Визначено технологічні рішення для зниження концентрації сполук нітрогену на 75 % в процесі очищення стічної води з метантенків після зброджування посліду птахів. Показано вплив структури косубстрату на зміну вмісту сполук нітрогену у стічній воді в процесі отримання біогазу з посліду. Визначено технологічні параметри (час гідравлічного утримання, співвідношення потоків) анаеробно-аеробного процесу очищення стоків

Ключові слова: зброджування посліду, косубстрат, стічна вода, нітрифікація-денітрифікація, анаеробно-аеробний процес

Определены технологические решения для снижения концентрации соединений азота на 75 % в процессе очистки сточной воды из метантенков после сбраживания помета птиц. Показано влияние структуры косубстрата на изменение содержания соединений азота в сточной воде в процессе получения биогаза из помета. Определены технологические параметры (время пребывания воды в системе, соотношения потоков) анаэробно-аэробного процесса очистки стоков

Ключевые слова: сбраживание помета, косубстрат, сточная вода, нитрификация-денитрификация, анаэробно-аэробный процес

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TECHNOLOGY OF ANAEROBIC-AEROBIC PURIFICATION OF WASTEWATER FROM NITROGEN COMPOUNDS AFTER OBTAINING BIOGAS

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1. Introduction

In the anaerobic utilization of the waste of livestock breeding, especially of poultry breeding, there is a problem of wastewater utilizing after the separation of the solid phase in the separator at the outlet of a methane tank [1]. In the course of processing the waste of the biogas production, while loading the methane tank with 5 % of the dry substance at the volume of a methane tank of 3000 m^3 and with keeping raw materials there for 24 days, 90 m^3/day of wastewater are produced [2]. Wastewaters are highly concentrated and have a high content of nitrogen and ammonium nitrogen (up to 180 mg/dm^3), which complicates the technology of their purification and makes their dumping into natural bodies of water impossible. Therefore, the development of the technological solutions of the purification of the wastewater with the high concentration of ammonium ions is an urgent problem.

2. Analysis of scientific literature and the problem statement

To reduce the content of the ammonium compounds in the fermenter and in wastewater, the co-fermentation is used with adding to the remainder the crop wastes [3] (corn, cane, beet pulp [4], sparging and paper waste) or the food production wastes [5] in various ratios depending on the raw materials that are used as co-substrate. In this case, the remainder concentration makes about 30 % [6].

According to this approach, while increasing the population of birds, it is necessary to increase the amount of the agricultural wastes, the delivery of which increases the cost of biogas. Therefore, determining a co-substrate composition, the use of which reduces the content of nitrogen ions in the environment and increases the content of the remainder, is an important problem [7].

The technologies that are used for the utilization of ammonium ions in wastewater, various modifications of the biological methods are used [8]: sequential anaerobic-aerobic, or with the return of the wastewater into the anaerobic reactor, unoxidic processes and their combinations [9]. The methods are hardly effective, because reducing the concentrations of the nitrogen compounds mainly occurs at the expense of increasing the biomass of the microorganisms, but not due to the formation of the molecular N_2 [10], which is released into the atmosphere. Moreover, high concentration of ammonium ions can cause the death of the microorganisms or inhibiting their life processes.

One of the new methods is the use of *anammox*-bacteria [11]. Such technology with using special carriers for the immobilization of the microorganisms allows purifying the wastewater with ammonium nitrogen with the efficiency of 97 % [11] while using the ammonium nitrogen concentration of 150 mg/dm^3 [12]. Using sequentially a series of reactors with anaerobic, aerobic and unoxide conditions of the flow of biological processes, the authors of the work [13] managed to reach indicators of 2 mg/dm^3 at the outlet of wastewater constructions while purifying the wastewaters with ammonium nitrogen concentration of 20–50 mg/dm^3 at the inlet. It should be noted that in the offered decisions of purifying the wastewater from nitrogen compounds, the ammonium ion concentration up to 70 mg/dm^3 is used, which is much lower than in the wastewater after the fermentation of the remainder [14].

That is why this work is dedicated to the solution of the problem of the utilization of the nitrogen compounds in wastewater from the methane tank after the fermentation of the remainder with the view to its reusing.

3. The purpose and objectives of the study

The aim of the work is to develop a technological solution of reducing the concentration of nitrogen compounds in the wastewater from methane tanks.

To achieve this aim, the following tasks were solved:

– researching the influence of co-substrate structure regarding the content of ammonium ions in the wastewater from the methane tank;

– determining the time of hydraulic retention of wastewater in an aerobic reactor, depending on the concentration of ammonium ions and COD;

– establishing the ratio between the aerated and anaerated streams of wastewater for the implementation of the anammox-process.

4. Materials and methods of studying the way of purifying wastewater from the methane tank from the nitrogen compounds

4. 1. The studied materials and the equipment for determining the influence of co-substrate on the contents of ammonium ions in wastewater from a methane tank

The main substrate was the remainder of chickens with the humidity of 32 %. Paper (cellulose – 98 %, lignin – 2 %) [15], reed (cellulose – 43.5 %, lignin – 37 %) [16], hemp (cellulose – 34–48 %, hemicellulose – 21–37 %, lignin – 16–28 % depending on the grade) [17] were used as the co-substrate.

The choice of the co-substrates was based on:

– global increase in the use of paper as packing material;

– increase in the agricultural areas under hemp, the sorts of which do not contain any narcotic substances and are used for the production of paper and other substances, the wastes of which need recycling;

– reed overgrowth on the river banks of Ukraine and cheap methods of its collection and delivery;

– the largest output of biogas in the process of fermentation.

The fermentation of the remainder was carried out in the reactors, the volume of which is 1.8 dm^3, at the temperature of 36±2 °C for 21 days.

For the measurement of the ion concentration NH_4^+, NO_3^-,NO_2^-, the PH-meter I-160MI was used. The analysis is carried out by standard methods [18].

4. 2. Materials and equipment for studying the method of purifying waste water from nitrogen compounds

The installation with the volume 1,8 dm^3 where the process of the denitrification took place, has a mechanic stirrer in its set-up for the intensification of the mass exchange processes, and synthetic carriers for the immobilization of microorganisms association enriched with anammox-bacteria. The concentration of the biomass in an anaerobic reactor reaches 9–10 g/dm^3. The speed of stirring was 50–60 r/m.

The composition of the biogas that was obtained in the process of denitrification was determined with the help of the gas chromatographer LCM-8-MD (1987) by a standard method [19]. The chromatograph contains two columns, one for determining H_2, O_2, CH_4, N_2, the other – for determining CO_2. The column temperature is 50 °C, the temperature of the evaporator is 50 °C, the temperature of the detector is 50 °C, the detector current is 50 мA. The gas – carrier is argon, the gas flow speed is 30 cm^3/min. The volume of the gas sample on the first column is 2.5 cm^3, on the second one it is 1 cm^3.

The percentage content of gases – H_2, CO_2, N_2, CH_4 i O_2 in the gas mixture

$$C=KSM, \qquad (1)$$

where C is the percentage content of a certain gas component in a mixture; K is the coefficient of the gas component (according to Table 1); S is the area of the triangle on the chromatogram.

Table 1

The coefficients of gas components after the denitrification

Gas component	Coefficient, K
N_2	0,0069
O_2	0,0052
H_2	0,0016
CO_2	0,0217
CH_4	0,0024

Aeration was carried out in rectangular tanks, at the bottom of which there were two aerators with the size of pores of 0.4 mm. The air consumption accounted for 3.5 dm^3/min. The concentration of the biomass in the aerobic reactor reaches 6–7 g/dm^3.

Chemical oxygen comsumption was determined by a standard method [20].

4. Results of studying the method of purifying wastewater from the methane tank after fermentation of the remainder

To determine the influence of the structure of cellulose-containing raw materials on the process of utilization of the ammonium ions in methane fermentation and the reduction of its concentration in wastewater, three types of co-substrate were used: paper, reed, hemp, which gave the highest yield of biogas with the ratio of the components remainder/co-substrate 1:1. The content of nitrogen compounds and COD in wastewater after the fermentation (21 days) is listed in Table 2. Nitrates were not formed in the process of methanogenesis. It should be noted that the biogas contained up to 9 % of nitrogen with the use of hemp, and 7 % with the use of other co-substrates.

As it can be seen from the data of Table 2, with the use of paper and cane waste (example 1, 2) as co-substrate, the content of COD at the outlet from the methane tank by 1.5 times lower than with the use of hemp. At the same time, the concentration of ammonium ions with the use of hemp and paper waste (sample 1, 3) is 2 times lower than with the use of reed as a co-substrate in fermentation of the remainder. The composition of the co-substrate does not influence the concentration of nitrite ions. It should be noted that the nitrate ions during the fermentation of the remainder with any co-substrate are not formed.

For purifying wastewater from nitrogen, it is supposed to divide the decant into two streams. In one stream, the purification of the wastewater occurs in aerobic conditions, in the second one – in anaerobic. Fig. 1 shows the dynamics of the changes in the concentration of ammonium ions in wastewater depending on the period of water retention in the aerobic reactor.

Table 2

Contents of nitrogen and COD in the wastewater from methane tank

Indicator	1. Remainder – paper wastes (1:1)	2. Remainder – reed (1:1)	3. Remainder – hemp (1:1)
COD, mg O_2/dm^3	1200±50	1040±50	1800±100
NO_2^-, mg/dm^3	2,0±0,04	1,4±0,03	1,6±0,03
NH_4^+mg/dm^3	74±3,5	138±7	69±3,5

As it can be seen from Fig. 1, the oxidation of ammonium ions occurs in a different time interval depending on the composition of wastewater. So, in the systems with fewer organic compounds (1 and 2), the ammonium utilization starts earlier: in 45 minutes for system 1, and in 1 hour for system 2. With the concentration of COD 1800 mg O_2/dm^3 (system 3), the oxidation of ammonium ions begins in 2 hours and lasts for a longer period of time. From Fig. 1, it can be seen that the lower the concentration of COD, the faster the process of oxidation of ammonium nitrogen is (2).

To determine the maximum reduction of the concentration of the ammonium ions and the formation of nitrogen (N_2) in the anaerobic reactor, the ratio of the volumes of aerobic and anaerobic streams was investigated. The wastewater after the aeration process was settled and directed to the anaerobic reactor-denitrificator, in which there was the anaerobic purification of the other part of the wastewater after the separation. The microbial destruction of an organic substance and the reduction of the COD value took place in the reactor before the aerated water arrival. The time of retaining water in aerobic and anaerobic reactors is the same. The reactor contains fibrous carrier, on which the hydrobionts, enriched with anammox-bacteria, are immobilized.

Fig. 2 shows the change in the concentration of ammonium ions depending on the ratio of the streams that are directed to the aerobic reactor with a period of wastewater retaining for 1 hour for chart a; for 1.5 hours for chart b; and for 2 hours for chart c. The change in the concentration of ammonium ions in the reactor after bringing the aerobic stream was not taken into consideration.

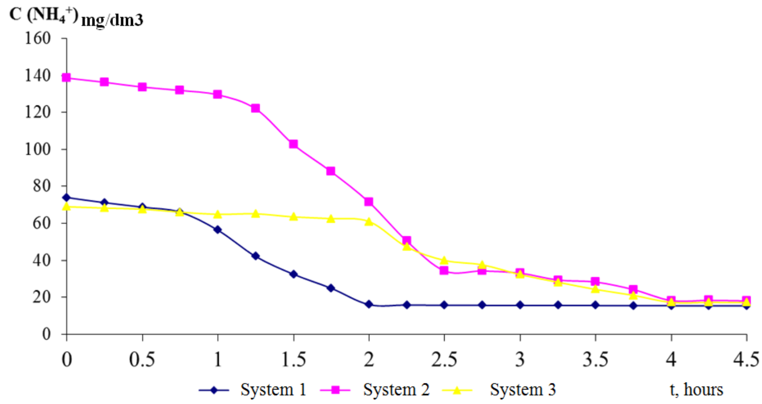

Fig. 1. Change of the concentration of ammonium nitrogen C (NH_4^+) depending on the time of hydraulic water retention (t) for various co-substrates: 1 — paper waste, 2 — reed, 3 — hemp (systems are listed in accordance with Table 2)

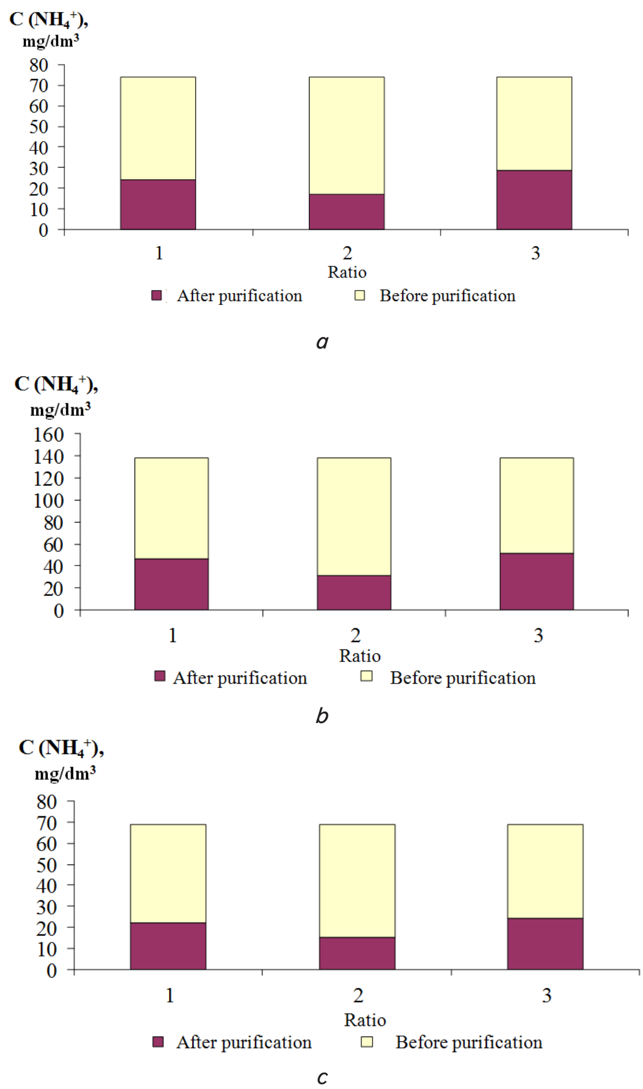

Fig. 2. Change of the concentration of ammonium ions (C (NH_4^+) at the outlet depending on the ratio of aerobic and anaerobic streams: *a* — 1:1, *b* — 2:1; *c* — 3:1, respectively

As it can be seen from Fig. 2, the largest reduction of the initial concentration of ammonium ions occurs while using the ratio of aerobic and anaerobic flows 2:1. According to the chromatographic analysis, the nitrogen content in biogas reaches 30 % while using the ratio of the streams from the aerobic and the anaerobic reactors for all systems 2:1. When one changes the ratio of the flows, the nitrogen content in biogas is reduced to 25 %±3 % with the use of the ratio of 1:1, and to 22 %±2 % with the use of the ratio of 3:1.

The degree of the purification of wastewater by the suggested method is listed in Fig. 3.

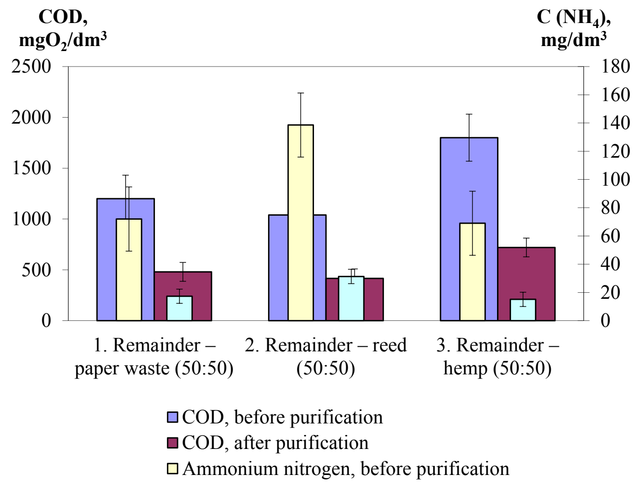

Fig. 3. Indicators of wastewater COD and C(NH_4^+) before and after the purification by the aerobic-anaerobic method with dividing the flows by the ratio of 2:1 with different hydraulic retention of the wastewater depending on the COD and the ammonium ion concentrations. The time of the retention: system 1 – 1 h; system 2 – 1,5 h; system 3 – 2 hrs

The set parameters of the process, which are listed in Fig. 3, allow significantly reducing the ammonium ions content in wastewater, which makes it possible to reuse it in the technological process of obtaining biogas from the poultry remainder.

5. Discussion of the results of reducing the concentration of nitrogen compounds in the wastewater from a methane tank

Based on the data of Table 2, we can state that the use of cellulose-containing raw materials with the low content of lignin (samples 1 and 3) reduces the content of the ammonium ions in wastewater by means of the primary destruction of hydrocarbon compounds, but not proteins, contained in the remainder, because the formation of ammonium ions (sample 2) occurs as a result of their destruction. The increase in lignin in the co-substrate to 37 % (sample 2) slows down the speed of the destruction of reeds, which leads to a decrease in the COD content in wastewater in relation to raw materials, containing less lignin (hemp 16–28 %).

The increased content of lignin also influences the development of the microorganisms contained in biocenose of a fermenter, because it makes the access to the biomass difficult and reduces the speed of the increase in the biomass. This leads to the increased content of the ammonium ions, which are formed through the decomposition of urea.

Thus, to reduce the content of the ammonium ions in wastewater from methane tanks in the process of the fermentation of the remainder it is necessary to use cellulose–containing raw materials which are low in lignin.

Based on the data in Fig. 2, the ratio of flows during decant splitting after the separation depends on the concentration of the ammonium ions and COD. With the increase in COD and in ammonium ions, the part of the water that is directed to the aerobic reactor, is larger than the other part which is directed to the anaerobic reactor. In the aerobic reactor, the utilization of organic substances is the first to occur, followed by the oxidation of nitrogen to NO_2^- [15]:

$$NH_4^+ \rightarrow NH_2OH \rightarrow (NOH) \rightarrow NO_2. \quad (2)$$

Under these conditions, the formation of NO_3^- takes place in the process of nitrification.

According to the received data (Fig. 2), the ratio 2:1 of the aerobic and anaerobic flows is appropriate.

The time of the hydraulic retention of wastewater in the aero tank depends primarily on the concentration of COD. As it can be seen from Fig. 1, the rational time of the water retention in the aero tank is, h: in the first one – 1–2 hours; in the second one – 1.5–2.5 hours; in the third one – 2–4 hours. The reduction of the COD content in the anaerobic process is slower than in the aerobic one, because the metabolism speed of aerobes is 3–6 times as high. So, the time of the hydraulic retention of water will be determined by the decrease in the value of COD in the anaerobic reactor.

When mixing the streams by using anammox-bacteria, formation of molecular nitrogen occurs according to the scheme:

$$NH_4^+ + NO_2^- \rightarrow N_2 + 2H_2O. \quad (3)$$

Molecular nitrogen also forms during the life of the other kinds of microorganisms with the consumption of the ions of ammonium and nitrate. In addition to molecular nitrogen in anaerobic reactor (denitrificator), the products of the methane fermentation (CH_4, H_2, CO_2) are also formed. However, unlike the biogas that is produced in a methane tank, the biogas from the denitrificator is low in calories due to the high content (30 %) of the molecular nitrogen (not flammable component) and CO_2 and can be used to remove oxygen from the water before stilling it after the aerobic reactor.

6. Technological process of purification of the wastewater from methane tank from the ammonium ions for its reusing

On the basis on the obtained data, the technological solutions of the anaerobic-aerobic purification of wastewater with the high content of ammonium nitrogen after the fermentation process of obtaining methane from the remainder were developed. The principle of technology lies in dividing the flow after the separation into two. The first is directed to the aerobic additional purification, where the process of nitrification simultaneously takes place. The second is directed to denitrificator, which contains the association of microorganisms enriched with anammox-bacteria. During the streams joining, the process of forming molecular nitrogen from ammonium and nitrite takes place in the denitrificator by equation (3). The scheme of the purification of wastewater from the organic compounds and the nitrogen compounds after the formation of biogas in the process of the fermentation of the remainder process is shown in Fig. 4.

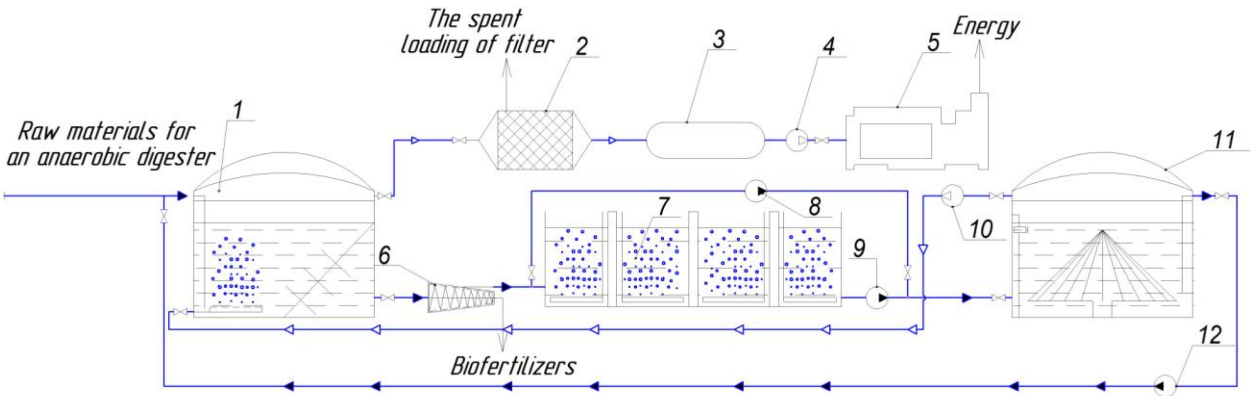

Fig. 4. Scheme of purification of the wastewater after methanogenesis: 1 — methane tank; 2 — filter for biogas purification; 3 — gas holder for accumulating purified biogas; 4 — compressor of the biogas supply for cogeneration; 5 — cogeneration plant; 6 — separator; 7 — aero tank; 8 — pump for wastewater pumping from the methane tank to denitrificator; 9 — pump of wastewater supply from the aero tank to denitrificator via the ejection system; 10 — pump for the supply of the gas formed in a methane tank for bubbling; 11 — denitrificator; 12 — pump for the purified water supply for reusing

Wastewater after separator 6, where the solid fraction is removed, is divided into two streams in the ratio of the aerobic: anaerobic purification (1.5–2): 1 and is supplied to aerobic 7 and anaerobic 11 reactors. A greater part of the wastewater arrives in aero tank 7, where its purification from organic compounds takes place and there is the process of nitrification with the formation of NO_3^- and NO_2^-. Another part, which is enriched with ammonium nitrogen NH_4^+, arrives in the anaerobic reactor 11, where it is purified from organic compounds and biogas is formed. Biogas with the help of the pump 10 is pumped for bubbling in the main methane tank 1.

After the process of aeration, the wastewater is pumped 9 to ejection, where the air is removed and its stilling takes place. The wastewater from the aero tank arrives in the anaerobic reactor 11, to which the water from the methane tank was directed 1. In anaerobic reactor 11, the process of denitrification takes place with the course of reactions (4) and:

$$NO_3^- + \text{organic substances} = N_2 + CO_2 + H_2O. \quad (4)$$

Reactor 11 is equipped with inert carriers to increase the number of microorganisms and hydrobionts, as well as to intensify the process of purifying wastewater. The anaerobic reactor 11 is also equipped with the special loading stirrer to intensify the process of the formation of molecular nitrogen.

After being purified from organic substances and the nitrogen compounds, the wastewater returns to the methane tank 1, where once again takes part in methane fermentation. The suggested technological solution allows using wastewater of methane tanks after purifying as circulating, which reduces the biogas costs and reduces the anthropogenic load on the environment. In addition to the positive environmental effect, the use of such technologies allows additional receiving biological fertilizers which contain all of the necessary components for the development of plants.

8. Conclusions

1. The concentration of ammonium ions and COD in the wastewater after the process of methanogenesis from the remainder is influenced by the component composition of the used co-substrate. Co-substrate, which has higher lignin content, contributes to increasing ammonium ions content twice in relation to the substrates, the lignin content of which does not exceed 25 %; besides, it does not increase the content of organic compounds in wastewater. Lower lignin content leads to increasing the content of organic substances in wastewater by 70 %.

2. With dividing the flow, the time of the hydraulic retention of wastewater in the aerobic reactor depends on the concentration of organic substances and ammonium ions. With the concentration of COD up to 1200 mg O_2/dm^3 and the concentration of ammonium ions up to 70 mg/dm^3, 1–2 hours is the rational time of the hydraulic retention of the wastewater in the aero tank, with increasing the concentration of ammonium ions up to 130 mg/dm^3 the rational time is 1.5–2.5 hours, with increasing the COD up to 1800 mg/dm^3 the rational time is 2–4 hours.

3. The suggested technology allows reducing the concentration of ammonium ions by 75 % by dividing the wastewater to conduct processes of nitrification and denitrification (1,5–2):1.

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