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Changes in quantity of non-biodegradable organic micropollutants in municipal wastewater based on COD fractionation

Ewelina Płuciennik-Koropczuk*, Sylwia Myszograj

University of Zielona Gora, Faculty of Civil Engineering, Architecture and Environmental Engineering; Institute of Environmental Engineering Szafrana St.15, 65-246 Zielona Góra, Poland, emails: E.Pluciennik@iis.uz.zgora.pl (E. Płuciennik-Koropczuk), S.Myszograj@iis.uz.zgora.pl (S. Myszograj)

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ABSTRACT

The article presents the possibility of using the chemical oxygen demand (COD) fractionation to the monitoring of non-biodegradable organic pollutants in municipal wastewater. Understanding the origin of pollutants contained in wastewater is important in the aspect of their treatment. The characteristics of municipal wastewater are mainly affected by the water supply and sewage infrastructure in a given area, as well as factors resulting from the way of life of residents. The presence of chemical compounds practically in all spheres of human life has meant that their quantity in municipal wastewater increased. The latest publications and reports on the qualitative characteristics of municipal wastewater indicate an increase in the scope of micro-contaminants identified in them, for example, priority substances and so-called emerging contaminants. To confirm the thesis that the quality of wastewater changes, as a consequence of the lifestyle of residents, 2 y research was carried out in a mechanical-biological wastewater treatment plant of 26,000 PE. Analysis of test results showed a reduction of biodegradable organic pollutants in raw wastewater from 82.8% ± 3.2% to 73.9% ± 4.9% and the reduction of the effectiveness of removing organic pollutants from wastewater, expressed in the COD index from 93% to 91%, while the efficiency determined for biochemical oxygen demand (BOD_5) remained unchanged. Analysis of COD fractions in treated wastewater showed a reduction in the share of non-biodegradable fractions. Differences in efficiency of removal of organic pollutants from wastewater expressed as BOD_5 and COD may indicate an increase in the amount of non-biodegradable micro-pollutants affecting the change in the quality characteristics of wastewater supplied to the wastewater treatment plant.

Keywords: Wastewater; Biodegradation; COD; BOD_5; COD fractions

1. Introduction

The latest reports and publications concerning the qualitative characteristics of the municipal wastewater show an increase in the number of identified substances primarily from the group of priority substances and so-called emerging contaminants.

From the group of priority substances for the aquatic environment listed in Directive 2013/39/EU of the European Parliament and of the Council of 12 August 2013 [1], among organic compounds hydrocarbons (e.g. anthracene, benzene, benzopyrene) and their derivatives are most commonly identified [2–4], while among inorganic substances heavy metals (e.g. cadmium, nickel, lead, mercury) and their compounds. The sources of these pollutants are not only industrial wastewater, but also domestic wastewater containing, among others cosmetics, detergents or pharmaceuticals. In the composition of both detergents and cosmetics are used generally available ingredients such as surfactants, natural extracts, and others. It moves away from simple, formerly used compositions in the direction of

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* Corresponding author.

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complex recipes. The results of the composition of domestic wastewater discharges from sanitary fittings and appliances (dishwasher and washing machine), showed significantly higher concentrations of pollutants in wastewater from the appliances. In addition in wastewater from the dishwasher and washing machines were identified also the largest quantity and the highest concentration of metals (As 145 μg/L, Al 0.27 mg/L, Cu 0.434 mg/L, Sr 0.071 mg/L, V 0.026 mg/L, Ba 0.0226 mg/L), which is the result of a small volume of aqueous wastewater and the associated concentration. While the wastewater from the washing machines is the largest single source of dissolved substances on average 82.2 g/wash [5,6]. In addition to an increase in turbidity, color intensity, phosphorus concentration, popular washing agents in the form of powders, liquids and capsules also increase the COD value, and the kinetics of biochemical oxygen demand (BOD_5) degradation indicates the presence of non-biodegradable compounds in washing agents [7].

In consumer products of everyday use such as cosmetics, clothing, household equipment nanomaterials and microplastics are commonly used. During the use and operation of products nano ingredients, nanoparticles and particles of microplastics can be released or washed out. It was shown that the content of released nanosilver in the wastewater from the washing machine was 11 μg/L [8].

The presence of nanoparticles in wastewater and sewage sludge has been confirmed in several studies. Due to the large antibacterial potential, a wide range of biocidal activity and ability to transmit to bacterial cells, silver nanoparticles can reduce the biodiversity of microorganisms and pose a threat to species used in biodegradation of pollutants, including microorganisms used in the processes of wastewater treatment [9–12].

Microplastic that is found in wastewater is mainly micro-particles of polyethylene and polypropylene in sizes from 450 to 800 μm derived from plastics, synthetic clothes and cosmetics (e.g. hair shampoos, toothpaste) [13,14]. In raw wastewater, the concentration of microplastics reached several thousand particles/m^3. A significant part of the microplastics is removed from the wastewater in the process of the initial sedimentation, which causes its accumulation in sewage sludge. In view of the low biodegradability, the microplastic together with stabilized sewage sludge goes to soil or with the sludge processed in another way along [15].

The current worldwide research shows the widespread occurrence of xenobiotics in the aquatic environment, among which specific group is Pharmaceutical and Personal Care Products (PPCPs) including pharmaceuticals and personal care products. The PPCPs constitute a major problem because in small quantities they pose a threat to the proper functioning of aquatic organisms. Therefore PPCPs are currently the subject of intensive research. An important source of these pollutants is single consumers using a wide range of over-the-counter medicines, the so-called over the counter (OTC) [16,17]. Pharmaceuticals unused and outstanding receivables are often directly into the sewage system [18]. The first studies on the presence of contaminants of pharmaceutical origin in the aquatic environment were conducted in the USA in the 1970s. At the end of the 20th century, rivers in Germany were also monitored, as well as treated wastewater and surface water and drinking water. Analgesics and anti-inflammatory, psychotropic, anti-epileptic medicines, beta-blockers, fat regulators and their metabolites, including antibiotics and hormones were detected. At the turn of the century, as a result of extensive research, it was also established that in surface waters there are most often OTC medicines, antibiotics, and hormones: oestrone, 17 β-oestradiol, oestriol and estrogens synthetic, inter alia etinyloestradiol [16,19–24].

Despite numerous literature reports indicating the presence of micro-pollutants in wastewater, which often reduce the susceptibility for biodegradation, there are no legal regulations regarding the maximum concentrations of these pollutants in wastewater, and the standard characteristics of wastewater is still based mainly on the values of indicators such as COD, BOD_5, total suspension, total phosphorus, and total nitrogen. Determination in detailed new pollutants and priority substances is expensive and time-consuming. Indirectly, the scale of the problem can be inferred, for example, on the basis of the share of biodegradable and non-biodegradable organic fractions in wastewater.

Acceptance of COD as the main parameter determining the amount of organic carbon in wastewater and the division of COD into fractions describing different degrees of their biodegradation is, for now, a significant extension of the characteristic of wastewater [25].

The total chemical oxygen demand (TCOD) of wastewater, divided into fractions, can be calculated in a simplified way according to Eq. (1) [26]:

$$\mathrm{TCOD} = S_S + S_I + X_S + X_I \qquad \mathrm{g\ O_2/m^3} \qquad (1)$$

and in detail Eq. (2) taking the sum of S_{LKT} and S_F as S_S and extending the content of the suspension fraction by C_X and X_H *i* [27]:

$$\mathrm{TCOD} = S_{\mathrm{LKT}}\ S_F + S_I + X_S + C_X + X_H + X_I \qquad \mathrm{g\ O_2/m^3} \qquad (2)$$

where S_S – COD of soluble readily biodegradable substrates, S_I – COD of inert soluble organic substrates, X_S – COD of particulate slowly biodegradable substrates, X_I – COD of inert particulate organic substrates, S_{LKT} – COD of volatile fatty acids, S_F – COD of fermentable organic substrates, C_X – COD of slowly biodegradable colloidal substrates, X_H – COD of heterotrophic biomass fraction.

The aim of this study was to use the method of fractionation COD to assess the nature of the organic pollutants in municipal wastewater. In research were analyzed the quality of the raw and treated wastewater taking into account biodegradable and non-biodegradable organic pollutants determined on the basis of the COD fractions.

2. Methodology

The share of fractions can be determined in various methods described in the literature. Some of them require a long time to obtain a result and additional analytical procedures deviating from standard determinations carried out in raw wastewater [28–33]. The commonly used methodology for determining COD fractions is ATV-DVWK A131P guideline, the dimensioning of single-stage sewage treatment plants with activated sludge, developed by the German Association of Engineers and Technicians of Wastewater Treatment [34].

This guideline is widely used in design practice and at the stage of optimization and modeling of the wastewater treatment process. It allows describing processes based on wider than basic characteristics of raw wastewater. According to which, the determination of the soluble readily biodegradable substrates (S_S), inert soluble organic substrates (S_I), particulate slowly biodegradable substrates (X_S) and inert particulate organic substrates (X_I) fraction is based on the determination of COD and BOD_5 in samples of filtered (0.45 μm) and unfiltered raw and treated wastewater.

The COD fractions were calculated based on the modified ATV-DVWK A131P methodology, which is described in the article new approach in COD fractionation methods [35]. According to modified methodology, the determination of the S_S, S_I, X_S and X_I fraction is based on the determination of COD and BOD_5 in samples of filtered (0.45 μm) and unfiltered raw and treated wastewater. Introducing modification allows determining the concentration of the S_S fraction in treated wastewater. Taking into account the kinetic coefficients *k* for raw and treated wastewater increases the accuracy of determining the shares of X_S and S_I fractions. The fractions were calculated as follow:

- COD of inert soluble organic substrates S_I was calculated from the difference between S_{COD} (dissolved organic pollutants) and $BODT_f$ of filtered treated wastewater:

$$S_I = S_{COD} - BODT_f \tag{3}$$

- COD of soluble readily biodegradable substrates S_S is calculated from the difference in the concentration of dissolved organic pollutants S_{COD} determined in raw filtered wastewater and fraction S_I:

$$S_S = S_{COD} - S_I \tag{4}$$

- COD of particulate slowly biodegradable substrates X_S is defined as the difference of total BOD, calculated on the basis of BOD_5 raw unfiltered wastewater and the rate of biochemical degradation and the easily decomposed dissolved fraction:

$$X_S = BODT - S_S \tag{5}$$

BOD_{Tot} of the treated wastewater was calculated taking into account the rate of biochemical degradation determined in the studies (*k*) for treated wastewater:

$$BODT = \frac{BOD_5}{k} \tag{6}$$

- COD of inert particulate organic substrates X_I is determined from the dependence:

$$X_I = X_{COD} - X_S \tag{7}$$

where X_{COD} is the TCOD of organic suspensions.

- TCOD of wastewater is the sum of all fractions:

$$TCOD = S_I + S_S + X_S + X_I \tag{8}$$

The research object was a mechanical–biological wastewater treatment plant located in Lubusz province, Western Poland a size of approximately 26,000 P.E., working in the activated sludge technology. Wastewater is supplied to the wastewater treatment plant by sanitary sewerage and transported by septic tankers. Raw and treated wastewater samples for testing were taken every two weeks for a period of 2 y (24 samples per year, six samples in each term of the season). The research was conducted in 2017–2018.

Wastewater samples were taken in accordance with PN-ISO 5667-10:1997. The COD and BOD parameters were determined according to standard methods: COD – with the potassium dichromate method (PN-74/C-04578.03, PN-ISO 6060:2006), and BOD_5 – with the manometric method, using the OxiTop Control OC110 measurement system. The dissolved fractions were determined in the wastewater filtered through a 0.45 μm filter. Chemical analysis was carried out directly after transporting wastewater samples to the laboratory.

3. Results and discussion

Table 1 presents the average annual wastewater parameters based on the data of the wastewater treatment plant operator. The operator controls one in the month the wastewater parameters. The annual COD and BOD_5 are average values from the monthly values of these parameters. The wastewater characteristic in 2016–2018 points to an increase in average wastewater daily flow and reduction of the COD and BOD_5 in raw wastewater. The reduction of the effectiveness of removing organic pollutants from wastewater expressed as COD from 96.5% to 92.9% was also found (in 2017: 95.4%).

The research conducted in 2017–2018 confirmed the tendencies of changes in the characteristics of wastewater found on the basis of exploratory data. The characteristics of organic pollutants in raw and treated wastewater based on 2 y of research are presented in Table 2. Analysis of average concentrations of organic pollutants in raw wastewater showed a decrease in the COD, BOD_5 and BODT values.

In raw wastewater, the average values of indicators in 2017 were, respectively: COD = 695.1 ± 59.4 mg O_2/dm^3, BOD_5 = 352.7 ± 42.1 mg O_2/L, and BODT = 576.7 ± 69.0 mg O_2/L, and in 2018: COD = 635.6 ± 30.8 mg O_2/L, BOD_5 = 287.7 ± 27.3 mg O_2/L, and BODT = 470.3 ± 44.8 mg O_2/L. During the research in 2017, the average daily flow was 2,740 ± 246 m^3/d and increased in 2018 to 3,370 ± 339 m^3/d.

The decrease in the value of COD, BOD_5 and BODT may be a consequence of the increase in the amount of wastewater

Table 1
Average annual wastewater parameters based on the data of the wastewater treatment plant operator

Wastewater sample	Year	Average daily flow Q, m^3/d	COD, mgO_2/L	BOD_5, mgO_2/L
Raw	2016	2,850	678.5 ± 129	306.3 ± 52.0
	2017	2,920	610.6 ± 103.3	292.9 ± 62.6
	2018	3,630	559 ± 81.2	244 ± 50.4
Treated	2016	2,850	23.6 ± 6.6	3.6 ± 2.0
	2017	2,920	28.5 ± 6.4	3.6 ± 2.0
	2018	3,630	33 ± 11.7	4 ± 1.4

flowing into the wastewater treatment plant. Parameters in treated wastewater were at a similar level. It was found that the removal efficiency of organic pollutants expressed as COD was reduced from 93% to 91%.

For wastewater which contains substances undergoing biochemical oxidation and that are quantitatively oxidizable by chemical means the biodegradability of pollutants can be estimated on the COD/BOD$_5$ ratio. Wastewater can be considered susceptible to biodegradation if 1.5 < COD/BOD$_5$ < 2.5. A high value of the COD/BOD$_5$ ratio (>2.5) indicates a slow decomposition and a high content of organic substances that are hardly decomposable or biologically indecomposable. In turn, the value of COD/BOD$_5$ < 2.0 indicates a significant content of biologically degradable contaminants [36–38]. The ratio COD/BOD$_5$ for raw and treated wastewater was respectively 2.1 ± 0.1 and 8 ± 1.5. If, in addition to easily biodegradable compounds, non-degradable or slowly degradable substances are present in the wastewater, this interpretation may be misleading.

Table 3 presents the values of individual COD fractions of raw and treated wastewater, determined in accordance with the modified ATV-DVWK A131P methodology (point 2, Eqs. (1)–(8)). In accordance with the methodology for determining the X_S and S_I fractions the kinetic coefficients k for raw and treated wastewater were determined. The k values were 0.189–0.191 and 0.157–0.161 d^{-1}, for raw and treated wastewater, respectively.

In raw wastewater, the highest COD values were noted for biodegradable fractions S_S and X_S. The COD of the dissolved fraction S_S was, respectively in 2017 and 2018: 381.3 ± 74.5mg O$_2$/L and 307.1 ± 56.1 mg O$_2$/L. The COD of slowly-biodegradable undissolved fraction X_S was on average 195.4 ± 54.2 mg O$_2$/L and 163.2 ± 88.9 mg O$_2$/L. The average COD values of non-biodegradable, undissolved and dissolved fractions were, in 2017: X_I = 86.5 ± 18.3 mg O$_2$/L, S_I = 31.9 ± 6.2 mg O$_2$/L, and in 2018: X_I = 137.5 ± 38.3 mg O$_2$/L, S_I = 27.8 ± 7.9 mg O$_2$/L, respectively. In treated wastewater, the highest COD values were for dissolved non-biodegradable fractions S_I, 31.9 ± 6.2 and 27.8 ± 7.9 mg O$_2$/L, respectively in 2017 and 2018. In the case of non-biodegradable suspension fractions X_I, the value increased almost twice in 2018, while the concentrations of biodegradable fractions S_S and X_S were at a similar level, of about 7 mg O$_2$/L. The share of individual fractions in the TCOD presents Table 4 and Fig. 1.

The percentage share of individual fractions in the TCOD of raw and treated wastewater was different in the considered period of time. The largest share in raw wastewater was biodegradable fractions: dissolved fraction S_S and slowly-biodegradable undissolved fraction X_S, while the share of dissolved non-biodegradable fractions S_I was the lowest. In treated wastewater, the largest share in TCOD was the S_I fraction.

In 2018, the share of dissolved organic easily biodegradable pollutants (S_S fraction) in wastewater flowing into the treatment plant decreased from 54.6% ± 7.4% to 48.3% ± 8.5%. The share of slowly biodegradable organic pollutants in suspension (X_S) also decreased slightly. It was 28.2% ± 7.7% and 25.6% ± 13.4%, respectively in 2017 and 2018. The share of a dissolved non-biodegradable fraction (S_I) was similar

Table 2
Characteristics of organic pollutants in raw and treated wastewater

Wastewater sample	COD mgO$_2$/L	BOD$_5$ mgO$_2$/L	BODT mgO$_2$/L
2017			
Raw	695.1 ± 59.4	352.7 ± 42.1	576.7 ± 69.0
Treated	50.3 ± 7.3	8.0 ± 0.2	14.0 ± 0.9
2018			
Raw	635.6 ± 30.8	287.7 ± 27.3	470.3 ± 44.8
Treated	50.7 ± 11.2	6.7 ± 2.3	14.5 ± 0.4

Table 4
Share of individual fractions in the TCOD in raw and treated wastewater

Wastewater Sample	S_S	S_I	X_S	X_I
	%			
2017				
Raw	54.6 ± 7.4	4.6 ± 0.6	28.2 ± 7.7	12.6 ± 3.6
Treated	14.5 ± 2.2	63.2 ± 4.3	13.8 ± 3.1	8.5 ± 1.9
2018				
Raw	48.3 ± 8.5	4.4 ± 1.2	25.6 ± 13.4	21.7 ± 6.1
Treated	14.7 ± 3.1	54.4 ± 3.5	15.0 ± 3.0	15.9 ± 3.9

Table 3
COD fractions in raw and treated wastewater

Wastewater sample	S_S	S_I	X_S	X_I	TCOD
	mgO$_2$/L				
2017					
Raw	381.3 ± 74.5	31.9 ± 6.2	195.4 ± 54.2	86.5 ± 18.3	695.1 ± 59.4
Treated	7.2 ± 0.1	31.9 ± 6.2	6.8 ± 0.9	4.3 ± 1.5	50.3 ± 7.3
2018					
Raw	307.1 ± 56.1	27.8 ± 7.9	163.2 ± 88.9	137.5 ± 38.3	635.6 ± 30.8
Treated	7.2 ± 0.1	27.8 ± 7.9	7.4 ± 0.1	8.3 ± 3.4	50.7 ± 11.2

and amounted to about 5%. There was an increase in the share of non-biodegradable suspensions (fraction X_I) from 12.6% ± 3.6% to 21.7% ± 6.1%.

The literature data shows that the share of COD fraction in municipal wastewater is not constant and changes, sometimes considerably. In raw municipal wastewater, the fractions are, respectively: S_S 10%–30%, X_S 50%–65%, X_I 5%–20%, and S_I up to 10% [27,31]. The test results showed differences in the proportions between the S_S and X_S fractions, which may be connected with the sewage system supply. The shares of the S_I and X_I fractions were consistent with the literature data. The proportion of individual factions is significantly affected by the share of industrial wastewater. The share of COD fractions in municipal wastewater with a significant share of paper industry wastewater was, respectively S_S 4.2%, X_S 43.1%, X_I 13.2%, and S_I 39.5% [39].

In treated wastewater, differences in the share of non-biodegradable fractions were noted. Decreased the share of a dissolved non-biodegradable fraction (S_I) from 63.2% ± 4.3% to 54.4% ± 3.5%, and increased the share of non-biodegradable suspensions (fraction X_I) from 8.5% ± 1.9% to 15.9% ± 3.9%. It was also noted an increase in the share of total suspended solids fractions from 22.2% ± 2.9% to 30.9% ± 2.1% in 2017 and 2018, respectively which also confirms the particle size distribution in the non-filtered treated wastewater (Fig. 2). The average particle size in treated wastewater in 2017 amounted 40 to 100 nm, and 60 to 120 nm in 2018.

The total share of biodegradable (COD_S) and non-biodegradable (COD_I) fractions in TCOD in raw and treated wastewater presents Fig. 3.

The total share of biodegradable pollutants in the wastewater flowing into the treatment plant decreased from 82.8 ± 3.2 to 73.9 ± 4.9%, while in the treated wastewater the participation of biodegradable organic pollutants increased. Analysis of the COD fraction in raw wastewater (Fig. 1) showing a decrease in the share of S_S fraction and an increase in the share of X_I fraction with a similar share of other fractions (S_I and X_S), may indicate a slowdown in the hydrolysis of organic pollutants in the sewage system or be a consequence of, for example, connections non-biodegradable micropollutants into larger agglomerates.

Przywara et al. [40] determined COD fractions for wastewater treated in the anaerobic process under psychrophilic conditions. The share of biodegradable pollutants (fractions S_S and X_S) in raw wastewater ranged from 47% to 68%, while in treated wastewater non-biodegradable pollution predominated, ranging from 53.6% to 58.1%.

The determinant of good work of wastewater treatment plants in the scope of removing organic pollutants is the high share of non-biodegradable pollutants in wastewater

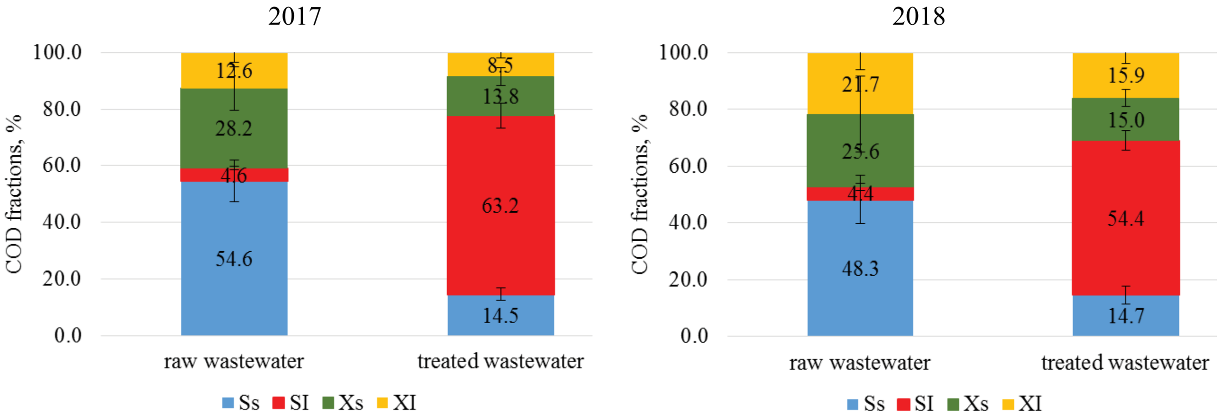

Fig. 1. Share of individual fractions in the TCOD.

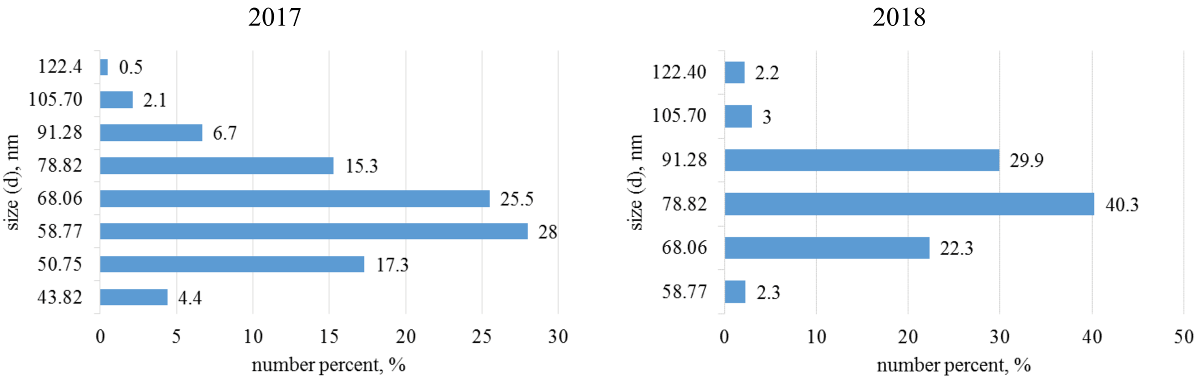

Fig. 2. Particle size distribution in the non-filtered treated wastewater.

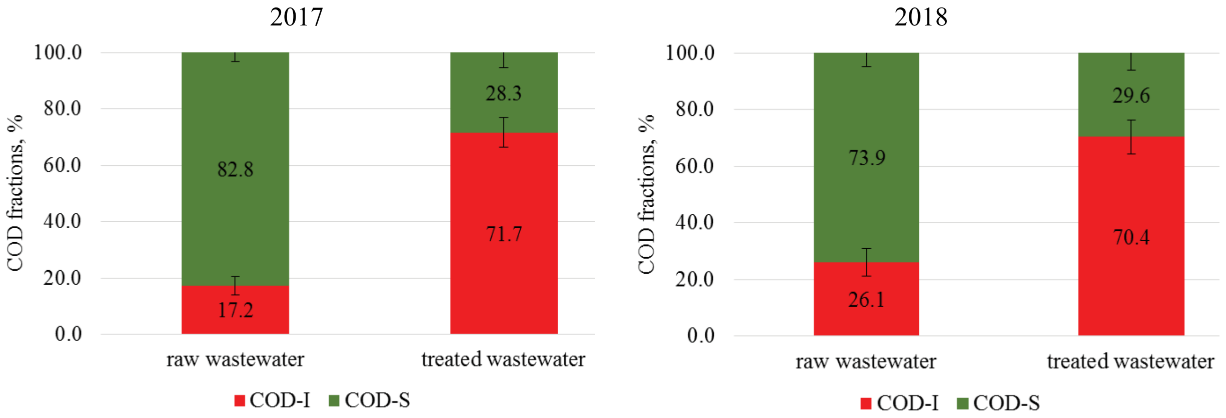

Fig. 3. Share of biodegradable (COD_S) and non-biodegradable (COD_I) organic compounds in TCOD.

discharged from the wastewater treatment plant. An increase in the share of biodegradable pollutants in treated wastewater indicates a worse efficiency of the treatment plant. This may be a consequence of a change in the nature of organic pollutants contained in wastewater, for example, as a result of an increase in the number of non-biodegradable micro-pollutants.

The growing diversity of pollutants identified in municipal wastewater requires the search for indicators that describe well the characteristics of wastewater. It is becoming common to use computer tools to model processes in treatment plants using both simple models, for example, neural networks, and advanced ASM models. The accuracy of the mathematical model depends on the number of variables entering into the analysis in the model [41,42]. Based on the conducted research, it has been shown that the COD fractionation method is a good indicator for assessing the nature of organic pollutants in municipal wastewater. It allows a more detailed assessment of the organic impurities, compared with standard summary indicators such as BOD_5 and COD. Determination of the four basic fractions does not require the use of advanced analytical methods and provides many details on the quality of the wastewater. In connection with the kinetics decomposition of pollutants that is appropriate for given wastewater, it allows determining with high accuracy the share of biodegradable and non-biodegradable pollutants. The literature data shows, that COD fractions are most often used to characterize raw wastewater, while COD fractionation of treated wastewater is less common. Periodic determination of the COD fractions may be a method of early monitoring of changes in the nature of pollutants influent to the wastewater treatment plant in relation to their biodegradability. In the case of identified changes in the effectiveness of the wastewater treatment plant, determining the COD fraction in raw and treated wastewater is required, and can be helpful in optimizing wastewater treatment processes. Based on the share of biodegradable and non-biodegradable fractions in treated wastewater, the effectiveness of wastewater treatment processes can be assessed with greater accuracy. Knowledge of the COD fractions in treated wastewater is important due to the migration of organic pollutants, including micro-pollutants from wastewater to the aquatic environment and may be an important part of its monitoring. The COD fractions can be a useful parameter in modeling processes occurring in surface waters, which are treated by wastewater receivers. Further research in this area is necessary.

4. Conclusions

- COD fractionation is a good analytical indicator of wastewater quality and for assessing the nature of organic pollutants in municipal wastewater.
- It allows a more detailed assessment of the organic impurities, compared with standard summary indicators such as BOD_5 and COD.
- Determination of the four basic fractions, in connection with the kinetics decomposition of pollutants, allows determining with high accuracy the share of biodegradable and non-biodegradable wastewater compounds.
- Based on the share of biodegradable and non-biodegradable fractions in treated wastewater, the effectiveness of wastewater treatment processes can be assessed with greater accuracy.
- Knowledge of the COD fractions in treated wastewater may be an important part of aquatic environment monitoring.

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