Engineering Journal of Satbayev University
Volume 147 (2025), Issue 6, 1-7

https://doi.org/10.51301/ejsu.2025.i6.01

Investigation of electrodialysis membrane processes for sodium sulfate solutions with alkaline and acid regeneration

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Abstract. This article considers the possibility of processing sodium sulfate (Na_2SO_4) solutions generated in various industrial processes into valuable products - sulfuric acid (H_2SO_4) and sodium hydroxide (NaOH) using the electrodialysis method. The problem of processing sodium sulphate solutions is relevant due to their significant volume and high content of inorganic impurities, while traditional methods of purification are energy-intensive and economically inexpedient. The authors have analysed the existing methods of obtaining and processing sodium sulphate solutions by electrodialysis using MC-40, MA-41 and MB-2I membranes and Ralex BM membranes. In purpose of electrodialysis the schemes of 3-chamber and 6-section electrodialyzers using cation- and anion-exchange membranes of MC-40, MA-41 (RF) and EDC1R, EAC1R (PRC) grades were applied. The scheme of bench installation of a multichamber electrodialyzer using EDAM and EDCM membrane brands was considered. The conditions of experiments on 3 installations according to the method of probabilistic-deterministic planning of experiments by Malyshev V.P. The dependences for the first installation were obtained: the degree of conversion on the concentration of Na_2SO_4, the duration of the process on the cathodic density and the content of $MgSO_4$ impurity. Dependences of energy consumption on concentration of initial solution and current density at 3 installations allow to judge about expediency of electrodialysis process.

Keywords: *sodium sulfate, electrodialysis, membrane processes, sodium hydroxide, sulfuric acid*.

Received: 28 July 2025
Accepted: 15 December 2025
Available online: 31 December 2025

1. Introduction

In the industrial cycle of ore and concentrate processing, which involves the extensive use of reagents such as H_2SO_4 and NaOH, a low-concentration sodium sulfate solution is generated as a waste or by-product. The purification of such dilute solutions is considered economically unfeasible and energy-intensive. One of the methods for converting sodium sulfate into concentrated alkali and acid is through membrane-based electrodialysis processes [1-8] driven by an electric current.

In article [9], the process of electrodialysis of a Na_2SO_4 solution using MC-40, MA-41, and MB-2I membranes is studied, with the aim of producing concentrated solutions of alkali and acid. The sodium sulfate solution used is a by-product of the battery scrap recycling process. The authors conducted experiments using model solutions of Na_2SO_4 (0.5 mol/l), H_2SO_4 (0.005 mol/l), and NaOH (0.01 mol/l), and presented several cell configurations employing ion-exchange membranes. The best results were achieved using a three-compartment, non-flow-type electrodialyzer equipped with cation- and anion-exchange membranes. The resulting concentrations of the products were 0.34 mol/l for NaOH and 0.23 mol/l for H_2SO_4, with specific energy consumptions of 0.84 kWh/mol for NaOH and 1.10 kWh/mol for H_2SO_4. The main drawbacks of the process include low productivity and significant gas evolution in the cell compartments.

Publication [10] presents a process flow diagram for the treatment of $Na_2SO_4 \cdot 10H_2O$ precipitate via electrodialysis. This precipitate forms at temperatures of 8–10ºC. The technological scheme includes the following equipment: a three-compartment electrolyzer, with a PbO_2 anode and a stainless-steel cathode; circulation pumps; and solution storage tanks. The initial concentrations of the solutions were: sodium sulfate – 120 g/l, NaOH – 5 g/l, and H_2SO_4 – 7 g/l. Upon completion of the process, the resulting concentrations reached 150 g/l for NaOH and 182 g/l for H_2SO_4, under a current density of 2 A/dm^2 and a processing time of 5 hours. The system demonstrated a productivity of approximately 1 kg/(m^2·h) and a specific energy consumption of 7.5 kWh/kg. The authors note the following drawbacks of the process: significant gas evolution, the need for preliminary purification of the solutions from impurities, and a limitation on current density-exceeding 3 A/dm^2 is undesirable due to the potential deterioration of process parameters.

Research [11] describes the conversion of sodium sulfate into H_2SO_4 and NaOH using Ralex BM membranes. The investigation focused on the effects of current density and the concentrations of acid and alkali on membrane transport

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Engineering Journal of Satbayev University. eISSN 2959-2348. Published by Satbayev University

efficiency. The system's productivity increased proportionally with current density and decreased as Na_2SO_4 conversion progressed. The current density in the experiments ranged from 200 to 1000 A/m^2, while the concentrations of acid and alkali varied from 0.5 to 4 N. The setup employed platinized electrodes, with a solution circulation rate of 2.5 l/h.

2. Materials and methods

Sodium sulfate solutions were prepared using analytical grade Na_2SO_4 reagent in accordance with GOST 195–77 [12]. The chemical composition of the substance used is presented in Table 1.

Table 1. Composition of initial sodium sulfate

Name of indicator	Pure for analysis
Mass fraction of sodium sulfur dioxide Na_2SO_3, %, not less than	98
Mass fraction of water-insoluble substances, %, not more than	0.003
Acidity	Shall withstand the tests outlined in s. 3.4.
Mass fraction of alkali Na_2CO_3, %, not more than	0.05
Mass fraction of thiosulfates (S_2O_3), %, not more than	0.02
Mass fraction of chlorides (Cl), %, not more than	0.005
Mass fraction of iron (Fe), %, not more than	0.0005
Mass fraction of arsenic (As), %, not more than	0.00002
Mass fraction of heavy non-ferrous metals (Pb), %, not more than	0.0005

In the present work, experiments were conducted on three setups differing in design and solution circulation schemes, in order to compare key performance indicators.

For electrodialysis of sodium sulfate solutions with $MgSO_4$ admixture to obtain H_2SO_4 and NaOH, an installation in the form of rectangular plates and frames made of Plexiglas was made. The electrodialysis process was carried out in a three-compartment apparatus (Figure 1) using MC-40 and MA-41 ion-exchange membranes [13, 14]. According to the manufacturers' specifications, the average electrical resistance of both membrane types is in the range of 10.0-11.0 $\Omega\cdot$cm^2.

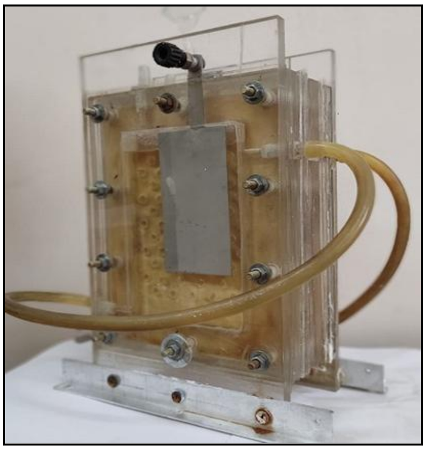


Figure 1. Electrodialyzer 3-section

The initial sodium sulfate solution, with a concentration of 20-80 g/l and supplemented with $MgSO_4$, was fed into the central (working) compartment of the apparatus. The catholyte and anolyte were prepared as follows:

– catholyte, distilled water with the addition of 2 ml of 30% NaOH solution;

– anolyte, distilled water with the addition of 2 ml of 300 g/l H_2SO_4 solution.

A platinized electrode served as the anode, with dimensions of 50×100×2 mm and an anode surface area of 37.5 cm^2; the cathode was made of stainless steel, sized 55×100×1 mm with a surface area of 41.25 cm^2.

Experiments were conducted varying the following parameters:

– current intensity from 0.4 to 1.0 A;

– electrodialysis duration from 2 to 8 hours;

– $MgSO_4$ impurity concentrations of 2, 4, 6 and 8 g/l.

Figure 2 shows an improved design of a three-compartment electrodialyzer with solution circulation and pressurized tanks. The total solution volume, including the pressurized tanks, was 600 ml.

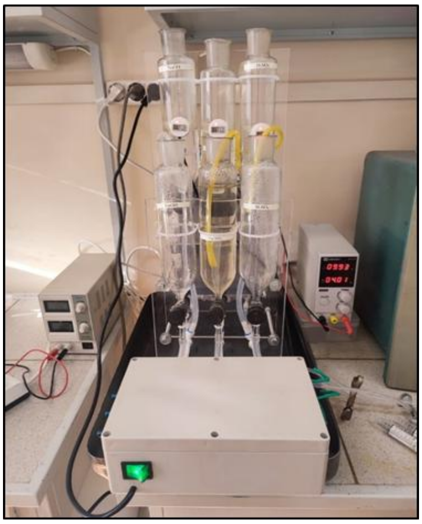


Figure 2. Electrodialysis plant with continuous circulation of solutions

Electrodialysis was performed using ion-exchange membranes EDC1R and EAC1R (PRC) [15, 16]. According to the manufacturers' data, the electrical resistance of these membranes averages 4.5-5.5 $\Omega\cdot$cm^2. The setup consists of six compartments arranged in the following order: cathode – cation-exchange membrane (CEM) – anion-exchange membrane (AEM) – anode – AEM – CEM – cathode. The distance between compartments is 10 mm. A platinized platinum (Pt) anode is installed in the central compartment, while iron (Fe) and titanium (Ti) cathodes are placed in the outer compartments.

Electrodialysis of sodium sulfate solutions on the pilot-scale setup was carried out in continuous circulation mode with simultaneous concentration of alkali and acid solutions (Figure 3). The volumes of H_2SO_4, NaOH, and Na_2SO_4 solutions were each 2 L. EDAM and EDCM [17] ion-exchange membranes with a specific electrical resistance of 2.5-3 $\Omega\cdot$cm^2 were used. A platinized electrode served as the anode, while a stainless-steel electrode was employed as the cathode.

In the last two setups, the circulation rate of electrolytes was controlled by adjusting the power supply voltage to the membrane pumps and by the electrolyte level in the pressurized tanks. The densities of the Na_2SO_4, H_2SO_4, and NaOH solutions were measured using a D6 Excellence digital densitometer.

Figure 3. Bench installation for electrodialysis of Na_2SO_4 solutions

Electrodialysis experiments with sodium sulfate solutions were conducted following the probabilistic-deterministic experimental design method developed by V.P. Malyshev [18-20].

3. Results and discussion

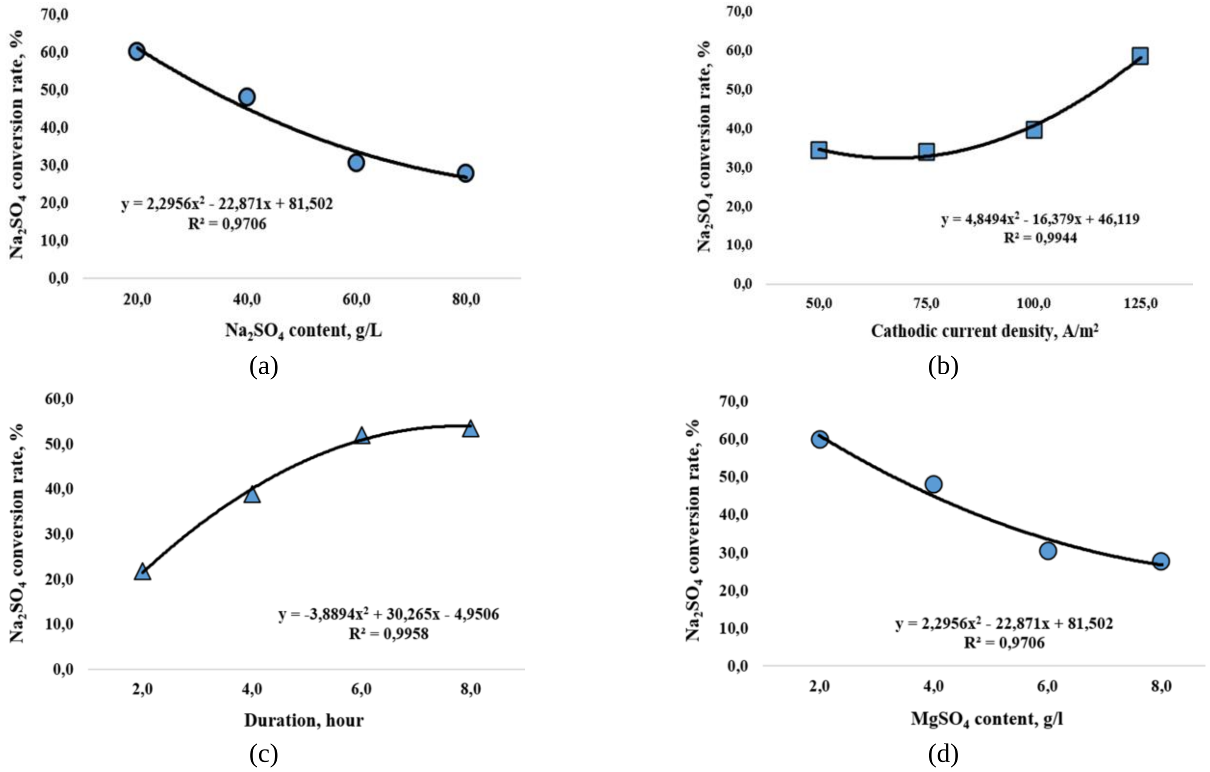
The conditions of experiments on electrodialysis of sodium sulfate with magnesium sulfate admixture in a 3-chamber electrodialyzer, and the results of experiments are given in Table 2.

Table 2. Conditions of experiments on electrodialysis of Na_2SO_4+$MgSO_4$ solutions

N	Na_2SO_4, g/l	$MgSO_4$, g/l	Current intensity, A	Duration, hour	$Y_{(i)}$ exp
1	20	4.00	0.60	4	63.92
2	20	4.00	0.40	2	16.01
3	20	4.00	1.00	8	93.05
4	20	4.00	0.80	6	66.7
5	40	2.00	0.60	2	34.66
6	40	2.00	0.40	8	53.1
7	40	2.00	1.00	6	53.42
8	40	2.00	0.80	4	50.67
9	60	8.00	0.60	2	13.53
10	60	8.00	0.40	8	35.55
11	60	8.00	1.00	6	48.43
12	60	8.00	0.80	4	24.61
13	80	6.00	0.60	2	22.97
14	80	6.00	0.40	8	32.35
15	80	6.00	1.00	6	39.22
16	80	6.00	0.80	4	16.48

Figure 4 presents the dependencies of the conversion degree of the solution on the initial Na_2SO_4 concentration, cathodic current density (ranging from 50 to 125 A/m^2), electrodialysis duration and $MgSO_4$ impurity content.

Electrodialysis was performed at cathode current densities, $d_{(k)}$, ranging from 50 to 125 A/m^2. According to the presented graphs, the degree of Na_2SO_4 conversion increases with both the current density and the duration of the process.



(a) (b) (c) (d)

Figure 4. Dependence of the Na_2SO_4 conversion degree on various factors: (a) – initial Na_2SO_4 concentration; (b) – cathodic current density; (c) – electrodialysis duration; (d) – $MgSO_4$ impurity concentration

Figure 5 illustrates the dependence of the current strength on the duration of electrodialysis.

The gradual decrease in current strength at the end of the sodium sulfate electrodialysis process under a constant voltage is due to the reduction in salt concentration in the central compartment, which in turn leads to a decrease in the solution's electrical conductivity.

Figure 6 shows the dependence of specific energy consumption during electrodialysis of sodium sulfate solution on the initial Na_2SO_4 concentration, cathode current density, and $MgSO_4$ impurity content. Figure 6a shows that at high initial sodium sulfate concentrations (60-80 g/l), the specific energy consumption for electrodialysis is approximately 5 kWh per 1 kg of Na_2SO_4.

With an increase in current density (Figure 6b), energy consumption increases accordingly.

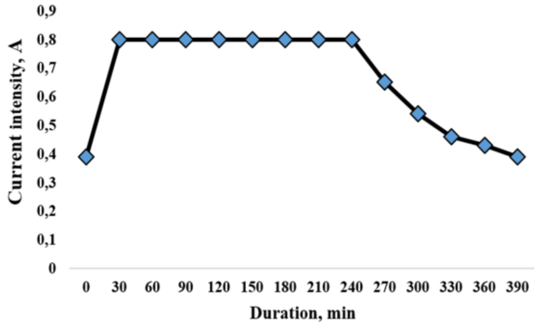
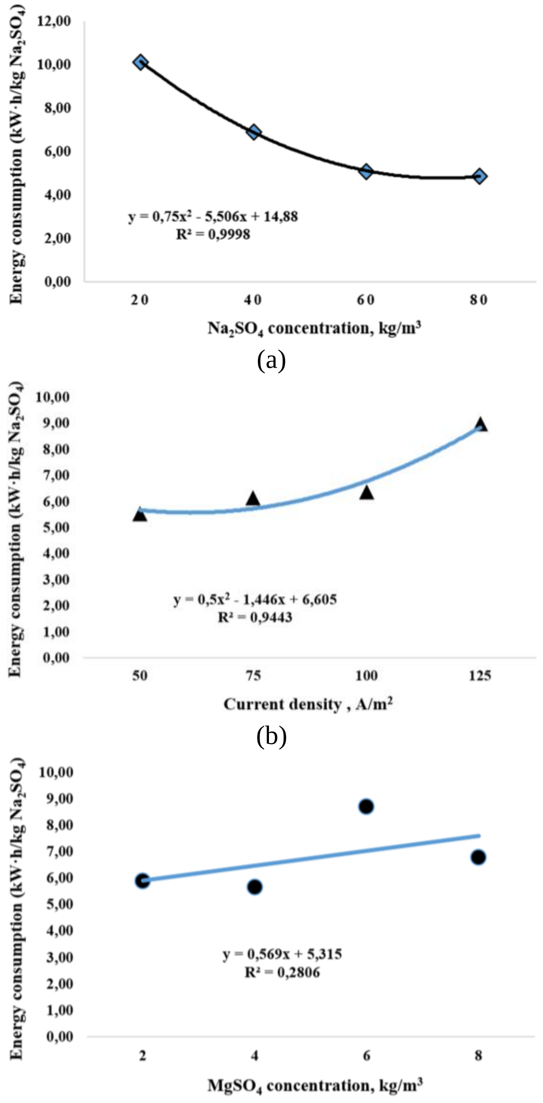


Figure 5. Dependence of current strength on the duration of Na_2SO_4 solution electrodialysis



(a)

(b)

(c)

Figure 6. Dependence of specific energy consumption during electrodialysis on various factors: (a) – initial Na_2SO_4 concentration; (b) – current density; (c) – $MgSO_4$ impurity concentration

In the circulation setup, a dependence (Figure 7) was established between energy consumption per unit mass of salt and an average current density at the platinized anode of 60 A/m^2. At sodium sulfate concentrations ranging from 60 to 150 g/l, energy consumption was found to be in the range of 4-7 kW·h/kg.

Reducing the salt concentration in the solution entering the electrodialysis leads to a significant increase in energy consumption from 7 to 34 kW·h/kg. In the apparatus the distance between the cathode and anode was 3 cm. The increase in energy consumption was attributed to the rise in electrical losses along the circuit: platinized anode – H_2SO_4 solution – anion-exchange membrane – Na_2SO_4 solution – cation-exchange membrane – NaOH solution – steel cathode. Depending on the temperature and composition of the solutions in the electrodialysis cell compartments, the total electrical resistance of the system ranged from 2.8 to 10.9 Ω.

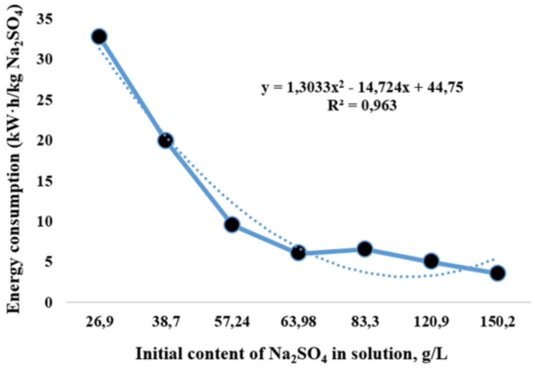


Figure 7. Dependence of specific energy consumption per 1 kg of Na_2SO_4 on the initial sodium sulfate concentration

For comparison of electrodialysis performance, experiments were conducted on a pilot-scale setup equipped with 10 pairs of ion-exchange membranes. The conditions of the sodium sulfate electrodialysis experiments, as well as the conversion degree values obtained on the pilot setup at different initial solution concentrations and current densities, are presented in Table 3.

Table 3. Conditions of experiments

N	Na_2SO_4 g/l	Current density, A/m^2	Duration, min	Na_2SO_4 conversion rate, %
1	86.3	17.02	600	8.7
2	78.8	14.15	1140	13.73
3	66.7	13.16	1080	7.20
4	61.9	23.10	300	5.53
5	79.6	118.57	240	36.53
6	50.5	60.24	300	22.92
7	39.0	89.64	300	42.33
N	NaOH circulation volume, L	H_2SO_4 circulation volume, L	Na_2SO_4 circulation volume, L	
1	14.2	14.6	13.9	
2	71.86	75.45	69.1	
3	67.19	65.92	65.68	
4	21.48	19.46	19.23	
5	17.68	12.77	12.98	
6	23.97	19.90	23.91	
7	20.94	21.51	22.17	

At duration of electrodialysis from 4 to 19 hours, consumption of initial solution of sodium sulfate (Na_2SO_4) 13.9-69.1 liters and current density 13.2-118.6 A/m^2 the degree of conversion of sodium sulfate was 5.5-42.33%. The energy consumption for the electrochemical process was 0.95-2.11 kW·h per 1 kg of Na_2SO_4.

Figure 8 shows the dependence of energy consumption for electrodialysis on the initial concentration of sodium sulfate.

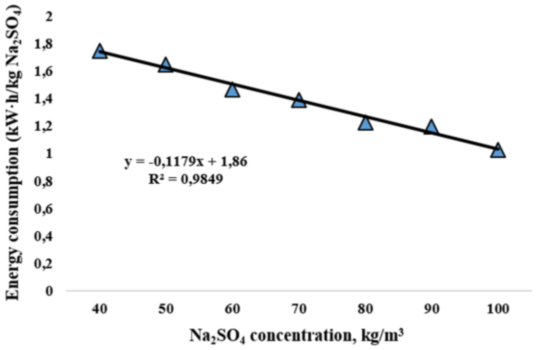


Figure 8. Dependence of energy consumption for electrodialysis on initial concentration of sodium sulfate

For predicting the required duration of the electrodialysis process of sodium sulfate solutions to achieve the desired degree of conversion, a dependence of the degree of conversion on the current density was established. Based on the data from Table 3, the values of current density and degree of conversion were recalculated using interpolation. Figure 9 presents the dependence of the degree of conversion of Na_2SO_4 on the current density at an electrodialysis duration of 1 hour and an average salt concentration of 65 g/L.

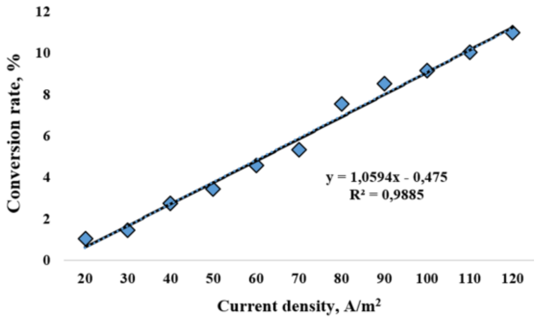


Figure 9. Dependence of conversion rate on current density

Figure 9 clearly demonstrates that an increase in current density leads to a consistent increase in the degree of conversion of sodium sulfate during the electrodialysis process.

4. Conclusions

A series of experiments by the method of planning experiments with solutions of Na_2SO_4, in the presence of magnesium sulfate impurity $MgSO_4$ at concentrations from 2 to 8 g/l was carried out. During the electrodialysis process, the possibility of magnesium precipitation as a hydrated precipitate was identified, along with the simultaneous production of target products – alkali and acid.

A series of experiments was conducted using the experimental design method with sodium sulfate (Na_2SO_4) solutions of varying concentrations. The methodology was refined, and dependencies of the key electrodialysis parameters were obtained under steady-state conditions without solution circulation. This enabled the development of an approach for predicting the process duration required to achieve complete conversion of the initial solution, as well as determining the calculated number of electrodialysis stages needed for operation of the unit in continuous flow mode.

The main performance indicators of the sodium sulfate electrodialysis process, determined based on solution density and controlled according to Faraday's law, are the degree of conversion and the specific energy consumption per unit mass of Na_2SO_4.

Dependences of sodium sulfate conversion degree on initial solution concentration, current density during electrodialysis and process duration are described by polynomials of the second degree.

The scaling up of sodium sulfate solution electrodialysis technology is significantly limited by the high specific energy consumption for salt processing. From this perspective, the use of multi-chamber electrodialyzers is advisable, as they enable an increase in the energy efficiency of the process.

Author contributions

Conceptualization: BSB; Data curation: AAD, GZM; Formal analysis: YBT, AAD, BSB; Funding acquisition: VSM, GZM; Investigation: YBT, GZM; Methodology: BSB, GZM; Project administration: BSB, VSM; Resources: AAD, GZM; Software: BSB; Supervision: VSM, YBT; Validation: YBT, VSM; Visualization: AAD, BSB; Writing – original draft: AAD, BSB; Writing – review & editing: BSB, YBT. All authors have read and agreed to the published version of the manuscript.

Funding

The research was carried out within the framework of grant funding from the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan for 2023-2025 in the priority area «Rational use of natural resources, including water resources, geology, processing, new materials and technologies, safe products and structures», project No. AP19677216 «Research and development of technologies, equipment for electrodialysis of solutions of tungstate, sodium sulfate with the regeneration of alkalis, acids».

Acknowledgements

The authors express their sincere gratitude to the editor and anonymous reviewers for their constructive comments and valuable suggestions, which have significantly improved the quality of this manuscript.

Conflicts of interests

The authors declare no conflict of interest.

Data availability statement

The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding author.

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Сілті және қышқыл регенерациясымен натрий сульфаты ерітінділерінің электродиализінің мембраналық процестерін зерттеу

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Аңдатпа. Бұл жұмыста әртүрлі өндірістік процестерде түзілетін натрий сульфаты (Na_2SO_4) ерітінділерін электродиализ әдісі арқылы құнды өнімдерге – күкірт қышқылына (H_2SO_4) және натрий гидроксидіне (NaOH) қайта өңдеу мүмкіндігі қарастырылады. Натрий сульфаты ерітінділерін қайта өңдеу мәселесі олардың көп мөлшерде түзілуімен және бейорганикалық қоспалардың жоғары құрамымен өзекті, ал дәстүрлі тазарту әдістері энергияны көп қажет етеді және экономикалық тұрғыдан тиімсіз. Авторлар МК-40, МА-41 және МВ-2И, сондай-ақ Ralex BM мембраналарын қолдана отырып, натрий сульфаты ерітінділерін электродиализ арқылы өндіру мен қайта өңдеудің қолданыстағы әдістеріне талдау жүргізді. Электродиализ жүргізу үшін катион- және анионалмастырғыш МК-40, МА-41 (РФ) және EDC1R, EAC1R (ҚХР) маркалы мембраналары қолданылған 3 камералы және 6 секциялы электродиализатор сұлбалары пайдаланылды. Сондай-ақ EDAM және EDCM маркалы мембраналар қолданылған көп камералы стендтік қондырғының сұлбасы қарастырылды. Үш қондырғыда тәжірибелер жүргізу шарттары В.П. Малышевтің ықтималдық-детерминистік эксперименттерді жоспарлау әдісімен құрастырылды. Бірінші қондырғы үшін келесі тәуелділіктер алынды: Na_2SO_4 концентрациясынан конверсия дәрежесі, катодтық ток тығыздығы мен $MgSO_4$ қоспасының мөлшеріне байланысты процесс ұзақтығы. Бастапқы ерітіндінің концентрациясы мен ток тығыздығына байланысты энергия шығынының үш қондырғы бойынша алынған тәуелділіктері электродиализ процесінің орындылығын бағалауға мүмкіндік береді.

Негізгі сөздер: *натрий сульфаты, электродиализ, мембраналық процестер, натрий гидроксиді, күкірт қышқылы.*

Исследование мембранных процессов электродиализа растворов сульфата натрия с регенерацией щелочи и кислоты

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Аннотация. В данной работе рассматривается возможность переработки растворов сульфата натрия (Na_2SO_4), образующихся в различных промышленных процессах, в ценные продукты – серную кислоту (H_2SO_4) и гидроксид натрия (NaOH) с использованием метода электродиализа. Проблема переработки растворов сульфата натрия актуальна в связи с их значительным объемом и высоким содержанием неорганических примесей, при этом традиционные методы очистки являются энергоемкими и экономически нецелесообразными. Авторами проведен анализ существующих методик получения и переработки растворов сульфата натрия электродиализом с использованием мембран МК-40, МА-41 и МБ-2И и мембран Ralex BM. С целью проведения электродиализа применены схемы 3-х камерного и 6-ти секционного электродиализаторов с использованием катионо-, анионообменных мембран марок МК-40, МА-41 (РФ) и EDC1R, EAC1R (КНР). Рассматривалась схема стендовой установки многокамерного электродиализатора с использованием мембран марки мембран EDAM и EDCM. Составлены условия проведения экспериментов на 3-х установках по методу вероятностно-детерминированного планирования экспериментов Малышева В.П. Получены зависимости для первой установки: степень конверсии от концентрации Na_2SO_4, продолжительности процесса от катодной плотности и содержание примеси $MgSO_4$. Зависимости расхода энергии от концентрации начального раствора и плотности тока на 3-х установках позволяют судить о целесообразности проведения процесса электродиализа.

Ключевые слова: *сульфат натрия, электродиализ, мембранные процессы, гидроксид натрия, серная кислота.*

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