

UDC 628.16 THE USE OF A TWO-CHAMBER ELECTROLYSER FOR THE OXIDATION OF AMMONIUM SULPHATE

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Abstract

Electrolysis methods were used to purify water from ammonium compounds in a two-chamber electrolyzer. An increase of ammonium sulphate concentration leads to increase of oxidation intensity, which is associated with the concentration factor and the increase of the electrical conductivity of solutions. The increase of ammonium concentration from 0.56 to 6.67 mg-eq/dm³ allows to increase the current output to a certain extent, but an increase in the current density leads to a decrease of this indicator. In the process of electrolysis at initial ammonium concentrations of 0.56-6.67 mg-eq/dm³ at a current density of 86.2–172.4 A/m², the pH-level of the medium in the anolyte decreases to ~2, what is explained by the oxidation of ammonium to nitrates with the subsequent formation of nitric acid, which leads to acidification. It has been established that when using solutions prepared with tap water at the same concentrations, ammonium oxidation is faster than in solutions prepared with distilled water. This method is highly efficient (99–100 %) and provides reliable water purification from ammonium. *Keywords:* water desalination; electrodialysis; ammonium sulphate; current output.

ВИКОРИСТАННЯ ДВОКАМЕРНОГО ЕЛЕКТРОЛІЗЕРУ ДЛЯ ОКИСНЕННЯ АМОНІЮ СУЛЬФАТУ

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Анотація

Для очищення води від сполук амонію використовували методи електролізу у двокамерному електролізері. За умови підвищення концентрації амонію сульфату спостерігається підвищення інтенсивності окиснення, що пов'язано як з концентраційним фактором, так і підвищенням електропровідності розчинів. Підвищення концентрації амонію від 0.56 до 6.67 мг-екв/дм³ дозволяє до певної міри збільшити вихід за струмом, проте підвищення густини струму призводить до зниження даного показника. У процесі електролізу за вихідних концентрацій амонію 0.56-6.67 мг-екв/дм³ та густині струму 86.2-172.4 А/м² спостерігається зниження pH середовища в аноліті до ~2. Це пояснюється окисненням амонію до нітрату з послідуючим утворенням нітратної кислоти, що і призводить до підкислення. Встановлено, що на разі використання розчинів, приготованих на водопровідній воді, за однакових концентрацій амонію, окиснення останнього проходить швидше, ніж в розчинах, приготованих на дистильованій воді. Даний спосіб є високоефективним (99-100 %) та забезпечує надійне очищення води від йонів амонію.

Ключові слова: знесолення води; електродіаліз; амонію сульфат; вихід за струмом.

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Introduction

Water resources in Ukraine are unevenly distributed, which creates an urgent need for an efficient water supply system. Surface water is the main source of water supply for municipal, industrial and agricultural sectors. The densest hydrographic network is in the western, northern and central parts of Ukraine, while in the south of the country there is a lack of fresh water for fullfleged providing of the population, a situation has got critical after the terrorist attack on the Kakhovka Hydroelectric Power Plant. In the eastern part of the country, the situation is extremely catastrophic: in addition to the scarcity of surface water, the region is characterized by high levels of pollution, largely caused by industrial facilities and the military-industrial complex, making the only potential water bodies available for water supply almost impossible to use.

Today, the issue of uninterrupted water supply to the population is still relevant. The current state of the water supply and sewerage sector is in a dilapidated and inadequate condition and has a number of unresolved problems: lack of financial resources necessary for the proper operation and maintenance of water supply systems, unsatisfactory condition of facilities and equipment, resulting in huge losses of resources during transportation, imperfect management of the sector and the regulatory framework to ensure its effective functioning [1]. Due to the low availability of water resources, the policy of efficient water use should include measures aimed at minimizing resource losses during transportation and optimal use of waste water for its reuse. To address these issues, it is necessary to develop complex low-waste water desalination technologies, such as mine water [2; 3] or mineralized water from various contaminants [4; 5]. To address these issues, it is necessary to develop complex low-waste water desalination technologies, such as mine water [2, 3] or mineralized water from various contaminants [4; 5].

The problem of excessive concentrations of nitrogen-containing compounds is increasingly common when water is abstracted from surface or groundwater sources. Although nitrogen is a major component of air and is naturally transformed in the geosphere, its excess in certain forms can cause disruption to ecosystems and make water treatment more complicated.

Sources of ammonium nitrogen can be accidental leaks and discharges from food, coke

and chemical enterprises, as well as discharges from municipal water supplies. However, the main and most significant source of ammonia and ammonium pollution is agriculture. As a result of animal breeding, ammonia is formed during the decomposition of organic material; when it interacts with rainwater, ammonia is converted into ammonium ions and enters the hydrological system through infiltration. The greatest danger is posed by the uncontrolled use of nitrogen fertilisers to stimulate plant growth. For example, ammonium fertilisers, which include ammonium sulphate, ammonium chloride and ammonium nitrate, have gained some popularity due to their effectiveness in increasing yields. Excessive use of ammonium fertilisers increases the content of ammonium ions and dissolved ammonia in groundwater, which can cause pollution of connected surface waters. The danger of ammonia pollution of surface waters is bioaccumulation, poisoning of the aquatic life and its subsequent death, which disrupts the self-regulation of the ecosystem. With excessive decomposition of organic residues, the concentration of ammonia will only increase, which can lead to the active development microalgae, of i.e. cause eutrophication of the water body. In addition to the danger of disrupting the stability of aquatic ecosystems, ammonium ion and ammonia pollution poses a certain threat to the human body. Prolonged consumption of poorly treated water contaminated with ammonium nitrogen can lead to high blood pressure, acid-base imbalance, disruption of the nervous and reproductive systems, and liver, kidney and lung diseases.

Recently, many studies have been conducted on water purification from ammonium ions [6], including biological purification [7–9], chemical precipitation [10], ion-exchange [11; 12], sorption on natural inorganic sorbents [13–16], catalytic oxidation [17] and membrane desalination [18; 19]. However, these methods have their own disadvantages and advantages (Fig. 1).

Biological methods [20; 21] usually combine several processes, such as partial nitrification, denitrification, and anaerobic oxidation of ammonium ions [22]. These methods [23] are characterised by high efficiency [24] in wastewater treatment and are among the most widely used methods. However, in the case of groundwater, artesian and surface water purification from ammonium ions, their use is complicated, since natural waters are quite pure and do not contain organic carbon, phosphates, and often nitrites and nitrates in sufficient concentrations. In this case, it is difficult to activate biochemical treatment processes, and there is a high risk of high residual ammonium concentrations in the treated water. In addition, these methods are very sensitive to changes in temperature [25], and the water treatment time is quite long and sometimes reaches several days, which is impractical and unacceptable in modern water treatment technologies. The disadvantages of these methods also include high-energy consumption and high process costs.

Reagent methods [26] have a moderate process cost, are quite effective both in the treatment and post-treatment of wastewater containing ammonium in significant concentrations, but require certain pH-level and temperature values and are accompanied by significant reagent consumption. The treatment process produces waste that requires additional costs for its processing. It is advisable to use fairly accessible and relativelv inexpensive magnesium compounds as a precipitant [27], but in this case, phosphates, what are quite valuable substances [28], are removed to waste. The main disadvantages of these methods are the lack of efficiency at low ammonium concentrations and additional water pollution by the reagents used.

Ion-exchange methods have shown high efficiency in the removal of biogenic pollutants, in particular nitrates and phosphates [29; 30]. These methods [31-33] also provide effective ammonium removal from water at a relatively low cost, but these methods are effective only when water is completely desalinated. Since complete softening is not required, and natural waters always contain significant concentrations of calcium and magnesium ions, ion-exchange water purification from ammonium ions will be inefficient and economically inexpedient due to the need to recycle used regeneration solutions.



Fig. 1. Methods of treatment for ammonia removal in water and wastewater

Therefore, methods based on the use of natural sorbents are more effective, but the process of ammonium sorption on zeolite [34] is very slow. It is worth noting that different adsorbents have different capacities, but with a short contact time between water and the sorbent, the capacity of almost all ammonium sorbents is small and decreases sharply with a decrease in the process time. In addition, at low ammonium concentrations in water, the sorption capacity of natural sorbents is insignificant.

Catalytic oxidation allows for fast and efficient water treatment, but these methods are quite expensive and require high temperature and pressure. The use of oxidants such as hydrogen peroxide or ozone is appropriate for moderate ammonium concentrations in water, but in this case, there is a significant overconsumption of oxidising agents to reduce the ammonium content to acceptable levels [35].

The main problems in the application of reverse osmosis methods [36] are the rather complicated problems of concentrate utilisation.

At present, the processes of electrochemical ammonium oxidation are virtually unexplored. Therefore, we used electrochemical methods to purify water from ammonium.

The aim of this study is to investigate the electrochemical oxidation of ammonium and to determine the effect of water composition, electrolysis time and current density on the efficiency of ammonium oxidation in a two-chamber electrolyzer.

The following tasks were set:

- to determine the effect of solution composition, process time and current density on

 $B = \frac{V_s(C_{in} - C_i)}{K_F \cdot I \cdot t} \cdot 100, \tag{1}$

The specific electrical conductivity (Ohm⁻¹·m⁻¹) in the electrolyzer was calculated by the formula:

$$\chi = \frac{L \cdot I}{U \cdot S'} \tag{2}$$

Electricity consumption (kWh/g-eq) was calculated using the formula:

$$\Pi_{\rm e} = \frac{I \cdot \tau \cdot U \cdot M_{\rm e}}{\Delta C \cdot V_{\rm p}},\tag{3}$$

where V_s – the volume of solution, dm³; C_{in} – the initial concentration of ions in solution, g/dm³; C_i – the concentration of ions in the ith sample, g/dm³; K_E – the electrochemical equivalent (K_E = $M_e \cdot 0.03731$ g-eq/(A·h)); M_e – the equivalent mass of the transferred substance (M_e = M/m), g-eq; M – the molar mass of the transferred substance

the efficiency of ammonium oxidation processes in a two-chamber electrolyser;

- to determine the effect of water composition on the efficiency of electrochemical oxidation of ammonium sulphate solutions.

Materials and methods

In the process of ammonium sulphate electrolysis, model solutions were used in tap and distilled water with an ammonium concentration of 0.56–5.00 mg-eq/dm³ and sulphate concentration of 0.56–6.67 mg-eq/dm³.

The electrolysis processes were carried out in a two-chamber electrolyser with an anionic membrane MA-41. The alloy steel plate 12X18H10T was used as a cathode, while a titanium plate coated with ruthenium oxide served as an anode. The area of the electrodes was $SC = SA = 12 \text{ cm}^2$, and the current strength was 0.1 and 0.2 A.

The working solution was placed in the anode chamber, and the cathode chamber contained a 0.05–0.2 N solution of NaOH. In some studies, 100 mg/dm³ of sulphates or 30 mg/dm³ of chlorides were added to the cathode chamber. Samples were taken from the anolyte at regular intervals. In general, the electrolysis time was 30–270 min. The residual ammonium concentration and pH-level were determined in the samples.

The current output (V, %) was calculated as the ratio of the actual amount of substance transferred during electrolysis to the theoretical amount of substance calculated according to Faraday's law:

Results and Discussion

At the first stage of research, we studied the oxidation of ammonium at its initial concentrations of ammonium sulphate in the anolyte from 0.56 to 2.22 mg-eq/dm³ in distilled and tap water. Figure 2a shows that even without the addition of chlorides, the anolyte undergoes a fairly intense oxidation of ammonium.

With an initial ammonium concentration in the distilled water solution of 0.56 mg-eq/dm³ at a current density of 86.2 A/m², the ammonium concentration in the sample decreases by 0.39 mg-eq/dm³ in 30 minutes. Thus, ~69.6 % of the ammonium present in the water is oxidised within 0.5 hours.

At the same time with the same current density, at an initial ammonium concentration of 1.11 mg-eq/dm³, \sim 39.6 % is oxidised, i.e. its amount decreases by 0.44 mg-eq/dm³, what is close to the values at an initial concentration of 0.56 mg-eq/dm^3 . Since in both cases the electricity consumption was almost the same at 0.69-0.70 A-h/dm³, the amount of oxidised ammonium is approximately the same. In the second case, a slightly higher value of 0.44 mg-eq/dm³ can be explained by a 2-fold higher initial concentration of ammonium in the solution and a higher current output of oxidised ammonium - 4.19% and 3.19 %, respectively, initial when the concentration was reduced from 1.11 to 0.56 mg-eq/dm^3 (Fig. 2b).

In 30 minutes, when the current density is doubled from 86.2 A/m² to 172.4 A/m², the electricity consumption increases to 1.40 A-h/dm³, which allows oxidising 0.62 and 0.67 mg-eq/dm³ of ammonium at its initial concentrations of 1.67 and 2.22 mg-eq/dm³ respectively. Increasing the ammonium concentration allows to increase the current output to a certain extent, but increasing the current density leads to a decrease in this indicator.

In the process of electrolysis at initial ammonium concentrations of 0.56-2.22 at a current density of 86.2-172.4 A/m², a decrease in the pH-level of the medium in the anolyte is

observed. The acidification of the solution can be explained by the decomposition of ammonium, which leads to the transition of sulfuric acid into the solution, and acidification of water is also possible due to the formation of nitrogen dioxide. Due to the low concentrations of alkali in the catholyte and salt in the anolyte, the electrical conductivity of the solution was low in all cases.

At a current density of 86.2 A/m^2 and initial concentrations of ammonium sulphate of 0.56–2.22 mg-eq/dm³, the rate of ammonium oxidation is slightly higher than the rate of ammonium chloride oxidation at the same concentrations.

As can be seen from Fig. 2a, when using solutions prepared with tap water with the same concentrations, ammonium oxidation is faster. In this case, at a current density of 86.2 A/m² complete oxidation of ammonium occurs in 45 and 75 minutes at an initial concentration of 0.56 and 1.11 mg-eq/dm³, increasing the concentration to 1.67–2.22 mg-eq/dm³ requires a longer process (90 minutes). The current yield was higher in comparison with distilled water solutions, but in all cases it was rather low – 4.41–10.04 % (Fig. 2c).

The initial pH-levels of ammonium sulphate solutions prepared with tap water (7.16–7.29) is significantly higher compared to distilled water (5.43–5.97), which is explained by the presence of hydrocarbonates, which provide neutral pH values in the initial solutions. Since the tap water in Kyiv has the following chemical composition, the concentration of calcium ions is ~2.9– 3.2 mg-eq/dm^3 , magnesium – 0.9–1.5 mg-eq/dm³, sulphates and chlorides – 14.9–53.1 mg/dm³, and hydrocarbons – 3.8–4.7 mg-eq/dm³. However, in the course of electrolysis, the pH-level of the anolytes decreased rather rapidly to values of ~2.0 (Fig. 2c).

The increase of the ammonium concentration leads to an increase in current output and a decrease in electricity consumption (Fig. 4). During the electrolysis, $(NH_4)_2SO_4$ in both tap water and distilled water practically does not lead to a significant increase in the electrical conductivity of solutions (Fig. 2d).





Fig. 2. Dependence of the ammonium concentration (a), current output (b), pH (c), and electrical conductivity of water (d) during ammonium oxidation in the anolyte on the time of electrolysis in a solution of distilled (1; 2; 3; 4) and tap water (5; 6; 7; 8) at a current density of 86.2 A/m² (1; 2; 5; 6; 7; 8) and 172.4 A/m² (3; 4) at initial ammonium concentrations ((NH₄)₂SO₄), mg-eq/dm³: 0,55 (1; 5), 1,11 (2; 6), 1,67 (3; 7), 2,22 (4; 8).

At a current density of 86.2–172.4 A/m² in solutions prepared with distilled water, the ammonium concentration was reduced to only 0.07-0.14 mg-eq/dm³. At a current density of 86.2 A/m^2 , it was possible to completely oxidise ammonium in tap water. Thus, the redox processes in the anolyte were most likely not affected by hardness ions. Presumably, the presence of chlorides in tap water and their possible oxidation in a slightly acidic and acidic environment accelerates the process. When the concentration of ammonium sulphate is increased to 2.78, 5.00, and 6.67 mg-eq/dm³ in distilled water, complete ammonium oxidation occurs in 60, 120, and 150 minutes at a current density of 172.4 A/m² (Fig. 3a).

The process of complete oxidation of ammonium sulphate with an initial ammonium concentration of 5.00 mg-eq/dm³ took 105 minu-

tes, i.e. the process was 15 minutes faster in tap water compared to distilled water (Fig. 3a).

In this case, the current yield of oxidised ammonium with concentrations of $0.56-2.22 \text{ mg-eq/dm}^3$ in distilled water was in the range of 1.24-7.68 %. With an increase in concentration to $2.78-6.67 \text{ mg-eq/dm}^3$, the current yield reached 7.37-31.2 % (Fig. 3b). In all cases, a decrease of pH-level of the medium from 5.43-7.04 in the initial solutions to values of ~ 2 after 60–150 minutes of electrolysis was observed (Fig. 3c).

With an increase in the concentration of ammonium sulphate, an increase in the intensity of oxidation is observed, which is associated with the concentration factor and the increase in the electrical conductivity of solutions. Due to the increase in the number of charged particles in the anolyte at the beginning of the process and their decrease at the end of the process, which is explained by the oxidation of ammonium ions, as well as the probable removal of nitrogen oxides from the water, the voltage change pattern shows clearly defined zones of increased electrical conductivity and decreased voltage (Fig. 3d). A solution of ammonium sulphate in tap water has a higher electrical conductivity compared to distilled water. With an increase of the ammonium concentration in the solution, the electricity consumption required for ammonium oxidation decreased. However, there was no significant difference when using both distilled and tap water (Fig. 4).



Fig. 3. Dependence of the ammonium concentration (a), current output (b), pH (c), electrical conductivity (d) of water in the anolyte on the electrolysis time in a solution of distilled (1; 2; 3) and tap water (4; 5) at a current density of 172.4 A/m² at initial ammonium concentrations ((NH₄)₂SO₄), mg-eq/dm³: 2.78 (1; 4), 5.00 (2; 5), 6.67 (3)



Fig. 4. Dependence of electricity consumption for the oxidation of ammonium sulphate on the composition of the solution prepared with distilled (a) and tap water (b) and the electrolysis time

Since the process of ammonium oxidation is accelerated at a low concentration of chlorides in the anolyte, it is advisable to catalyse this process by gradually adding chlorides to the anode chamber. To implement this idea, it is advisable to introduce chlorides into the catholyte, after which they will pass through the anionic membrane together with hydroxide anions and enter the anode chamber.

It is worth noting that the formation of oxidised chlorine compounds accelerates the complex processes of ammonium oxidation, while the destruction of ammonia slows down with increasing sulphate concentrations. Obviously, this fact can be explained by a side reaction that results in the formation of persulfate anions on the anode. The formed persulfate anions hardly oxidise ammonium, which results in a certain slowdown in the destruction of ammonia during electrolysis.

Ammonium sulphate was prepared in distilled and tap water, and the results of its electrochemical oxidation in the anolyte are shown in Figure 4. Thus, the introduction of chlorides at a concentration of 0.84 mg-eq/dm³ into the catholyte does not significantly accelerate the process of ammonium oxidation. In addition, the rate of ammonium oxidation is approximately the same in distilled and tap water solutions. There is also no difference in the current output of oxidised ammonium and in the dynamics of changes in the pH-level of the solutions (Fig. 5).

Thus, the main advantage of the ammonium oxidation process prepared in a tap water solution is a reduction in energy consumption for ammonium oxidation due to the higher electrical conductivity of the system.



Fig. 5. Dependence of ammonium concentration (1; 2), current output during ammonium oxidation (3; 4), pH (5; 6) and specific electrical conductivity (7; 8) in the anolyte on the time of electrolysis at a current density of 172.4 A/m², at an initial ammonium concentration ((NH₄)₂SO₄) of 2.78 mg-eq/dm³ in a solution of distilled (1; 3; 5; 7) and tap (2; 4; 6; 8) water, at a concentration of [Cl⁻] = 0.84 mg-eq/dm³ in the cathode chamber

The results show that when water is passed through the anode chamber of a two-chamber electrolyzer, effective oxidation of ammonium sulphate is possible in a wide range of initial concentrations. The basic flow diagram of natural and wastewater purification from ammonium is shown in Fig. 6. As it can be seen from the above flow chart, water containing ammonium sulphate is first fed into the receiving chamber (1). Then, using a pump (2), the water is fed to the electrolyser (3), where ammonium is oxidised in the anode area, water is disinfected and organic impurities are oxidised, and then it is fed to the purified water tank (4).



Fig. 6. Schematic flow chart of natural and waste water treatment from ammonium: 1 - intake chamber; 2, 5 - pumps; 3 - two-chamber electrolyser; 4 - purified water tank; I - water supply for treatment; II - water supply to the consumer

The proposed technology for water purification from ammonium sulphate is highly efficient and ensures reliable water purification.

Conclusions

1. The processes of ammonium oxidation in a two-chamber electrolyser have been studied as a function of solution composition, process time, and current density. It is shown that an increase in both the concentration and current density leads to an increase in the oxidation efficiency.

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2. The oxidation rate of ammonium sulphate is higher in tap water solutions compared to distilled water, which is explained by the higher concentration of chlorides, which are the catalysts of the process, in tap water. Reduced energy consumption for ammonium oxidation due to the higher electrical conductivity of the system is the main advantage of ammonium extraction from tap water compared to distilled water.

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