Problems of advanced nitrogen removal from municipal wastewaters in Kharkov (Ukraine)

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Abstract

Among the methods of nitrogen removal biological method is the most effective, economical and environmentally friendly. Deep removal of nitrogen compounds is carried out using microbiological nitrification, denitrification, the anammox process. The purpose of the work is to study composition of nitrogen pollution in wastewater, delivered into the Kharkiv WWTP, to determine the nitrifying capacity of activated sludge in aeration tanks and to demonstrate prospects of the treatment efficiency increase through an advanced nitrogen removal. Hydrochemical analysis of aqueous media was conducted via standard methods according to Ukraine standards demands The nitrifying capacity of activated sludge of treatment facilities was determined using the biochemical and the microbiological methods. It was found that in the municipal wastewater, entering the WWTP, organic nitrogen makes approximately 50 percent of the total nitrogen value. The total load of ammonium nitrogen and organic nitrogen on the treatment plants was extremely higher than the permitted level. It was determined that the concentration of nitrifying bacteria in the activated sludge and its enzymatic nitrifying activity were high enough for municipal wastewater treatment plants. The deep treatment process in the aeration tank with activated sludge includes deep nitrification, while anammox process and the denitrification process do not occur at all. $N\text{-}NH_4$ concentration after the treatment essentially decreases (up to 92.5%). The most perspective way to increase efficiency of the nitrogen compounds removal is to increase time of the aerobic treatment to allow advanced oxidation of organic compounds and to improve the nitrification process (decrease of residual ammonium nitrogen concentration) and organization of a denitrification process for deep removal of nitrites and nitrates.

Keywords: biological wastewater treatment, municipal wastewater, deep removal of nitrogen compounds, nitrification, nitrifying capacity of activated sludge, denitrification

1. Introduction

In countries where supply of potable water relies on surface waters (Central Europe, Northern America and Southern Africa) pollution by nitrogen and phosphorus compounds (eutrophication) is one of the most actual problems for maintaining ecological safety (EPA, 2009). To prevent eutrophication of water bodies and restore aquatic environment quality it is necessary to minimize emission of nutrients into receivers. The sources of these elements are the discharge of insufficiently treated municipal and industrial wastewater from treatment plants (WWTP) and flushes from agricultural and urban areas (storm wastewater (Gmur, 2003; Henze et al., 2004; Malovanyy et al., 2014; Iurchenko et al., 2020).

The decrease of nutrients content is possible through preliminary treatment of wastewater directly at the source (mainly in case of industrial effluents), and/or application of modern technologies increasing efficiency of treatment processes at municipal WWTP. Since, the first alternative can be problematic due to the technical and economical reasons, the second way – the increase of efficiency of nitrogen compounds removal – is considered as a viable option. Given that eutrophication of natural sources of drinking water becomes a global problem the implementation of highly efficient removal techniques for nutrients (nitrogen and phosphorus) is necessary.

Generally, nitrogen compounds are partially removed from the municipal wastewater during a conventional biological treatment processes performed in the activated sludge tanks (Gmur, 2003; Henze et al., 2004; Semenova et al., 2012; Gnida et al., 2016). Generally, the conventional biological treatment process is efficient enough. However sometimes a restrictive requirements regarding the composition of discharged wastewater may be exceeded.. Such efficiency also does not satisfy modern requirements for protection of natural reservoirs against the eutrophication. In Ukraine, majority of wastewater treatment plants were built on the basis of normative documents developed approximately 40 years ago (SNiP 2.04.03-85 "Canalization. External networks and constructions"). These documents are based on the kinetics and efficiency of organic carbon removal (COD, BOD). Nevertheless, during modernization and designing of new treatment plants advanced nitrogen compounds removal processes are being eventually introduced in Ukraine (Gmur, 2003; Henze et al., 2004; Nezdoyminov, 2013).

Currently, various methods of nitrogen compounds removal are known, among them physicochemical (release of ammonia, ionic exchange, chlorination by activated chlorine, reverse osmosis, electrochemical methods) and biological methods. The biological methods are based on microbiological processes with use of the activated sludge, and are considered as the most effective, and safe for the environment (Henze et al., 1999; Choubert et al., 2005; Yagov, 2008; Gujer, 2010; Holmes et al., 2019). Transformation of nitrogen compounds during the traditional biological treatment process includes following stages: microbiological assimilation, ammonification, and partial nitrification (Semenova et al., 2012; Holmes et al., 2019). During the advanced nitrogen removal, additionally advanced nitrification and denitrification are introduced (Henze et al., 2004; Semenova et al., 2012; Nezdoyminov, 2013). Microbiological nitrification is a chemoautotrophic process occurring in two phases. The ammonium oxidizing bacteria (AOB) carry out the first phase of nitrification in aerobic conditions, oxidizing ammonium up to the nitrite. In the second phase of nitrification – nitrite bacteria (NOB) oxidize nitrites to nitrates in aerobic conditions (Ward et al., 2011). To remove nitrites/nitrates from wastewater a denitrification process is applied (more precisely – of dissimilation denitrification) (Semenova et al., 2012). The dissimilation denitrification is a catabolic process of the heterotrophic microbiological reduction of nitrites/nitrates by protons of organic substrata under anaerobic conditions.

During biological wastewater treatment a change of aerobic and anaerobic conditions is carried out due to a consecutive passage of the sludge through anaerobic and aerobic zones of the activated sludge tanks (division of oxygen modes in space) or due to change of anaerobic and aerobic periods of processing in the same reactor of full mixture (division of oxygen modes in time). Reactors in which aerobic conditions (created by pneumatic aeration) and anaerobic conditions (created by stirring) are performed within a single reactor are called the sequencing batch reactors (SBR) (Choubert et al., 2005). Technological schemes of nitrification-denitrification processes (Henze et al., 1999; Gmur 2003; Henze et al., 2004) can be divided according to following parameters:

– number of sludge systems in the process (single sludge system or two-sludge system);

– number of aeration and stirring zones (one-stage, two-stage or circulating mode);

– type of organic substrate, used for denitrification (external organic substrate, for example methanol – "post-denitrification", or without external substrate – "pre-denitrification");

– operating mode (flow-through scheme or seqencing work scheme – aerobic and anaerobic conditions alternate in the same zone).

To implement such technologies, foreign and domestic experts have currently developed more than ten types of technological schemes, which are based on both physico-chemical and biological processes of wastewater treatment.

Now leading experts in the field of environmental protection associate the possibility of a radical improvement in the quality of water treatment from nitrogen compounds with the anammox process – anoxide oxidation of ammonium during the reduction of NO^{2-} to gaseous nitrogen by anammox-planktomycetam (Ding et al., 2013; Malovanyy et al., 2014; Wu et al., 2019;

Jiantao et al., 2020; Iurchenko and Tsytlishvili 2020). But this environmental and economic advantages method can be carried out in certain conditions of wastewater treatment (high concentration of NH_4^+, absence of organic compounds, low concentration of dissolved O_2).

According to modern concepts, nitrifying microorganisms include: ammonium oxide bacteria and archaea (AOB and AOA), which carry out 6-electron oxidation of NH_3 to NO_2^- (phase I nitrification), and nitrite oxide bacteria (NOB, phase II nitrification), which perform 2-electron oxidation NO_2^- to NO_3^-, as well as "complete NH_3 oxidizers" (comammox) bacteria, which carry out the 8-electron oxidation of NH_3 to NO_3^- (Ding et al., 2013; Lancaster et al., 2018; Wu et al., 2019).

The ecological risk of nitrogen presence is related with its form. Nitrites are considered as the most toxic, nitrates as the safest, while ammonium occupies the average position in such classification (Henze et al., 2004; EPA, 2009). Required treatment efficiency is based on standards for treated effluents discharged into water bodies. Different values of admissible nitrogen concentrations are presented in Table 1. In the EU countries only the total concentration of nitrogen (or Kjeldahl nitrogen – the sum of N-NH_4 and N-organic) in wastewater is standardized, and in the eutrophication sensitive areas should not exceed 10 mg/L for wastewater from settlements up to 100 thousand person equivalents (PE), and 15 mg/L for wastewater from settlements above 100 thousand PE. In Ukraine only soluble mineral nitrogen forms are under scrutiny. The admissible concentration of mineral soluble nitrogen is 9.55 mg/L for fish farming reservoirs and 13.16 mg/L for reservoirs, used for drinking supply. Concentrations of organic nitrogen are not supervised, and the absence of this control parameter extremely complicates the rational organization of wastewater treatment process, and also diminishes efficiency of the eutrophication protection measures.

Concentrations of nitrogen compounds in wastewater are variable and changeable at various stages of treatment process. Transformation of nitrogen compounds begins already during transport of wastewater to the treatment facilities. In particular, carbamide disintegrates with formation of ammonium (ammonification process) as a result of microbial destruction, and ammonium concentration in the wastewater entering treatment plants can range from 20 up to 50 mg/L. Concentrations of nitrates and nitrites in transported wastewater decrease due to the spontaneous denitrification - microbiological restoration of these compounds to gaseous compounds of nitrogen (mainly – N_2). Concentrations of nitrate-ions at the entrance to the treatment plant is insignificant, and also content of nitrite-ions is low (usually less than 1 mg/L) (Henze et al.; Gmur, 2003; Yagov, 2008).

Content of organic nitrogen compounds (high-molecular-proteins, proteids, low-molecular-amino acids, amines, amides, carbamide) in wastewater discharged into the sewer system can achieve 50−70 percent of the total quantity of nitrogen compounds. But as a result of ammonification processes during wastewater transport a share of the organic nitrogen decreases down to 15−35% at the entrance to the treatment plant (Henze et al., 2004; Yagov, 2008).

The most radical transformation of all nitrogen forms occurs during the biological treatment process. At the beginning, ammonium and organic nitrogen are removed with organic compounds (BOD, COD) during assimilation processes of the activated sludge. The next major stage of the biological treatment is nitrification, i.e. the microbiological oxidation of ammonium nitrogen into nitrite- and nitrate-ions. At the advance treatment in anaerobic (anoxic) conditions nitrite- and nitrate-ions are reduced to gaseous nitrogen by microbiological way (denitrification) (Henze et al., 2004; Yagov, 2008; EPA, 2009).

The purpose of this work is to present composition of nitrogen pollution in wastewater delivered into the Kharkov biological treatment plant, to determine the nitrifying capacity of activated sludge in aeration tanks and to demonstrate prospects of the treatment efficiency increase through an advanced nitrogen removal.

2. Materials and Methods

The Kharkov city is a big industrial center of Ukraine. There are two wastewater treatment plants in the Kharkiv city with the total capacity of 1.1 million m^3 per day. The sequence of treatment processes at these facilities includes mechanical and biological processes, disinfection and sludge processing. The following study presents results for one of the treatment plants (Kharkiv biological WWTP, with capacity of 300 000 m^3) where municipal and industrial wastewaters are delivered.

Investigations of the process of wastewater nitrification in biological treatment plants were focused on: i) determination of nitrogen compounds concentrations (ammonium nitrogen, nitrites and nitrates) in the dynamics of processing in the aeration tank; ii) determination nitrifying capacity of the activated sludge of these treatment facilities.

Hydrochemical analysis of aqueous media ($N\text{-}NH_4$ – colorimetric with Nessler’s reagent, $N\text{-}NO_2$ – colorimetric with α-naphthylamine, $N\text{-}NO_3$ – colorimetric with sodium salicylate, organic N ($N_{org.}$) – after wet mineralization titrometrically, pH – electrometrically, COD – arbitration method adding potassium dichromate) was conducted via standard methods according to Ukraine standards demands (List, 2013). The oxygen concentration in aqueous media was established using a portable oxygen meter (model YSI 55, Dissolved Oxygen Meter (USA)).

The nitrifying capacity of activated sludge of treatment facilities was determined using the biochemical method (Iurchenko, 2007) on the basis of the activity of the enzyme, catalyzing the reaction of hemolitoautotrophic oxidation of ammonium – hydroxylamine oxidoreductase (Lancaster et al., 2018).

In laboratory inhibitory experiments with activated sludge, to suppress nitrification and identify the process for incubation, we used wastewater models (similar in salt composition to urban wastewater, but without organic compounds) and pyrazole, an inhibitor of the first phase of nitrification, at a concentration of 10 mg/L.

The concentration of nitrifying bacteria of the first nitrification phase (AOB) in activated sludge was determined using the microbiological method of limiting dilutions (Vinogradova et al., 2012). Statistical processing of data was conducted via computer program Microsoft Excel.

3. Results and discussion

3.1. Nitrogen compounds in the Kharkiv wastewater

To study an influence of industrial wastewater with different contents of nitrogen compounds we focused on effluents from machine-building (heavy agricultural machinery) and milk processing plants. These factories discharge the biggest volume of industrial wastewater to the studied treatment plant. Results show (Table 1) that organic and ammonium nitrogen forms from the machine-building plant are present at small concentrations and not exceed effluent discharge standards for wastewater facilities in the Kharkiv area (the max possible admissible concentration for submission in a sewer network in Kharkiv is 18 mg/L). Concentrations of ammonium nitrogen from the milk processing plant were higher than values for wastewater from the machine-building plant approximately 5 times, and for organic nitrogen 10 times. Since, during the biological treatment process most of organic nitrogen mineralizes up to ammonium nitrogen, resulting load of the latter one at the treatment facility exceeds admissible level. Total nitrogen concentrations (organic nitrogen + ammonium nitrogen) are at the level of 11.3 up to 21.8, that exceeds the admissible level (18 mg/L). Concentrations of nitrogen oxidized forms – nitrites and nitrates – in both types of industrial wastewater are low. These data correspond with the characteristics of the content of nitrogen compounds in industrial wastewater obtained by other researchers. (Gmur, 2003; Malovanyy et al., 2014; Gnida et al., 2016).

In the municipal wastewater entering the biological treatment plant (Table 2), organic nitrogen makes approximately 50 percent of the total nitrogen value, which confirms the data of others authors (Gmur, 2003; Henze 2004; Yagov 2008; Jiantao et al., 2020). In the treated effluent its share decreases down to 36%.

Table 1. Concentration of various forms of nitrogen in the industrial wastewater

Industrial wastewater	**Concentration N (mg/L)**				
	organic	**NH_4^+**	**NO_2^-**	**NO_3^-**	**total**
Machine-building plants	9.0–12.0	2.3–9.8	0–0.14	0–0.5	13.0–22.3
Milk plants	56.0–121.0	10.6–22.0	0–0.28	0–0.4	66.4–141.1
Standards for wastewater entering WWTPs	No standard	18.0			No standard

Table 2. Concentration of nitrogen forms in the raw and treated wastewater at the Kharkiv WWTP (monthly average values)

Wastewater	**Concentration N (mg/L)**				
	organic	**NH_4^+**	**NO_2^-**	**NO_3^-**	**total**
Raw wastewater	16.8	16.2	0.3	0.4	33.7
Treated wastewater	7.0	1.8	1.0	9.5	19.3
Standards for treated wastewater	No standard	1.8	0.76	7.9	No standard

3.2. Indicators of municipal wastewater treatment from nitrogen compounds in aerotanks

Wastewater treatment at Kharkiv municipal WWTP No. 2 is carried out according to the traditional scheme: grates, sand traps, primary sedimentation tank, aeration tank, secondary sedimentation tank, disinfaction.

Averaged annual data of regular control of N-NH_4, N-NO_2 and N-NO_3 concentrations in incoming and discharging wastewaters from the wastewater treatment plant №2 into the river Udy are presented in Fig. 1.

As it is shown, N-NH_4 concentration after the treatment essentially decreases (up to 92.5%). In that time nitrite and nitrate concentrations increasing which are an obvious characteristic, firstly, of the deep wastewaters treatment and, secondly, of passing the full nitrification process (first and second phases).

Biological wastewater treatment takes place in four corridor aeration tanks with concentrated supply of activated sludge and dispersed supply of wastewater to the first half of the 2nd corridor (Fig. 2).

As presented in Fig. 3, concentrations of nitrates start to increase at the 2nd corridor of the aeration tank, and reach maximum values at the exit from the aeration tank (the end of 4th corridor). The concentration of nitrate nitrogen at the outlet of the aeration tank exceeds the permissible level (Table 2). That proves high activity of the nitrification process where not only N-NH_4, present in wastewater, is oxidized actively, but also this form of nitrogen is formed as a result of the mineralization of N_{org}. The residual concentration of N_{org} remains high at the exit from the aeration tank in comparison with specifications of EU for wastewater discharged in natural reservoirs. This fact can be explained by a partial resistance of nitrogen incorporated into organic compounds to the biological oxidation (Henze et al., 1999), and a partial mineralization of activated sludge.

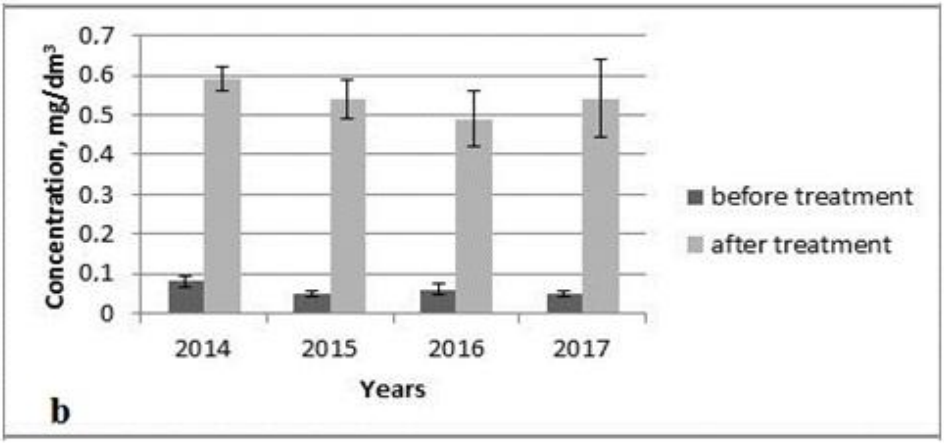

Fig. 1a. Average annual concentration dynamics of ammonium nitrogen (a) in wastewater before and after treatment (Iurchenko et al., 2020)

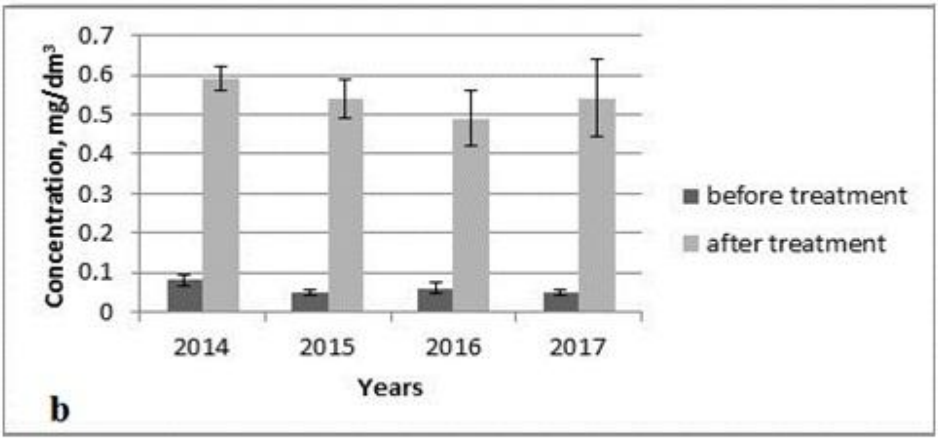

Fig. 1b. Average annual concentration dynamics of nitrite nitrogen (b) in wastewater before and after treatment (Iurchenko et al., 2020)

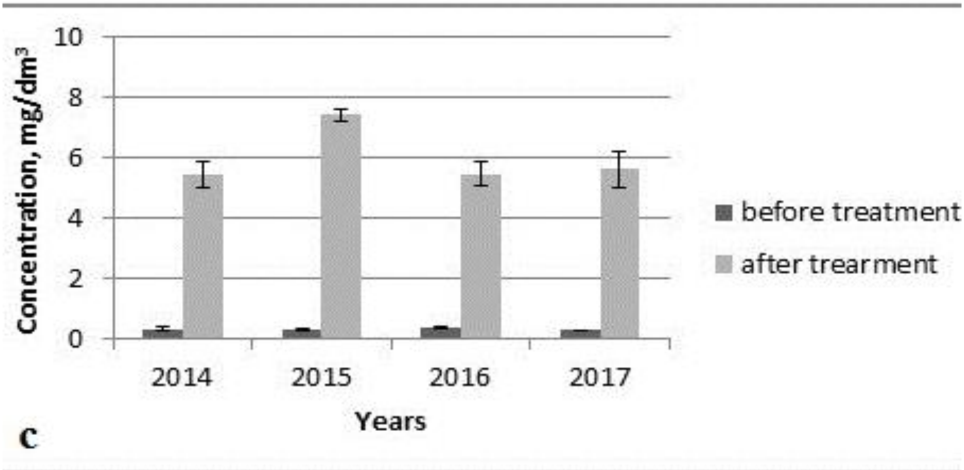

Fig. 1c. Average annual concentration dynamics of nitrate nitrogen (c) in wastewater before and after treatment (Iurchenko et al., 2020)

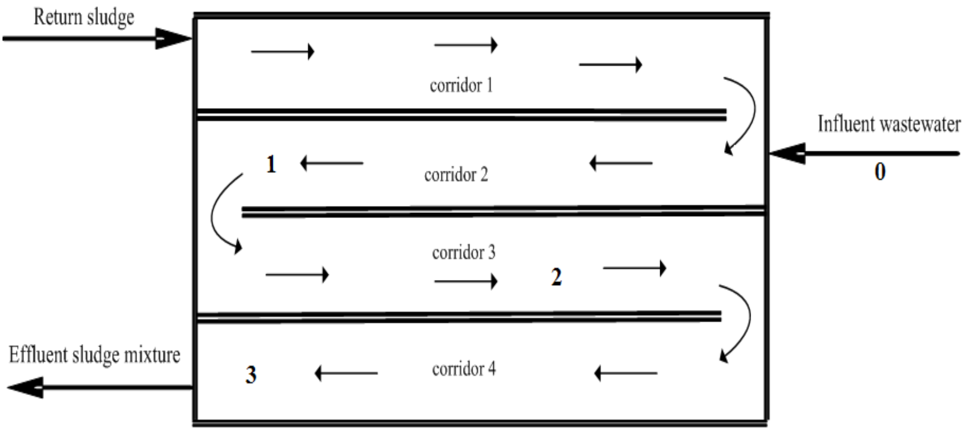

Fig. 2. Scheme of the flows in aeration tank at the Kharkiv WWTP and sampling points during research (sampling points 0 – etntering sewage, 1 – 2-nd corridor, 2 – 3-rd corridor, 3 – the end of 4th corridor)

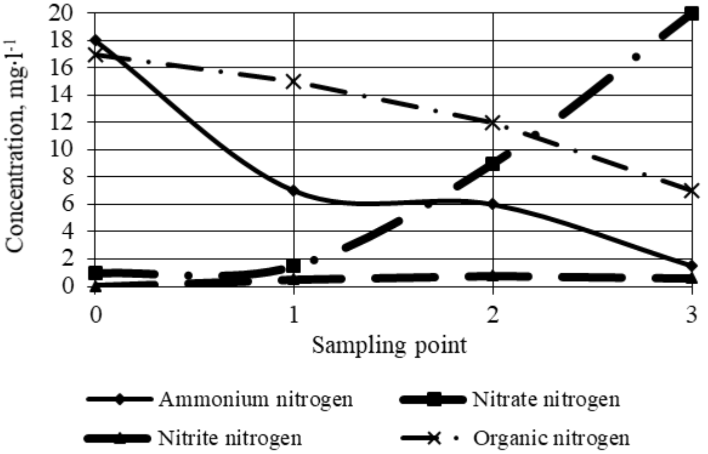

Fig. 3. Scheme of the flows in aeration tank at the Kharkiv WWTP and sampling points during research (sampling points 0 – etntering sewage, 1 – 2-nd corridor, 2 – 3-rd corridor, 3 – the end of 4th corridor)

Data of microbiological nitrification bacteria concentration determination in activated sludge of biological wastewater treatment plant and its nitrifying activity are presented in Table 3.

Table 3. Microbiological characteristics of the nitrifying ability of activated sludge from the municipal wastewater treatment plant No. 2

Sludge samples	**COD, mg/l**	**O_2, mg/l**	**Concentration of AOB, cells/$g_{org.\ sludge}$**	**Activity of hydroxylamine oxidoreductase, μg formazan/ ($g_{dry\ sladge}$·min)**
Beginning of aeration tank (point 1)	110–160	1.2–1.6	10^6–10^7	99.3–194.0
Ending of aeration tank point 3)	30–52	2.2–2.4	10^6–10^8	105.5–321.0

As the presented data show, AOB concentration in activated sludge from wastewater treatment plant in the ending of aeration tank-displacer increases as a result of passing nitrification in this zone and reaches 10^6–10^8 cells/$g_{org.\ sludge}$. Similar concentrations of AOB were found in activated sludge of municipal WWTP and other specialists (Fitzgerald et al., 2015). An increase in the nitrifying capacity of sludge was also evidenced by an increase in its hydroxylamine oxidoreductase activity. The concentration of nitrifying bacteria in the activated sludge and its enzymatic nitrifying activity were high enough for municipal wastewater treatment plants (Iurchenko, 2007).

Oxidation of ammonium nitrogen in the anammox process (the presence of anammox bacteria in activated sludge) can be established in laboratory experiments when suppressing nitrification using an inhibitor (pyrazole) as the difference between the mass of oxidized $N\text{-}NH_4$ in the variant "sludge with an inhibitor of autotrophic nitrification" and "sludge killed by heat denaturation". In the conducted sludge studies, this difference was practically zero. Consequently, the anammox process in the surveyed structures did not develop, which was facilitated by the absolute concentrations of $N\text{-}NH_4$ (low to optimize this process), rather high concentrations of COD and dissolved oxygen. Similar results were obtained (Shanyun et al., 2015), which showed that the contribution of anammox bacteria to removed nitrogen during traditional biological treatment of municipal wastewater is 2.1–6.9%.

Calculation of balance for oxidized and reduced forms of nitrogen in the entrance and exit from the biological reactor testifies, that in the investigated biological reactor the denitrification process of nitrates and nitrites practically does not occur. Concentration of nitrates in the treated wastewater exceeds limit values (7.9 mg/l). This threatens with excessive removal of sludge from secondary sedimentation tanks during spontaneous denitrification. It is necessary to organize denitrification in biological treatment facilities before the settling stage (Gmur, 2003). For denitrification of superfluous nitrates nitrogen the organic substrate the ratio $BOD:N\text{-}NO_3 \sim 4$ is necessary (Gmur, 2003; Henze, 2004). Since, at the entrance to the biological reactor concentration of BOD_5 in wastewater exceeds 110 mg/l denitrification more than 28 mg/l $N\text{-}NO_3$ is possible through partial recycling of treated wastewater at the entrance to the biological reactor.

Optimization of the denitrification process at the Kharkiv WWTP is considered as the most perspective way to achieve the European specifications on total nitrogen concentrations in the treated wastewater. Modernization process of the investigated biological treatment plant is focused on the increase of the nitrogen and phosphorus removal efficiency. This requires taking into account detailed information on organic nitrogen concentrations entering the treatment facility and its transformations during processing.

4. Summary and Conclusions

The current study based on the Kharkiv WWTP allowed us to draw following conclusions:

– N_{org} concentration in wastewater entering the investigated treatment plant makes about 49.9 percent of the total N; the highest concentration of Norg occurs in the milk processing plant effluents;

– the total load of ammonium nitrogen and organic nitrogen (which in the process of biological wastewater treatment turns into ammonium nitrogen during the mineralization of organic compounds) on the treatment plants was extremely higher than the permitted level. Treatment processes in the aeration tank with activated sludge include deep nitrification, while anammox process and the denitrification process do not occur at all;

– the concentration of nitrifying bacteria in the activated sludge and its enzymatic nitrifying activity were high enough for municipal wastewater treatment plants;

– the most perspective way to increase efficiency of the nitrogen compounds removal is to increase time of the aerobic treatment to allow advanced oxidation of organic compounds and to improve the nitrification process (decrease of residual ammonium nitrogen concentration) and organizing a denitrification process for deep removal of nitrites and nitrates;

– modernization of the investigated facility shall also point out the necessity of improving of standardization for nitrogen compound concentrations in the Ukraine wastewaters.

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