



## **STATISTICAL PROCESS CONTROL OF REMOVAL OF NITROGEN COMPOUNDS IN THE WASTEWATER TREATMENT PLANT IN KROSNO**

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### *Abstract*

The aim of the paper was to determine the possibility of the use of Shewhart control charts to monitor changes in the forms of nitrogen, showing the quality of wastewater discharged from the wastewater treatment plant in Krosno in the years 2010-2015. The performed statistical analysis showed the highest number of cases of elevated nitrate nitrogen and/or ammonium nitrogen when the temperature of treated wastewater was below 8-9°C. This low temperature resulted in adverse effect on the activity of bacteria that were involved in biological removal of nitrogen. It was found that in the winter months, the second stage of nitrifying bacteria responsible for oxidation of nitrite to nitrate, exhibited higher activity than the denitrifying bacteria. Graphical presentation of total nitrogen content using control charts for the mean of the process revealed the exceedance of the upper specification line ( $USL = 10 \text{ mgN}_{\text{tot}} \cdot \text{dm}^{-3}$ ) in the months from December to April. It was observed that the total nitrogen removal process in the months from June to November was stable with a very high 90% reduction in biogenic activity. The obtained results confirm the efficacy of control charts as a tool which can easily be applied in the statistical process control of total nitrogen removal in municipal wastewater treatment plants.

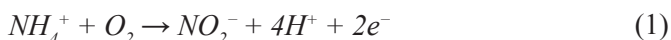
**Keywords:** control chart, biogenic compounds, wastewater treatment plant

## INTRODUCTION

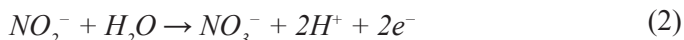
Council Directive 91/271/EEC of May 21, 1991 concerning urban wastewater treatment defines the requirements for effective removal of biogenic compounds for all wastewater treatment plants in sensitive areas subjected to eutrophication (Directive ... 1991). 99.7% of the area of Poland is situated in the catchment area of the Baltic Sea, therefore, the area is considered “sensitive” that requires limiting nitrogen and phosphorus compounds which are then discharged to surface waters. The deadline of December 31<sup>st</sup>, 2015 was set as a timeframe for a 75% reduction of total nitrogen and phosphorus compounds coming from municipal sources and being discharged into water. As required by the Directive, total nitrogen concentration in municipal effluent discharged from treatment plants to sensitive areas subject to eutrophication may not exceed 10 mg·dm<sup>-3</sup> in the case of treatment plants with the population equivalent (PE) of above 100 000. It is allowed to increase this value up to 20 mg·dm<sup>-3</sup> only for daily mean tests when the wastewater temperature while utilising a biological chamber of a treatment plant is  $\geq 12^{\circ}\text{C}$  (Annex ... 1991). The applicable Polish legislation specifies the permissible concentration of total nitrogen (10 mg·dm<sup>-3</sup>) or its minimum percentage reduction for household wastewater or for municipal wastewater discharged into water or soil (70 ÷ 80%). These figures refer to the mean annual value of this indicator taken from the daily mean samples collected in the analysed year (Regulation ... 2014).

Total nitrogen is the sum of organic nitrogen and inorganic nitrogen forms (ammonium nitrogen N-NH<sub>4</sub><sup>+</sup>, nitrate nitrogen N-NO<sub>3</sub><sup>-</sup> and nitrite nitrogen N-NO<sub>2</sub><sup>-</sup>). Biological nitrification is the most popular process of the removal of ammonium nitrogen from municipal wastewater. However, it does not, in the strict sense, result in nitrogen removal, but merely changes its form. Nitrification proceeds in two stages: through the oxidation reaction of ammonium NH<sub>4</sub><sup>+</sup> ion form to nitrite NO<sub>2</sub><sup>-</sup> (nitritation) followed by NO<sub>3</sub><sup>-</sup> nitrates (nitratation) (Sadecka 2010).

In the first stage of nitrification, dominant bacteria of *Nitrosomonas* kind oxidize NH<sub>4</sub><sup>+</sup> ions to NO<sub>2</sub><sup>-</sup> ions in accordance with the reaction:



The second stage of nitrification is mainly carried out by *Nitrobacter* and *Nitrospira* bacteria and involves oxidation of the established nitrite ions to NO<sub>3</sub><sup>-</sup> nitrate ions:



Because negligible amounts of nitrites and nitrates are the products of proper nitrification, the process should be combined with the removal process of the NO<sub>3</sub><sup>-</sup> ions. NO<sub>3</sub><sup>-</sup> can be reduced to NH<sub>4</sub><sup>+</sup> and then incorporated into the

biomass of the bacterial cell (*assimilation*) or they can be reduced to gaseous nitrogen forms such as nitrogen oxides and particulate nitrogen through the dissimilatory reduction of nitrates (denitrification) (Sadecka 2010).

The rate of growth of nitrifying bacteria depends mainly on the available process substrates (concentration of ammonia and dissolved oxygen) and on the temperature of the treated wastewater. It is commonly known that nitrifying bacteria exhibit tolerance in reasonably wide (10°C to 35°C) temperature ranges. The optimal range for *Nitrosomonas* development is in the range of 25-35°C, while temperatures below 9-10°C stunt the growth of the bacteria. Zhu *et al.* (2008) report that at temperatures below 15°C, nitrite oxidizing bacteria predominate over ammonium-oxidizing bacteria. The nitrification process is completely inhibited at a temperature of about 5°C mainly due to lack of *Nitrosomonas* growth (Bartoszewski *et al.*, 2011, Hao *et al.*, 2002). The optimum temperature for the development of denitrifying bacteria stands at 20°C whereas its decrease below 5-8°C greatly affects the growth of these microorganisms (Bartoszewski *et al.* 2011).

Due to the prospect of improving the reliability of treatment plant, many of them have a system of regular measurements covering not only the quantity but also the quality of treated wastewater. Supervision of the removal of contaminants, including biogenic compounds, in treatment plants serving agglomerations above 100 000 the population equivalent (PE), is often carried out using special SCADA control systems (MPGK Krosno 2017, SPGK Sanok 2013). Not only do these systems allow for visualization of technological processes and analogue measurements but also they have the ability to signal exceedance, operating conditions and equipment failures. Being compatible with Microsoft products, they can generate reports and summaries of variables stored in a database. Such a large set of numbers can, however, be challenging for statistical analysis. Statistical process control (SPC) can be used to denote process interferences that may subsequently lead to its deregulation and in effect deterioration of the quality. This is most often analysed by Shewhart's control charts which are appropriately prepared graphical procedures (Shewhart 1981). The design of such control charts is not a complicated task but should be preceded with the determination of the frequency of observation and the number of variables to be analysed. In the case when numerical data are gathered in subgroups of the same number, the most common average value ( $\bar{x}_{av}$ ) chart for monitoring variables is used. Apart from marking the mean value of the observed variable in the form of so called central line (CL) on the graph, the typical chart contains two control lines: lower (LCL) and upper (UCL) (Statsoft 2017). Control limits are determined on the basis of variability within the subgroup. Single points corresponding to the mean values calculated from the variable are joined with lines. If such a line crosses the upper or lower control line or a nonlinear array of points depicting characteristics of variables is detected, deregulation of the process is observed (Statsoft

2017). Microsoft Office Excel and its add-ons can be an appropriate tool for analysing and visualizing data in the form of control charts.

The authors of the study made an attempt to assess the use of control charts for the analysis and monitoring of changes in nitrogen forms illustrating the quality of wastewater discharged from the wastewater treatment plant in Krosno in the years 2010-2015.

## **DESCRIPTION OF THE TESTED OBJECT**

The mechanical-biological wastewater treatment plant serving the agglomeration of Krosno with ENI exceeding 107 000 (Regional Inspectorate for Environmental Protection Rzeszów 2015) is located on the left bank of the Wisłok river in the northwestern part of the city. This wastewater treatment plant serves the city of Krosno and neighbouring boroughs such as Chorkówka, Iwonicz, Jedlicze, Korczyzna, Krościenko Wyżne, Miejsce Piastowe, and Wojaszówka.

The permissible amount of treated municipal wastewater in the rain-free period can roughly add up to average  $35\,410\text{ m}^3\cdot\text{d}^{-1}$  (Permit ... 2012). In addition, throughout a year household wastewater is delivered in the amount of about  $2\,000\text{ m}^3$ . The amount of industrial wastewater accounts for about 24% of the total wastewater. It is discharged from industrial plants such as Delphi Krosno JSC., GOODRICH KROSNO Ltd., KROS-GLASS, Krosno Glass Works „KROSNO” JSC., WSK PZL-KROSNO JSC., NAFTOMET Ltd. To a large extent, the sewage comes from lines which prepare metal parts for painting, from the chroming process as well as from a electroplating plant.

The technological process is based on the activated sludge method with chemically assisted phosphorus precipitation. Each of the two treatment lines consists of one pre-denitrification chamber, two dephosphatation chambers, two denitrification chambers and two nitrification chambers. Additionally, a station of chemical precipitation of phosphorus using PIX-113 was installed in the facility (Municipal Enterprise of Municipal Services, MPGK, Krosno 2017). The treated wastewater is discharged in a covered collector to the Wisłok River at a distance of  $135 + 050\text{ km}$  (Municipal Enterprise of Municipal Services, MPGK, Krosno 2017).

## **METHODOLOGY OF RESEARCH**

The values of physicochemical indicators made available by the Central Water and Wastewater Laboratory of Municipal Enterprise of Municipal Services in Krosno Ltd (Centralne Laboratorium Wody i Ścieków Miejskiego Przedsiębiorstwa Gospodarki Komunalnej w Krośnie Sp. z o.o.) were the source materials for the analysis. Samples of treated wastewater were collected twice

a month (Permit ... 2012). The study covered a six-year period from January 1, 2010 to December 31, 2015. The analysed dataset consisted of 72 records identified by five variables, illustrating changes in nitrogen forms in treated effluents. Basic descriptive statistics that is arithmetic mean, median, minimum, maximum, spacing, standard deviation ( $\sigma$ ) and coefficient of variation were defined for the variables. In addition, for the 6-year study period, the percentage of individual forms of nitrogen in the treated wastewater was calculated.

Due to the fact that the removal of nitrogen compounds depends to a large extent on the temperature of the treated wastewater, the degree of correlation was determined.

The procedure of preparing a control chart for the mean value of the process  $x_{av}$  was started from the central line, which was the arithmetic mean of the results of the total nitrogen measurements in the period of January to December ( $n = 12$ ). The upper and lower control limits were calculated applying the following formulas:

$$UCL = x_{av} + 3 \cdot (\sigma \cdot n^{-1/2}) \quad (3)$$

$$LCL = x_{av} - 3 \cdot (\sigma \cdot n^{-1/2}) \quad (4)$$

and then applied on the chart in the form of horizontal lines. The individual values for the mean concentration/mean reduction of total nitrogen for the following months were indicated as points and connected with a continuous line. In the case of analysis of the chart concerning changes in total nitrogen concentration, upper specification limit (USL) equal to the permissible  $N_{tot}$  value in outflow (USL=10 mg·dm<sup>-3</sup>) was additionally introduced. In the chart connected to nitrogen removal, however, the upper specification limit being the equivalent of the minimum permissible percentage of its reduction (USL= 70 ÷ 80%) was defined.

The analysis of the chart consisted of the visual inspection of the way in which the points were laid and of determining if their *scattering* was within the defined limits, if they were between the control lines (a stable process) or if the points were outside the specifications limits (Standard ... 1996).

The statistical and SPC analyses were conducted with the use of Excel 2010.

## RESULTS AND DISCUSSION

The results of the statistical analysis of the source data in the period of 2010 to 2015 are presented in Table 1 respectively.

**Table 1.** Characteristics of raw and treated sewage in the light of the content of nitrogen compounds

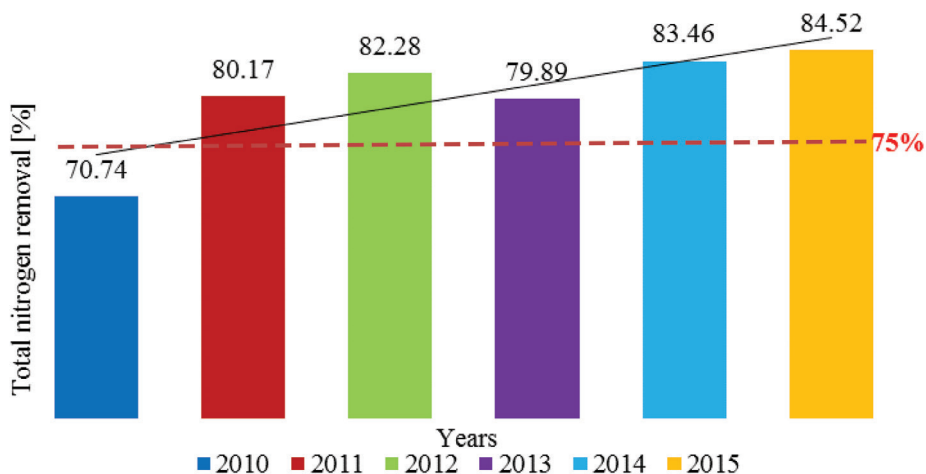
| Parameter         | Unit                | Descriptive statistics |               |               |                    |                          |
|-------------------|---------------------|------------------------|---------------|---------------|--------------------|--------------------------|
|                   |                     | Mean value             | Minimum value | Maximum value | Standard deviation | Coefficient of variation |
| Raw sewage        |                     |                        |               |               |                    |                          |
| Temperature       | °C                  | 12.9                   | 6.4           | 19.9          | 3.2                | 0.25                     |
| Ammonium nitrogen | mg·dm <sup>-3</sup> | 45.1                   | 4.2           | 117.0         | 14.8               | 0.33                     |
| Nitrite nitrogen  | mg·dm <sup>-3</sup> | 0.18                   | 0.00          | 0.87          | 0.20               | 1.10                     |
| Nitrate nitrogen  | mg·dm <sup>-3</sup> | 0.59                   | 0.05          | 64.28         | 3.71               | 6.25                     |
| Organic nitrogen  | mg·dm <sup>-3</sup> | 24.33                  | 4.06          | 63.10         | 11.34              | 0.47                     |
| Total nitrogen    | mg·dm <sup>-3</sup> | 69.76                  | 0.00          | 117.00        | 20.39              | 0.29                     |
| Treated sewage    |                     |                        |               |               |                    |                          |
| Temperature       | °C                  | 13.2                   | 3.8           | 20.7          | 4.0                | 0.30                     |
| Ammonium nitrogen | mg·dm <sup>-3</sup> | 2.09                   | 0.03          | 23.16         | 3.86               | 1.85                     |
| Nitrite nitrogen  | mg·dm <sup>-3</sup> | 0.17                   | 0.00          | 1.24          | 0.15               | 0.91                     |
| Nitrate nitrogen  | mg·dm <sup>-3</sup> | 7.67                   | 2.40          | 19.05         | 3.40               | 0.44                     |
| Organic nitrogen  | mg·dm <sup>-3</sup> | 2.66                   | 0.19          | 5.22          | 0.77               | 0.29                     |
| Total nitrogen    | mg·dm <sup>-3</sup> | 12.40                  | 6.03          | 32.50         | 5.22               | 0.42                     |

Source: own elaboration

In wastewater entering the sewage treatment plant in Krosno during the six analysed years, the total nitrogen content ranged from 4.2 to 117.0 mg·dm<sup>-3</sup> reaching the mean of 69.76 mg·dm<sup>-3</sup>. Ammonium nitrogen accounted for, on average, 65% of the total nitrogen, and the remainder was organic nitrogen.

The mean total nitrogen concentration in wastewater discharged from the Krosno wastewater treatment plant in the years 2010-2015 amounted to 12.4 mg·dm<sup>-3</sup>. The total nitrogen removal rate for the given period fell into the brackets of 37.2-92.6%, and the average efficiency of the 6-year period reached 80.1 ± 11.0%. The coefficient of variation was at an average level and roughly added up to 0.14. Total nitrogen concentrations were similar to those reported by other authors (Chmielowski and Ślizowski 2009, Chmielowski and Ślizowski 2010, Ślizowski and Chmielowski 2008).

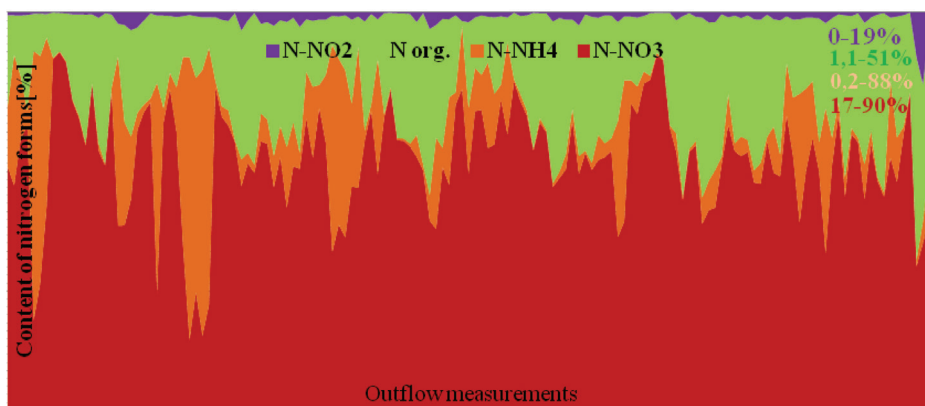
Figure 1 depicts changes in percentage removal of total nitrogen over the 6-year research period. At this time, almost 14% increase in efficiency of removal of this index up to the value of 84.5% was observed. In the years 2011-2015 the Krosno wastewater treatment plant ensured the reduction of total nitrogen compounds required by Council Directive 91/271/EEC at a minimum of 75%.



Source: own elaboration

**Figure 1.** The percentage of total nitrogen removal from the wastewater treatment plant in Krosno in 2010-2015

Figure 2 illustrates the percent content of individual nitrogen forms in sequentially analysed 144 treated wastewater samples. Nitrate nitrogen  $N-NO_3^-$  had the biggest (several dozen percent) share whereas the unstable form of nitrogen that is nitrite nitrogen  $N-NO_2^-$  had the lowest content.



Source: own elaboration

**Figure 2.** The percentage of nitrogen compounds in 144 samples of treated wastewater from the wastewater treatment plant in Krosno in 2010-2015



It can therefore be concluded that the second stage of the nitrification process that is the nitrite oxidation to nitrate proceeded with high efficiency. In winter, when the drainage temperature was below 12°C, higher ammonium nitrogen values were observed in the outflow because the NO<sub>2</sub> – oxidizing bacteria were dominant over the NH<sub>4</sub><sup>+</sup> ones (Yao and Peng 2017).

The total ammonium and nitrate nitrogen concentrations for the treated wastewater temperature <9.5°C in 11 cases exceeded the permissible value of 20 mg·dm<sup>-3</sup> for N<sub>tot</sub>, as proposed in the water and legal permit (Permit ... 2012). This proves the inhibition of not only the nitrification process but also the denitrification process. In the analysed years 34 cases of exceedance of the concentration at a level of 10 mg·dm<sup>-3</sup> were observed for N-NO<sub>3</sub> – when the temperature of the treated wastewater dropped below 9.4°C. 11 of such cases related to N-NH<sub>4</sub><sup>+</sup> were noted when the temperature was even lower and was below 8°C.

There were 37 cases of exceedance of the 75% reduction of total nitrogen in the 6-year study period. In all cases, this occurred when the outflow temperature from the treatment plant dropped below 10°C in the months of December to mid-April. The mean monthly temperatures for Krosno in these five months come to 3.1°C (November), -2.2°C (December), -5.4°C (January), -3.9°C (February) and 1.8°C (March) respectively (<https://pl.climate-data.org>). While carrying out research for a municipal sewage treatment plant in Košice, Kaczor (2008) observed the drop, below 12°C, in wastewater temperature in the open activated sludge chamber, which lasted from November to April. This means that unfavourable climatic conditions may take place in the treatment plant for almost half a year curbing the activity of microorganisms responsible for the process of nitrogen removal from sewage. This is confirmed by correlations between individual nitrogen forms present in outflow and the temperature calculated for the wastewater treatment plant in Krosno.

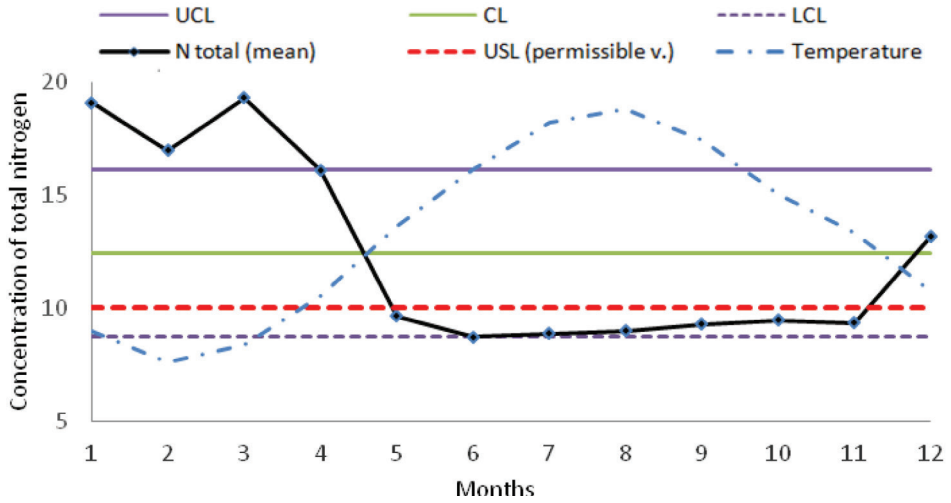
According to the Stanisiz (1998) scale, the negative correlation between total nitrogen and temperature appeared to be at a very high level that is at 70%. High correlations for nitrate nitrogen (-0.60) and ammonium nitrogen (-0.52) were also found. In the study on the effect of temperature on total nitrogen removal in the municipal sewage treatment plant in Książ Wielki, Bugajski (2011) also revealed a high correlation, 65%.

The last step of the statistical control of outflow from the wastewater treatment plant in Krosno was to draw up control charts for monthly mean values of total nitrogen concentration and the efficiency of its removal (Figures 3a and 3b). The “a” chart clearly shows that in the winter months, when the mean temperature of outflow was <10.5°C (January, February, March), the upper control line UCL was exceeded. Including the month of December, 4 exceedances of USL=10 mg·dm<sup>-3</sup> were noted. The total nitrogen removal process in this period should therefore be considered as unstable. 6 exceedances of control limits, including the exceedance of the lower control line LCL which lasted from January

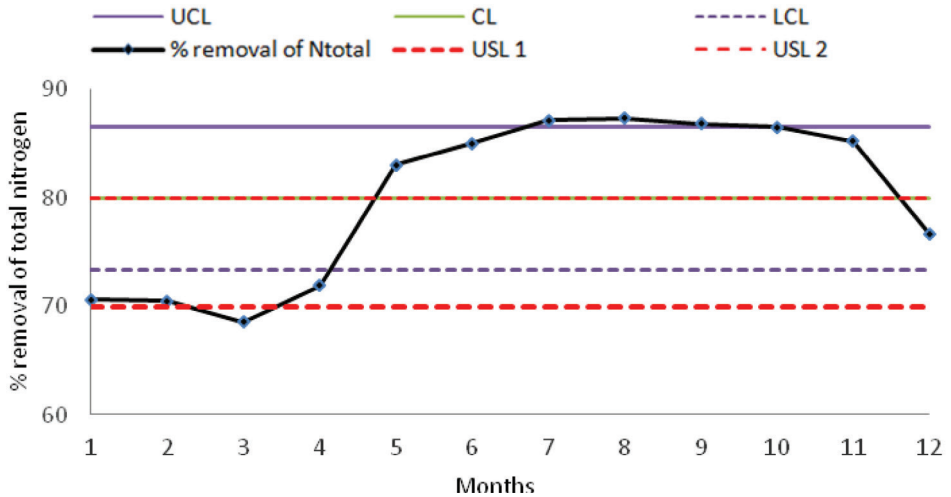


to April, were observed for the “b” chart. Comparing the two charts, it can be stated that during the period in question March was the month when it was evident that the nitrogen removal process was deregulated. Then the permissible value of  $USL1=70\%$  was also exceeded (Figure 3b).

a)



b)



Source: own elaboration

**Figure 3.** Control chart for monthly mean values: a) total nitrogen concentration, b) % removal of total nitrogen in wastewater discharged from the Krosno wastewater treatment plant in 2010-2015

It can be estimated, applying Shewhart control charts, that the overall nitrogen removal process in the months of June to November 2010-2015 was very efficient, reaching a 90% reduction in this biogenic compound.

## SUMMARY AND CONCLUSIONS

On the basis of the conducted analysis, it was found as follows:

1. During the 2010, 2011, 2012 and 2013 research years, the total number of nitrogen exceedances was 9, 8, 3 and 6 respectively in relation to the normative value specified in the applicable legislation (Regulation ... 2006, Regulation ... 2014). In 2014, 1 exceedance was noted, while in the year 2015 there was none. In total, in the years 2010-2015, the number of exceedances occurred in 19% of all analysed measurements.
2. Elevated nitrate and/or ammonium nitrogen values occurred when the temperature of the treated wastewater dropped below  $8 \div 9^{\circ}\text{C}$ . The low temperature resulted in an adverse effect on the activity of both nitrifying and denitrifying bacteria that were involved in the biological removal of nitrogen. During the considered research period, in 7.6% of the cases, the total ammonium and nitrate nitrogen concentrations exceeded the permissible  $\text{N}_{\text{tot}}$  value of  $20 \text{ mg} \cdot \text{dm}^{-3}$  for the treated wastewater  $<9.5^{\circ}\text{C}$ .
3. It is therefore essential to monitor not only total nitrogen but also its inorganic forms. The legislation of other EU members, for example Slovakia, defines the limit values for wastewater and special water pollution indicators also for ammonium nitrogen (Annex ... 2010).
4. In the winter months, nitrification bacteria of the second stage, responsible for the oxidation of nitrites to nitrates, exhibited higher activity than denitrifying bacteria. As suggested by Bartoszewski *et al.* (2011), the removal efficiency of nitrates in denitrification chambers can be improved by additionally adding methanol as a carbon source or by the increased nitrate recirculation.
5. The highest instability of the removal of total nitrogen was found with the increase of its concentration in the outflow. This was the case in March and was probably triggered by an inflow of thawing snow into the sewage system, which negatively affected both the nitrification and denitrification processes. It is therefore important during the thawing period to pay close attention to the correct operation of the facility.
6. Until 31 December 2015, the treatment plant in question fulfilled the requirement of 75% reduction of the total nitrogen compounds from municipal sources which were then discharged into the waters. It

therefore adapted to the requirements of the European Union law on effective removal of biogenic compounds.

7. The presented statistical process control techniques, which are based on, among others, Shewart's control charts, showed their usefulness in monitoring of the process of removing nitrogen compounds in urban sewage treatment plants.

*Research conducted under the framework of DS 3347/KISiGW/2017 were financed from grants for science granted by the Ministry of Science and Higher Education.*

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Received: 06.05.2017

Accepted: 29.11.2017