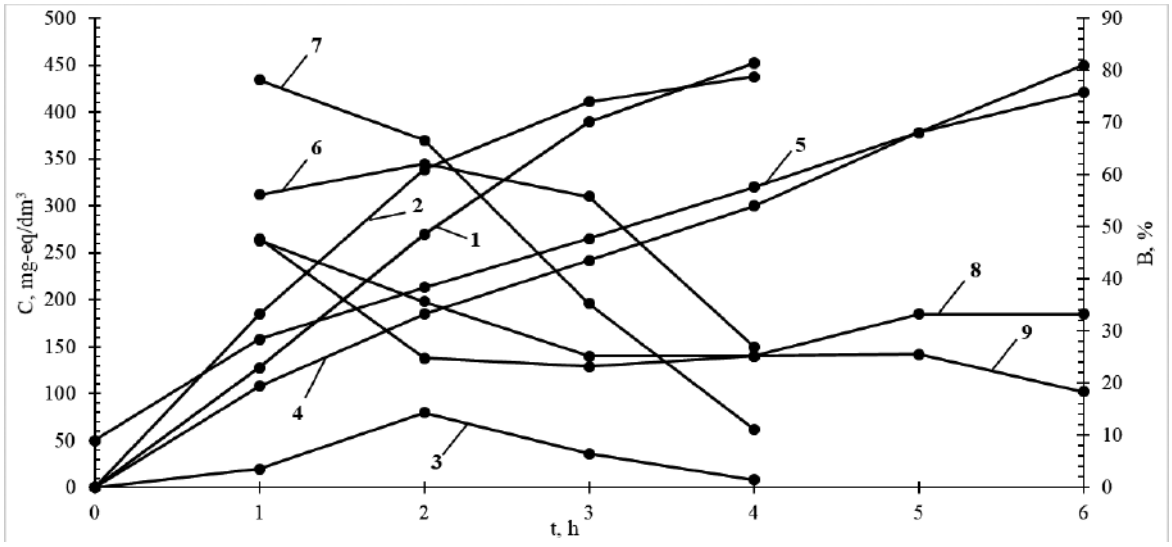


Figure 5.
Dependence of acidity, alkalinity, and acid current efficiency during electrolysis in a three-chamber electrolyzer (cationic membrane MC-40, anionic membrane MA-41) and a two-chamber electrolyzer (anionic membrane MA-41) at a current density of 3.97 A/dm³ of sodium sulfate solution (450 mg-eq/dm³) and alkali (53 mg-eq/dm³) (ACEmid(6)=50.2%; ALEmid(7)=47.81%; ACEmid(8)=33.4%; ALEmid(9)=27.3%)

1750 g-eq/dm³ (concentration of alkali 7.0%). This means that electrolysis can produce acid and alkali solutions suitable for reuse in the regeneration of ion-exchange resins.

The presence of hardness ions poses a significant problem in the electrolysis of regenerant solutions. In the presence of hardness ions in sulfuric acid regenerant solutions, the electrolysis process in three-chamber electrolyzers slows down significantly due to blockage of the cation exchange membrane by these ions and the formation of deposits during their hydrolysis. Therefore, electrolysis of acidic solutions containing magnesium ions (Figure 4) is complicated and requires additional measures to mitigate the negative effects of hardness ions.

In the anodic region, the acidity reaches a level of 1484 mg-eq/dm³ (acid output per current efficiency of 39%). A significant increase in alkalinity in the cathodic region cannot be achieved due to the fact that magnesium ions precipitate as magnesium hydroxide after hydrolysis. Therefore, from solutions containing hardness ions, acid can be separated along with the precipitation of magnesium hydroxide and suspension of calcium hydroxide.

As a result of the conducted research, it has been shown that during the electrolysis of acidic solutions in three-chamber electrolyzers, the output of alkali per current efficiency decreases due to the excess of protons in the working solution and their competition with sodium ions during transmembrane transport. During the electrolysis of neutral solutions, due to the better diffusion of hydroxide ions through the anion membrane compared to the diffusion of protons through the cation membrane, the working solution becomes more acidic and the acid output per current efficiency decreases. Therefore, neutralization of regenerating solutions before electrolysis is not advisable. Overall, during the electrolysis of acidic and neutral solutions, acid up to 7.5% and alkali up to 7% were obtained in the anodic and cathodic regions, respectively. The efficiency of the processes decreases when transitioning from three-chamber to two-chamber electrolyzes because in the anodic region, protons compete with cations during transmembrane transport through the cation membrane. In the cathodic region, hydroxide ions compete with sulfate anions during diffusion through the anion membrane.

The use of two-chamber electrolyzes is advisable for the electrolysis of regenerating solutions containing magnesium ions. In this case, due to the binding of hydroxyl anions with magnesium ions in the cathodic zone during the precipitation of magnesium hydroxide, their negative influence on the transmembrane transport of sulfate ions decreases, allowing the production of sulfuric acid with an acid output per current efficiency of 39% and an acidity level of 1500 mg-eq/dm³.

The results obtained from the electrolysis of alkaline regenerative solutions are shown in Figure 5.

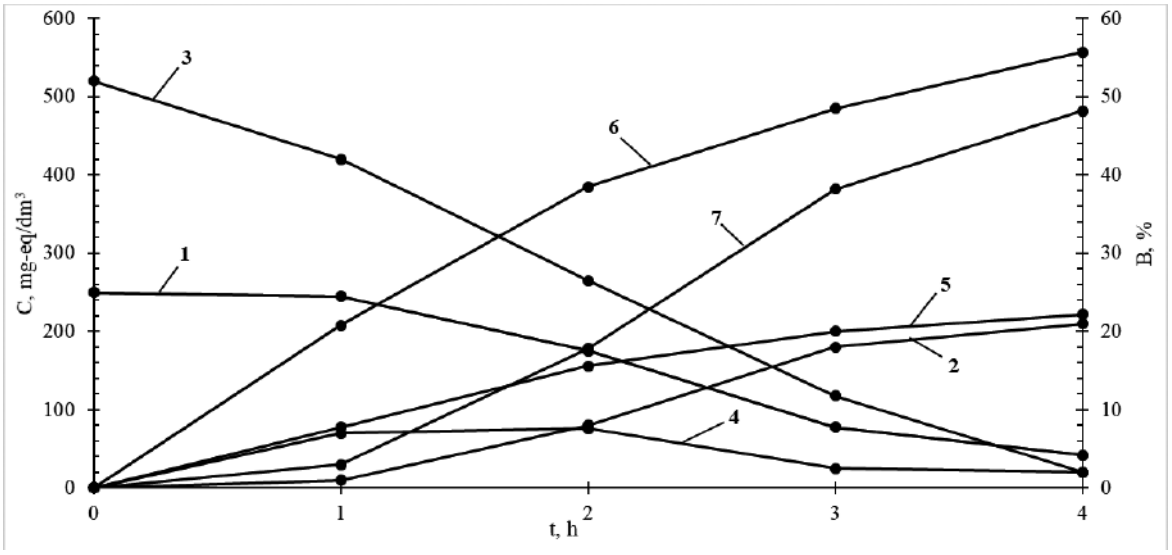


Figure 6.
Dependence of sulfate concentration, chloride concentration, acidity, alkalinity, and free chlorine content (m_{Cl2}) on time of electrolysis of sodium sulfate solution (C=250 mg-eq/dm^3), sodium chloride solution (C=517 mg-eq/dm^3), and sodium hydroxide solution (C=86 mg-eq/dm^3) in a three-chamber electrolyzer (cationic membrane MC-40, anionic membrane MA-41) at a current density of 3.97 A/dm^2 (Bmid^{S042-}=22.6%; Bmid^{Cl-}c=1.6%; Bmid^{Cl2}c=52.4%; ACEmid=24.1%; ALEmid=59.2%)

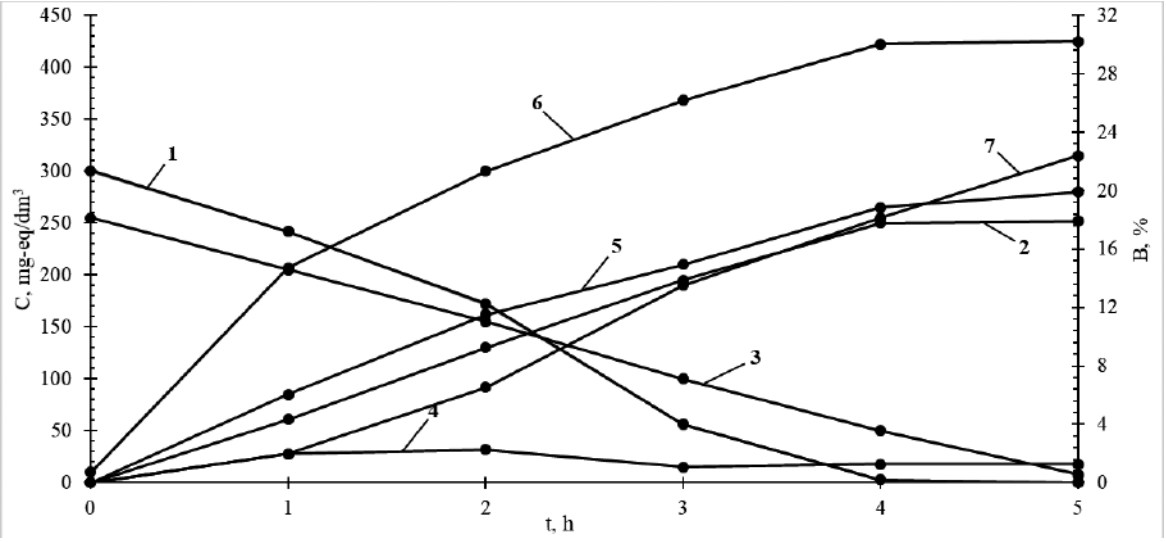


Figure 7.
Dependency of sulfate concentration, chloride concentration, acidity, alkalinity, and amount of free chlorine (mCl2) on electrolysis time of sodium sulfate solution (C=300 mg-eq/dm^3) and sodium chloride solution (C=250 mg-eq/dm^3) in a three-chamber electrolyzer (cationic membrane MC-40, anionic membrane MA-41) at a current density of 3.97 A/dm^2 (Bmid^{S042-}=22.3%; Bmid^{Cl-}c=1.5%; Bmid^{Cl2}c = 20.0%; ACEmid=25.0%; ALEmid=37.9%)

The concentration of alkali in the working solution was approximately 0.2% (53 mg-eq/dm^3). This is because an alkali solution with a concentration of up to 4% is commonly used for regenerating low-basicity anion-exchange resins, which are used for removing sulfates and chlorides. Low-basicity anion-exchange resins are effectively regenerated, hence the excess of alkali in the solutions is minimal. Overall, the decrease in alkali concentration in the working solution resulted in the efficient removal of sulfates from the working solution with the formation of sulfuric acid in the anodic region. In this case, the acid current efficiency in the three-chamber electrolyzer reached 50.2%, while in the two-chamber electrolyzer it was 33.4%. The alkali current efficiency was 47.8% and 27% respectively. In this case, the

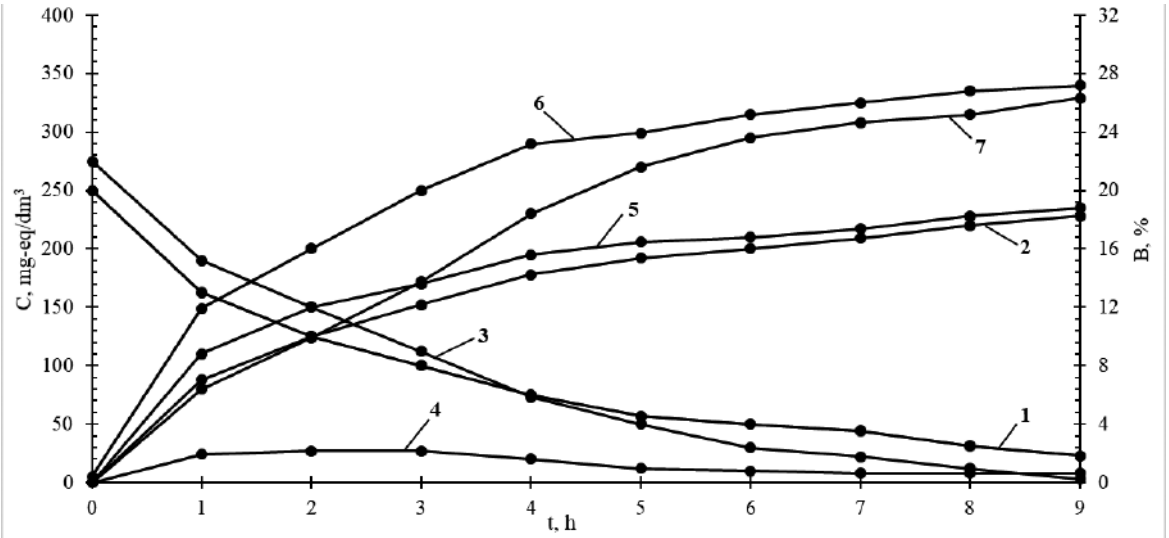


Figure 8.
Dependency of sulfate concentration, chloride concentration, acidity, alkalinity, and amount of free chlorine (m_{Cl2}) on electrolysis time of sodium chloride solution (276 mg-eq/dm^3) and sodium sulfate solution (250 mg-eq/dm^3) in a two-chamber electrolyzer (anionic membrane MA-41) at a current density of 3.97 A/dm^2 ($Bmid^{S042-}$=11.3%; $Bmid^{Cl-}$c=0.3%; $Bmid^{Cl2}$c =13.0%; ACEmid=11.7%; ALEmid=16.9%)

transition from a three-chamber to a two-chamber electrolyzer resulted in a decrease in acid and alkali current efficiency by 17% and 20% respectively, and the use of a two-chamber electrolyzer may be justified in certain cases.

It is known that besides carbonates, sulfates and chlorides are the main anions present in natural water. Therefore, during the regeneration of low-basicity anion-exchange resins, chloride ions are also present in the regenerant solution along with sulfates. The results of electrolysis of chloride-containing solutions are presented in Figure 6, Figure 7, and Figure 8.

In a two-chamber electrolyzer, the electrolysis process is slowed down due to diffusion through the anion exchange membrane of hydroxyl anions, in addition to chloride and sulfate ions, which are formed in the cathodic region. Overall, this not only leads to a decrease in the current efficiency of acid and alkali production, but also reduces the yield of free chlorine to only 13%, as opposed to three-chamber electrolyzers where it can reach 20–50%.

As seen from the results presented, the electrodialysis method allows for effective processing of spent regenerant solutions generated during the regeneration of anion exchange filters to produce sulfuric acid and alkali. In case the regenerant solution contains chloride ions alongside sulfate ions, the formation of free chlorine occurs. Therefore, the anodic chamber of the electrolyzer should be tightly sealed with the generated chlorine being vented to a reservoir containing lime milk for the production of calcium hypochlorite during the capture of free chlorine. This will ensure personnel protection from chlorine poisoning and efficient utilization of the secondary product. The anodic chamber and its structural elements should be made of corrosion-resistant materials. The room where the electrolyzer is located should be equipped with effective ventilation.

5. CONCLUSIONS

Based on the results obtained, it can be concluded that electrodialysis can effectively address the problem of processing neutral and acidic regenerative sulfate solutions containing monovalent cations and hardness ions, resulting in the production of alkali and acid solutions at concentrations suitable for reuse.

Regarding the investigation of electrolysis processes in model solutions that are similar in composition to solutions formed during the regeneration of anion exchange filters, it has been shown that during the electrolysis of sulfate-containing solutions, both neutral and with an excess of alkali, the electrolysis process proceeds with the formation of sulfuric acid and alkali at concentrations sufficient for their reuse in regeneration processes. It has been established that during the electrolysis of solutions containing chloride anions, accumulation of alkali occurs in the cathodic region, while in the anodic region, during

the first stage, accumulation of chloride ions occurs due to diffusion of these ions through the anionic membrane, and during the second stage, predominant oxidation of chloride ions with release of free chlorine takes place.

One advantage of these research studies compared to previous works, particularly [10, 11], is the utilization of significantly lower current density ranging from 3.97-6.62 A/dm^2, which allows for an increase in the energy efficiency of the process.

Depending on the purpose of the process and the desired outcomes, the choice of utilizing either a two- or three-chamber electrolyzer is possible under production conditions. In this case, a slight increase in energy consumption to enhance current density and electrolysis time for conducting the process is not decisive.

Industrial testing of the electrodialysis process is planned to be carried out in domestic thermal power plants and combined heat and power plants. Furthermore, the application of this method is also reasonable in other enterprises that employ water desalination technology.

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