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CONVERSION OF AQUEOUS AMMONIA SOLUTIONS USING AN ADAPTIVE WATER PURIFICATION SYSTEM

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Abstract. *Water purification from ammonium nitrogen is currently an urgent task for protecting drinking water sources and ensuring the required water quality for consumers in various sectors of the Ukrainian economy. The technological approaches covered in this article can be used to remove other ammonium-based compounds from water. The obtained results of the adaptive water purification system (AWPS) with a simulated highly concentrated aqueous ammonium solution (1,16 g/dm³) suggest their use in water purification technologies from hydrobionts and side-products of biogas systems (digestat purification). Pulsed electrochemical methods were used in the operation of the AWPS in combination with ultrasonic exposure on oxidation-reduction processes with controlled injection of gas mixtures. A high correlation between all the variables studied was established. The same was confirmed by the regression analysis made in the process of empirical modeling of the relationship between the treatment time and the change in pH and in the concentrations of ammonium nitrogen, nitrates and nitrites. That is, all the indicators of multiple correlation and determination in the constructed significant models indicate a very high correlation with the treatment time. The decrease in pH from 11 to 9.6 can be explained by the fact that acids were formed during the treatment of the model solution, which caused the decrease.*

The energy efficiency of the AWPS operation was assessed by analyzing changes in the concentrations of the main component of the simulated solution (ammonium) and its derivatives using the example of nitrites and nitrates and a fixed operating time of 60 min. The use of electrolysis methods allowed the conversion of ammonium in an aqueous solution into derivatives – an aqueous solution of nitrites and nitrates, to record changes in their concentrations, and when using membrane electrolysis to obtain them in an ionic form, which is optimally suitable for plant nutrition and direct synthesis of nitrogen-containing fertilizers with simultaneous application by irrigation or spraying.

Keywords: *water purification, ammonium, nitrites, nitrates, pulsed electrolysis, adaptive power source, current form, adaptation*

Relevance of the research. The entry of ammonium nitrogen and its derivatives into aqueous solutions is a common phenomenon. The traditional ways of entry of these substances (natural ones with organic compounds and manmade ones with industrial effluents) are supplemented now by technogenic ones, including the results of military hostilities in the territory of Ukraine.

The development of modern or improving existing technological approaches to the purification or conversion of nitrogen-containing compounds into useful forms will be constantly relevant. At present, the development of new technological solutions for hydrobiont cultivation systems is promising, and the existing approaches to closed water supply systems (RAS) require improvement, especially from the perspective of the development of hydroponics and aquaponics. Moreover, the issue of purification of biogas system discharges, the so-called digestate, has not been resolved globally. Phosphorus and other

substances are also present in the digestate, and we expect that electrochemical methods will also affect them, converting them into less toxic or useful derivatives in a regulated technological mode. The filtrate of water effluents from solid waste landfills also has excess concentrations of nitrogen forms and its purification is an important task.

Analysis of recent research and publications. Modern technological solutions for the purification of aqueous solutions from ammonia and ammonium use a diverse range of approaches, depending on the historical factors of their formation. The most common methods include biological (most often used in RAS), reagent, ion exchange, electrochemical, and sorption [2–5]. All of them have their strengths and weaknesses. In the recent publication on electrochemical purification of aqueous solutions from ammonium, there is a grounded approach [4, 5] but its key disadvantages are the non-recirculation of the technological cycle, the absence of pulsed current loading of the electrolyzer electrodes and the absence of ultrasound, which reduces the efficiency of conversions.

Given the importance of the development of renewable energy sources (biogas systems), industrial hydrobiont cultivation systems (RAS), and combined hydroponics and aquaponics systems, the development of technologies on ammonium purification and conversion into other forms from aqueous solutions [8–9] is the subject of research and development by many scientists.

Research objective.

Known progressive solutions [4, 5] use the correct basis, but the most promising are electrochemical methods that use electrolysis methods as controlled ones with high development potential. In our solutions described in previous publications [10–13], we have foreseen the advantages of electrochemical combined methods and considered their various technological variations, which were implemented when creating the AWPS. The difference between these solutions is that we additionally use pulsed electrolysis with an adaptive power source, additional regulated oxidation or reduction, and a more complex recirculation hydraulic scheme, which expands the possibilities of settings and the effect of electrolysis methods in combination with ultrasound. The task was to investigate the operation and energy efficiency of the developed ASOV under its action on a concentrated aqueous solution of ammonium.

The purpose of the research is to research the processes of ammonium conversion into nitrates and nitrites when using the AWPS and, based on the results obtained, to determine the efficiency of water purification and energy efficiency of individual units and the AWPS as a whole.

Materials and research methods. An artificially simulated aqueous solution was prepared from water from a well in a 200 l container with the addition of 0,5 l of a 25 % aqueous ammonium solution. 30 l of the model solution was fed into the AWPS per 1 experiment. The operating temperature at the beginning of the experiments was 19 °C.

The ammonium conversion process was going in recirculation mode through an electrolyzer with insoluble anodes (ORTA) on a titanium base, which contains 4 electrodes of 0,1 m × 0,2 m. The current partially passes through a group (3 pcs.) of electrolysis two-sided turbo hydrocyclones with an insoluble graphite anode with a diameter of 20 mm and a height of 20 cm. pH control was made using a pH-150 MI pH meter. To control the parameters of pulse electrolysis, a FNIRSI DSO-138 mini-oscilloscope was used, built into an adaptive power supply unit, and connected to the load voltage output terminals of the electrolyzer electrodes. The power source provided a current of ~ 45 V electrodes with a pulse electrolysis frequency of 3 Hz to the electrolyzer, and the pulse shape front increase mode was set. To record and control energy consumption for technological processes of AWPS, a PEACEFAIR wattmeter of PZEM-008 model with a maximum permissible current of 100 A, and an accuracy of ± 1 % was used. The total area of the system electrodes involved was 0,24 m^2.

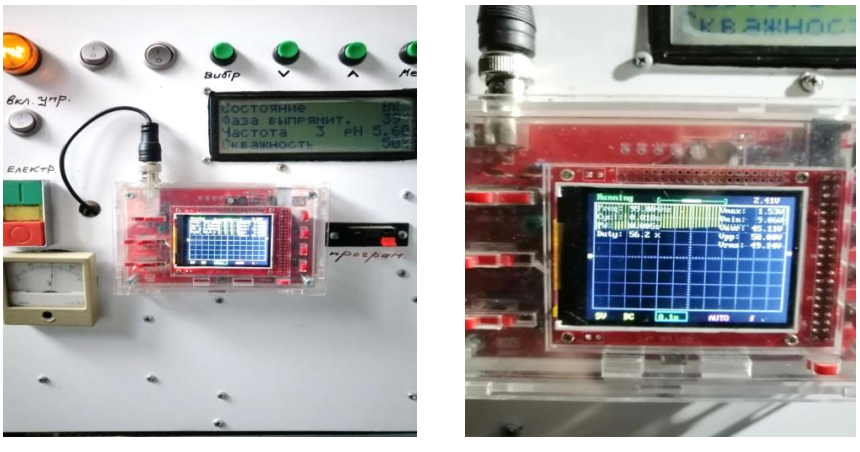
Chemical changes of the model solution during the operation of the AWPS were studied using three samples in the Laboratory of Hygiene of Natural Drinking Water of the State Institute named after O.M. Marzeev of the National Academy of Medical Sciences of Ukraine. The PI7.2/13 method was used for determining ammonium, the PI7.2/12 method – for nitrites, and the method according to DSTU 4078-2001 – for nitrates.

Research results and their discussion. AWPS when using an adaptive power supply unit with a pulsed supply mode to the electrolyzer electrodes is shown in Fig. 1. The basic functional and technological as well as general hydraulic diagram was given in [13].

The adaptive power supply of AWPS under operation is presented in Fig. 2.

The wattmeter for monitoring of the total energy consumption of AWSP is presented in Fig. 3.

Fig. 1. General view of AWPS



a b

Fig. 2. Overview of the adaptive power supply of electrolysis units

a – control and parameter monitoring panel, including an integrated portable oscilloscope; b – oscilloscope in operating mode

a) b) c) d) e) f)

Fig. 3. Data of the wattmeter on the general control of energy consumption during operation and conducting experiments:

a – energy consumption for ultrasound; b – energy consumption for electrolysis (dynamic); c – energy consumption per a pump (variable, there are two of them in the system); d – total energy consumption for the entire operation of AWPS along with the pumps (2 pcs.), electrolysis, ultrasound and control systems at the beginning of the experiment; e – after 20 min.; f – after 60 min

The actual electrode load current ranged from 1,84 to 3A depending on changes in the electrical conductivity of the model solution. It increases when ammonium is converted to nitrites and nitrates, as stronger electrolytes than ammonium are synthesized.

The current density is calculated by the largest current parameter 3 A/0,24 m^2=12,5 A/m^2.

The changes in the energy consumption of AWPS during the experiment are presented in Fig. 4.

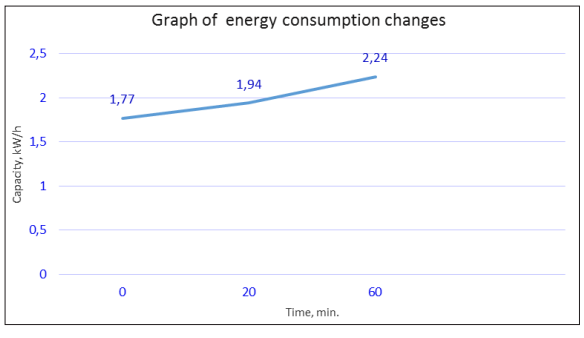


Fig. 4. Graph of changes in the energy consumption of AWPS

From the graph in Fig. 4 it is clear that during the experiment a change in the energy consumption of the unit was observed in the range from 1,77 to 2,24 kW·h, which indicates a change in the electrical conductivity of the model solution. Taking into account the volume of the latter in AWPS, the specific energy consumption for a change in the ammonium concentration from 1,16 g/dm^3 to 0.48 g/dm^3 is 66,8 W/l.

The results of the experiments are presented in the form of graphs (Fig. 5). As we can see from the presented dependences, a 2,5-fold decrease in the ammonium concentration is clearly observed during one-hour treatment of the solution. The concentration of nitrites and nitrates increases similarly.

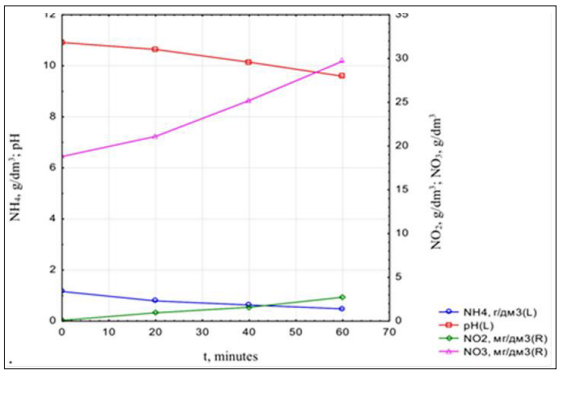


Fig. 5. Graphs of the changes in the studied indicators that occurred during the treatment of the model solution

During the operation of the unit, electrochemical processes occur, which are determined both by the composition of the source water and the specifics of the electrolyzers. For example, the synthesis of the coagulant is determined, among other things, by the Cl-content and the electrode material:

$$Fe + 3Cl^- + 3e = FeCl_3,$$
$$FeCl_3 + 3H_2O = Fe(OH)_3\downarrow + 3HCl.$$

The synthesis of the antiseptic – oxidant goes in a similar way due to the presence of a membrane – a ceramic semi-permeable membrane of 2 mm thick with an area equal to the area of the electrodes:

$$2NaCl + 2H_2O = 2NaOH + H_2\uparrow_{(cathode)} + Cl_2\uparrow_{(anode)}.$$

In the anode space, partial hydrolysis occurs with the formation of chloride and hypochloride acids:

$$Cl_2 + H_2O = HCl + HClO.$$

Synthesized in the cathode space NaOH is used to neutralize acids formed as a result of hydrolysis:

$$HCl + NaOH = NaCl + H_2O.$$

The study of correlations between the studied indicators was made using Pearson's parametric correlation and is presented as a Table 1.

1. Establishing mutual correlations

	t	**NH_4**	**NO_2**	**NO_3**	**pH**
t	1,000	–0,972	0,993	0,990	–0,989
NH_4	–0,972	1,000	–0,957	–0,931	0,928
NO_2	0,993	–0,957	1,000	0,991	–0,990
NO_3	0,990	–0,931	0,991	1,000	–1,000
pH	–0,989	0,928	–0,990	–1,000	1,000

Correlations marked in red are significant when p <0,05000

The empirical model of the change in NH_4 concentration during the treatment of the model solution depending on the treatment time is given as equation (1).

$$NH_4\_M = 1{,}095 - 0{,}011t, \quad (1)$$

where NH_4_M – model of change in NH_4 concentration, g/dm^3; t – treatment time, min.

Model characteristics are: multiple correlation – R = 0,972; determination – R^2 = 0,945; adjusted determination – R^2 = 0,917; Fisher F parameter (1,2) = 34,326; p < 0,02792; standard error of estimate is 0,084.

A comparison of the NH_4_M model and the results of the observations of changes in NH_4 concentration is shown in Fig. 6.

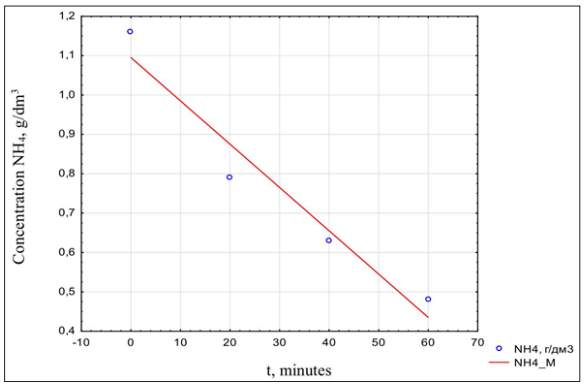


Fig. 6. Comparison of the NH_4_M model and field results during the treatment process

The empirical model of the change in NO_2 concentration during the treatment of the model solution depending on the treatment time is given as equation (2).

$$NO_2\_M=0,055+0,043t, \quad (2)$$

where NO_2_M – model of the change in NO_2 concentration, mg/dm^3; t – treatment time, min.

Model characteristics are: multiple correlation – R = 0.993; determination – $R^2 = 0.987$; adjusted determination – $R^2 = 0,98$; Fisher F parameter (1,2) = 148,73; p < 0.0067; standard error of estimate is 0,157.

Comparison of the NO_2_M model and the results of the observations of the change in NO_2 concentration are shown in Fig. 7.

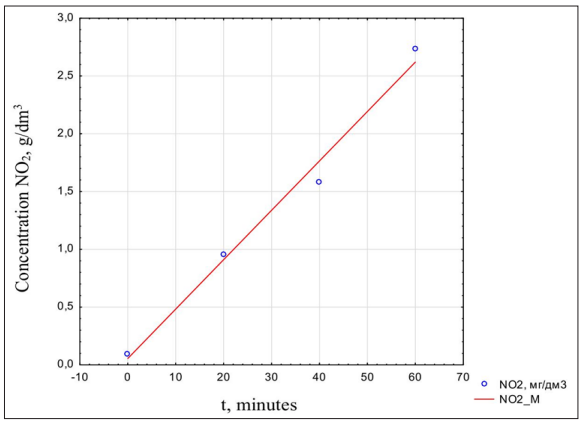


Fig. 7. Comparison of the NO_2_M model and field results during the treatment process

The empirical model of the change in NO_3 concentration during the treatment process depending on the treatment time is given as equation (3).

$$NO_3\_M= 18,18 + 0,184t, \quad (3)$$

where NO_3_M – model of the change in NO_3 concentration, mg/dm^3;

t – treatment time, min.

Model characteristics are: multiple correlation – R = 0,99; determination – $R^2 = 0,981$; adjusted determination – $R^2 = 0,97$; Fisher F parameter (1,2) = 103,54; p < 0,00952; standard error of estimate is 0,8087.

Comparison of the NO_3_M model and the results of the observations of the change in NO_3 concentration are shown in Fig. 8.

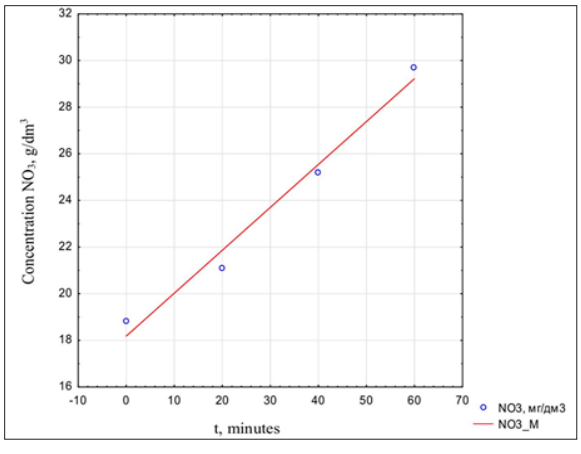


Fig. 8. Comparison of the NO_3_M model and field results

The empirical model of pH change during the treatment of the model solution depending on the treatment time is given as equation (4).

$$pH\_M= 10,989-0,0223t, \quad (4)$$

where pH_M is a model of pH change; t – treatment time, min.

Model characteristics are: multiple correlation – R = 0,989; determination – $R^2 = 0,979$; adjusted determination – $R^2 = 0,969$; Fisher F parameter (1,2) = 93,74; p < 0.001050; standard error of estimate is 0,103.

Comparison of the pH_M model and the results of the observations on pH change are shown in Fig. 9.

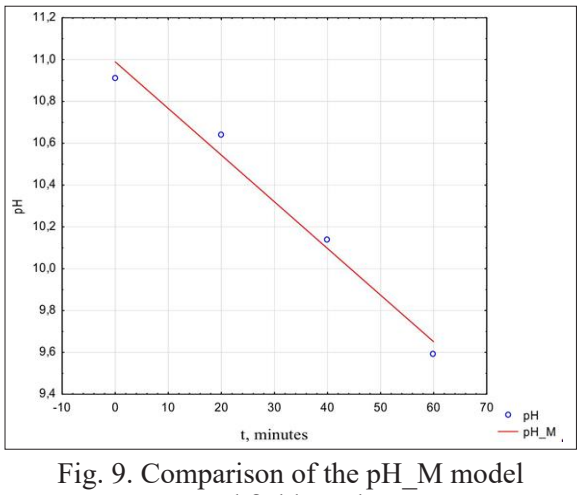


Fig. 9. Comparison of the pH_M model and field results

Conclusions

1. A high correlation was established between all the studied variables. This statistically confirms the influence of the treatment period on changes in the studied pH indicators and changes in the concentrations of nitrogen compounds.

It was confirmed by the regression analysis made in the process of empirical modeling of the relationship between treatment time and changes in pH indicators and in the concentrations of ammonium nitrogen, nitrates and nitrites. That is, all indicators of multiple correlation and determination in the developed significant models indicate a very high correlation with treatment time.

2. The decrease in pH from almost 11 to 9,6 can be explained by the fact that acids were formed during the treatment of the model solution, which caused the decrease.

3. The decrease in NH_4 concentration can be explained by the partial transition of nitrogen to compounds in the form of nitrates and nitrites and the possible formation of atomic nitrogen from ammonia nitrogen during the electrolysis process with its release into the atmosphere.

4. Probable chemical processes that occurred during the treatment of the model solution are:

– nitrites formed during the electrochemical oxidation of ammonium can be additionally converted into nitrates as a result of the reaction:

$$NO_{2^-} + O = NO_{3^-}, \quad (5)$$

when using oxygen from the decomposition products of water.

When an electric current passes through the treated water, atomic oxygen, hydrogen peroxide, and free radicals, which are strong oxidants, can be obtained as a result of electrochemical reactions. And if the treated water contains more than 20 mg/dm^3 of chlorides, there are simultaneous reactions to form chlorine, chlorine dioxide, and hypochlorous acid, which are capable of disinfecting water containing residual chlorine.

The main reactions occurring at the electrodes are:

at the anode:

$$2H_2O \rightarrow O_2 \uparrow + 4H^+ + 4e; \quad (6)$$

at the cathode:

$$2H_2O + 2e \rightarrow H_2 + 2OH^-, \quad (7)$$

at the anode:

$$3H_2O \rightarrow O_3 \uparrow + 6e + 6H^+; \quad (8)$$

at the cathode $2H_2O + O_2 + 2e \rightarrow H_2O_2 + 2OH^-$, (9)

at the anode: $2Cl^- \rightarrow Cl_2 + 2e$; 10)

$$Cl_2 + H_2O \rightarrow HClO + H^+ + Cl^-; \quad (11)$$

$$Cl_2 + OH^{-} \rightarrow HClO + Cl^-. \quad (12)$$

The direct electrolysis method in various versions enables us to purify water from iron, hydrogen sulfide, ammonium, manganese, reduce water hardness, remove taste, color, turbidity, and disinfect from bacteria and viruses.

Below are the main reactions occuring during the removal of ammonium.

$$2NH_3 + 3O_2 \rightarrow 2HNO_2 + 2H_2O, \quad (13)$$

$$NH_3 + 2O_2 \rightarrow HNO_3 + H_2O, \quad (14)$$

$$NH_3 + 4H_2O_2 \rightarrow HNO_3 + 5H_2O, \quad (15)$$

$$NH_3^- + 4HClO \rightarrow HNO_3 + 4HCl + H_2O. \quad (16)$$

5. The energy consumption for the AWSP operation is insignificant. When all the system blocks are under operation, the total energy consumption was from 1,77 kW to 2,24 kW, and the specific energy consumption was 66,8 W/dm^3.

6. The parameters of the electrolyzer with insoluble electrodes were previously determined. In our opinion, it is advisable to maximize the time of remaining the working solution in the interelectrode space. The feasibility of using the recirculating structure of the hydraulic circuit combined with the use of an adaptive power source for electrolyzers with a changing shape of the pulse current and the use of an electrolyzer of an original design with built-in ultrasonic emitters has been proven.

7. The proposed AWSP system is promising for constructing on its basis more complex systems for purification and disinfection of aqueous solutions containing complex combined pollutants of high concentration, for systems of aqueous solutions regeneration, purification of leachate effluents from solid waste landfills, effluents from biogas systems (digestate), mine waters and other multi-element and highly concentrated aqueous solutions.

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ПЕРЕТВОРЕННЯ ВОДНИХ РОЗЧИНІВ АМОНІЮ ПРИ ЗАСТОСУВАННІ АДАПТИВНОЇ СИСТЕМИ ОЧИЩЕННЯ ВОДИ

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Анотація. *Очищення води від амонійного азоту нині є актуальним завданням для захисту джерел питного водопостачання та забезпечення необхідної якості води для споживачів різних галузей економіки України. Технологічні підходи, висвітлені в цій статті, можуть бути використані для видалення із води інших сполук на основі амонію. Отримані результати роботи адаптивної системи очищення води (АСОВ) із змодельованим висококонцентрованим водним розчином амонію (1,16 г/дм³) передбачають їх використання в технологіях для очищення води після гідробіонтів та біогазових систем (очищення дигестату). У роботі АСОВ застосовували імпульсні електрохімічні методи у поєднанні з ультразвуковим впливом, окисненням і частковим відновленням з контрольованим інжектуванням газових сумішей. Встановлено високу кореляцію між усіма досліджуваними змінними величинами. Те ж підтвердили і регресійний аналіз, виявлений у процесі емпіричного моделювання зв'язків часу обробки зі зміною показників pH, та зміни концентрацій амонійного азоту, нітратів і нітритів. Тобто всі показники множинної кореляції і детермінації в побудованих значимих моделях свідчать про дуже високий кореляційний зв'язок з часом обробки. Зниження pH від майже 11 до 9,6 можливо пояснити тим, що в процесі обробки модельного розчину утворювались кислоти, які і стали причиною цього зниження. Проведено оцінювання енергоефективності роботи АСОВ та ефективності системи через аналізування змін концентрацій головного компонента змодельованого розчину (амонію) та похідних речовин від нього на прикладі нітритів і нітратів та фіксованим часом роботи 60 хв. Використання електролізних методів дало змогу провести перетворення амонію у водному розчині на похідні – водний розчин нітритів та нітратів, зафіксувати зміну їх концентрацій, при використанні мембранного електролізу отримати їх в іонній формі, яка оптимально підходить для живлення рослин та прямого синтезу азотовмісних добрив з одночасним внесенням методом поливу чи розпилення.*

Ключові слова: *очищення води, амоній, нітрити, нітрати, імпульсний електроліз, адаптивне джерело живлення, форма струму, адаптація*