

## Impact of Pharmaceuticals on the Individual Wastewater Treatment System

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### ABSTRACT

Intensive economic development causes the risk of introducing the so-called emerging contaminants (EC) into the environment. This group of contaminants includes pharmaceuticals, including non-steroidal anti-inflammatory drugs (NSAIDs), the consumption of which is steadily increasing in Poland as well as worldwide. Such specific contaminants may also cause problems at the stage of biological treatment of wastewater, especially in individual systems that are sensitive to changes in wastewater composition. The study examined an onsite wastewater treatment system based on the activated sludge technology, to which three different pharmaceuticals were dosed in the quantities normally used in therapy. The operation of the biological reactor under these conditions was analysed by comparing contamination indicators at the inlet and outlet of the treatment plant at different doses of pharmaceuticals. It was found that the doses used did not significantly worsen the effects of wastewater treatment, except for paracetamol, which at a dose above 1000 mg/d caused a slight reduction in the efficiency of the facility. The dosed pharmaceuticals also did not deteriorate the sedimentation properties of the activated sludge. Although typical doses of pharmaceuticals do not interfere with the operation of the biological reactor, residual substances and their metabolites may be released into the environment; it is therefore reasonable to consider the introduction of an additional treatment stage to remove micropollutants from the wastewater.

**Keywords:** wastewater treatment, small treatment plants, activated sludge, pharmaceuticals.

### INTRODUCTION

Continuous economic development entails the increase in production in various fields and the risk of introducing more and more waste into the environment. These are often the so-called emerging contaminants (EC), whose impact on the environment is not yet well recognised. The United States Geological Survey (USGS) defines that these are synthetic or naturally occurring chemicals that are not commonly monitored in the environment, but have the potential to enter the environment and directly cause adverse effects on human health, which is all the more dangerous because it is not yet well recognised. Monitoring of these substances is possible after introducing modern detection methods [Perez-Fernandez et al., 2017]. One of the fastest growing industries is the pharmaceutical industry. The

use of drugs, especially over-the-counter drugs, is constantly increasing; a large group of these are the so-called NSAIDs, i.e. non-steroidal anti-inflammatory drugs. It is estimated that between 5,000 and 10,000 different types of pharmaceuticals are available on the markets of various countries, and on average in Poland, approx. 30 packages of drugs are purchased per person per year [Pachnicka and Olejnik, 2019].

One of the important aspects of the increase in hazardous and difficult-to-degrade contaminants (emerging contaminants) in domestic wastewater is the increase in the use of dietary supplements and medicinal products by society, especially those that can be purchased over-the-counter. Poland is among the countries with the highest consumption (purchase) of them, e.g. it ranks first among EU countries in terms of carbamazepine consumption [Björleinius et al. 2018].

Pharmaceutical products enter water in the form of metabolites or in unchanged form [Boroń & Pawlak, 2015]. Seasonal variability in the presence and quantity of pharmaceuticals was also observed due to the seasonality of disease. The group of drugs identified in both raw and treated domestic wastewater is numerous, including anti-inflammatory, hypertension, anticonvulsants, antipsychotics and antibiotics. The active substances include, among others: ibuprofen, triclosan, sulfathiazole, naproxen, diclofenac, paracetamol, atenolol, ketoprofen, 17-alpha-estradiol and 17-beta-estradiol [Sui et al. 2010, Ternes et al. 2002, Behera et al. 2011].

The interpretation and analysis of the processes accompanying the transformation of pharmaceuticals in wastewater is hampered by the fact that there is a high variability in the concentrations detected at different sites and the presence of some pharmaceuticals in the influent, such as ibuprofen, tetrahydrocannabinol and naproxen, but also in treated wastewater – e.g. tetrahydrocannabinol, triclocarban, gemfibrozil and diclofenac; the presence of these substances in treated wastewater is of particular concern [Carmona et al. 2014]. The residue of many pharmaceuticals in the effluent from wastewater treatment plant is due to the non-adaptation of conventional wastewater treatment technologies to this type of specific contaminant [Tiedeken et al. 2017]. Analgesics are removed with varying degrees of efficiency – ibuprofen, ketoprofen, aspirin – often more than 90%, diclofenac – much worse – approx. 67–75%. Trimethoprim, diclofenac, tetracycline, ibuprofen and naproxen showed low effective removal during flow through wastewater treatment plants, with high stability both at the inflow and outflow of the wastewater treatment plants [Papa-georgiou et al. 2016, Leung et al. 2012].

The most common anti-inflammatory drugs are codeine, ibuprofen, diclofenac, naproxen, ketoprofen, acetylsalicylic acid, fenoprofen, paracetamol. These pharmaceuticals not only directly contaminate surface and groundwater, but also pose a problem at the stage of wastewater treatment on various scales. The European Union and Poland have not introduced a limit on the amount of emerging contaminants in water. The only country that has introduced regulations is Switzerland, where the law requires that the technological process of wastewater treatment be upgraded to remove micropollutants, especially pharmaceuticals [Kosiniak and Muszański,

2021]. Activities to reduce the impact of micropollutants, including pharmaceuticals, on the aquatic environment is included in the proposal to amend Directive 2000/60/EC of the European Parliament and of the Council of 23 October 2000 establishing a framework for Community action in the field of water policy.

In most cases, the treatment of municipal wastewater involves mechanical and biological methods. The efficiency of removing pharmaceuticals on suspensions depends on the nature of the substance and the interaction between drug particles and suspensions and is usually low [Wontorska and Wąsowski, 2018]. Higher efficiencies are achieved at the biological stage, where activated sludge is the basis of the technology. The reduction of pharmaceuticals then takes place through adsorption on sludge flocs and biodegradation. Hatoum et al. [2019] carried out studies on an SBR-type laboratory reactor, using different ages of activated sludge. They found that the efficiency and rate of removal of NSAIDs pharmaceuticals increases with the increasing age of biomass and concentration of activated sludge in the reactor. Ternes [2004] found that, in addition to the age of the sludge, the temperature at which the wastewater is treated has an impact on the increase in pharmaceutical removal rates. The adsorption efficiency of diclofenac, hydroxyzine and sulphonamides on sludge flocs reaching 75% was highlighted by Salgado et al. [2012]. The authors concluded that biological processes are the main mechanism for removal of pharmaceuticals, followed by adsorption and UV radiation. The removal of NSAIDs pharmaceuticals can be effectively performed by adsorption on activated carbon [Pirvu et al., 2022]. In this case, the pH and the adsorbent dose are important.

The aim of this study was to investigate the impact of selected pharmaceuticals from the NSAID group on the individual wastewater treatment system. A biological treatment plant based on the activated sludge method, additionally equipped with a sand filter and UV lamp, was selected for the study.

## MATERIALS AND METHODS

The study was carried out on an individual wastewater treatment system based on the activated sludge technology in the SBR system. The facility is located on private property and receives

wastewater from a single-family house with four residents (Fig. 1). The average household water consumption is  $0.573 \text{ m}^3/\text{d}$ . The raw wastewater flows into the so-called accumulation chamber with a capacity of  $1 \text{ m}^3$ , the task of which is to carry out mechanical pre-treatment and to level unevenness in the inflow. The wastewater is then pumped to an activated sludge chamber with a capacity of  $1.5 \text{ m}^3$ , where biological treatment processes take place. The working cycle of the bioreactor ends with sedimentation and discharge of the clarified wastewater into a sand filter with an area of  $0.1 \text{ m}^2$ , in the form of a layer of sorted sand with a thickness of 40 cm and a granulation of 1–3 mm, and then through a UV lamp into the outflow. At this time, the excess sludge is pumped into a sludge stabilisation chamber with a capacity of  $0.46 \text{ m}^3$ . The system is also equipped with a PAX coagulant dispenser for chemical precipitation of phosphorus. The coagulant is dosed into the reactor chamber, in the amount of  $50 \text{ ml}/\text{m}^3$  of chamber volume.

Air lifts are used to transport wastewater and sludge, and an automatic, suitably programmed controller is responsible for controlling the processes. The working cycle length of the biological reactor was set at 1 day. The influent is typical domestic wastewater. The users of the facility follow a high-protein and high-fat diet and do not take any pharmaceuticals on a chronic basis. They use biodegradable preparations to maintain cleanliness.

The study of the impact of pharmaceuticals on the treatment plant was carried out in four stages. It started with the preliminary stage (stage

0), which aimed to assess the efficiency of the wastewater treatment plant under normal operating conditions. This was followed by 3 main stages of the study, in which the doses of: diclofenac, ibuprofen and paracetamol were administered. Each main stage consisted of three or four series of studies, varying in dose size. Pharmaceuticals were dosed into the equalization tank once a day, at the doses shown in Table 1. Dose sizes were determined on the basis of the therapeutic recommendations for individual pharmaceuticals.

In each series, pharmaceuticals were dosed and wastewater samples were collected for four days, with a daily shift due to the length of the reactor working cycle (Table 2) and a weekly interval between series. Pharmaceuticals were dissolved in 100 ml of demineralised water before dosing into the tank. Wastewater samples were taken: raw wastewater after mechanical treatment (without pharmaceutical substance), raw wastewater after mechanical treatment (with pharmaceutical substance), after biological treatment, after treatment in a sand filter, after treatment in a UV lamp stage; a sample of activated sludge was also taken each time from the bioreactor while mixing its contents. A COD analysis was also carried out for a solution of pharmaceuticals in the proportion of 1 tablet per 1 litre of water.

The analyses of wastewater and sludge samples were carried out at the analytical laboratory of the Department of Hydraulic and Sanitary Engineering of the Poznan University of Life Sciences. In accordance with the applicable ATV-131 methodology

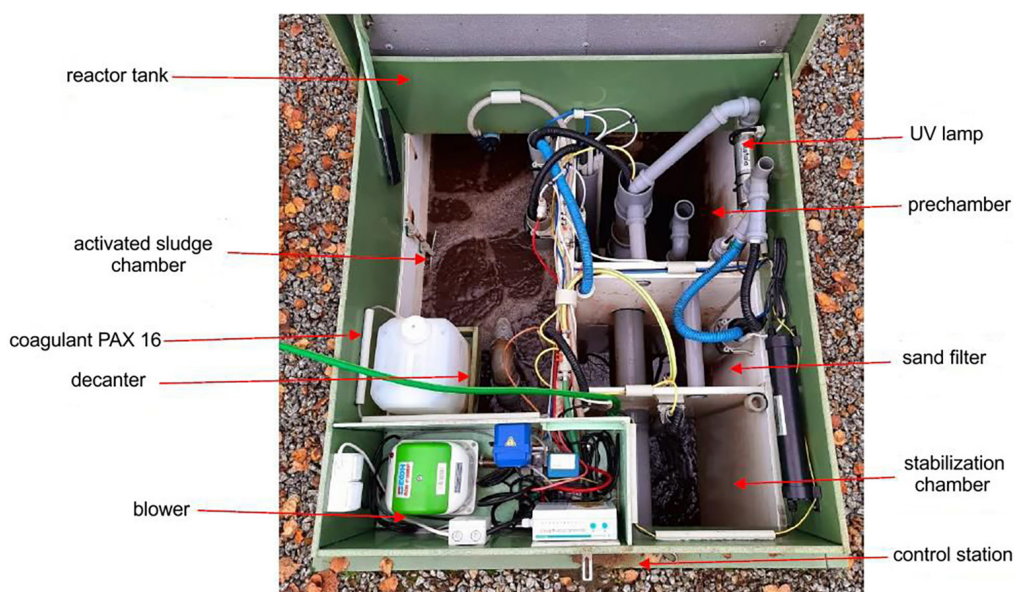


Fig. 1. Individual wastewater treatment system used in the study

**Table 1.** Doses of pharmaceuticals used in the study, mg/d

Stage Series	STAGE 1 Diclofenac	STAGE 2 Ibuprofen	STAGE 3 Paracetamol
Series 1	50	200	1000
Series 2	100	600	2000
Series 3	150	800	3000
Series 4	200	-	-

**Table 2.** Diagram of pharmaceutical dosing and sampling

Day 1	Day 2	Day 3	Day 4
Drug dosage	Drug dosage	Drug dosage	-
Sampling	Sampling	Sampling	Sampling

and the Polish Standard, the following contamination indicators were determined: chemical oxygen demand (COD), five-day biochemical oxygen demand (BOD<sub>5</sub>), ammoniacal nitrogen (N-NH<sub>4</sub>), phosphates (P-PO<sub>4</sub>), total suspended solids, organic suspended solids and control nitrate nitrogen (N-NO<sub>3</sub>). The results were read using the Merck NOVA 60 spectrophotometer. A sedimentation test was carried out for activated sludge and the Mohlman Sludge Index was calculated.

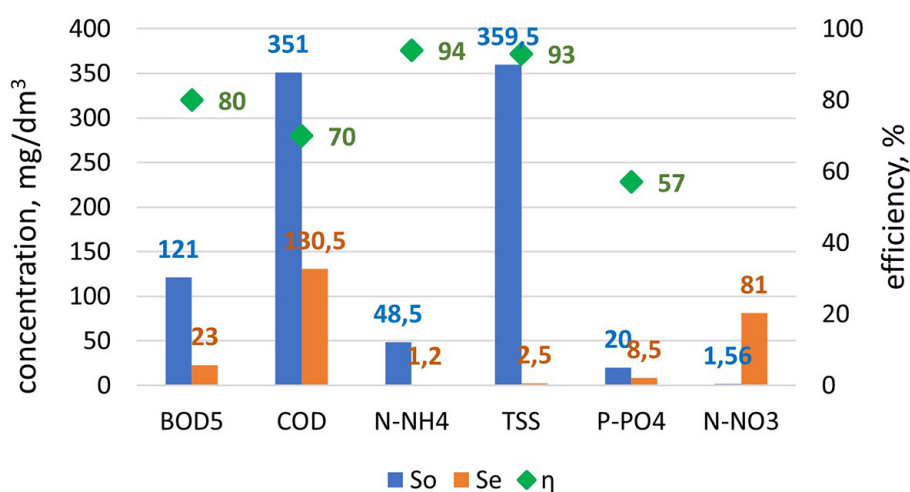
As part of the study, the microscopic analysis of the activated sludge was also carried out, involving microscopic observation and photography of the sludge samples and analysis of the resulting image. The Zeiss Axiostar Plus light microscope, with 100x magnification (10x objective, 10x eyepiece), was used for the analysis. The abundance and species analysis of the observed microorganisms occurring per 1 cm<sup>2</sup> were verified. This value

is calculated using the transverse and longitudinal lines when calibrating the apparatus: 1.00 mm – spacing between triple lines, 0.25 – spacing between thin lines. The results of the analyses from the entire study period were statistically analysed for a broader interpretation. Arithmetic mean, standard deviation and correlation analysis between variables were used for this purpose.

## RESULTS AND DISCUSSION

### Preliminary studies

A series of preliminary studies included quality analyses of the wastewater flowing into and from the treatment plant. The average values are shown in Fig. 2. The values of indicators for raw wastewater were within the ranges reported in the



**Fig. 2.** Average values of contamination indicators in the analysed treatment plant under normal operating conditions; S0 – raw sewage, Se – treated sewage



literature [Kaczor, 2009; Masłoń, 2015]. In contrast, the ratio of organic compounds expressed as COD and BOD<sub>5</sub> was 3:1, which indicated the presence of components that are more difficult to degrade compared to typical wastewater (on average COD:BOD<sub>5</sub> = 2:1), and may be related to the diet followed by the system users. The used pharmaceutical doses did not increase the COD values of the wastewater. The COD of the pharmaceutical solution in the previously given proportion was from 3,200 to 3,700 mgO<sub>2</sub>/dm<sup>3</sup>, however, the very high dilution in relation to the amount of the influent resulted in a change in the indicator value of approx. 0.1%. The high removal efficiency of organic compounds and suspended solids allowed satisfactory results in the treated wastewater. Nitrification was very effective, but denitrification was ineffective, which could be related to inadequate oxygen conditions and organic substrate availability. The removal of phosphorus was less than 60% effective, which could be related to an inaccurate coagulant dose. However, in onsite treatment plants from which the treated wastewater is discharged to own premises or within or outside the agglomeration of up to 2,000 PE, the removal of nutrients is not required. The activated sludge concentration in the bioreactor exceeded 9 g/dm<sup>3</sup>, which would indicate the need to remove excess sludge, but the composition of the sludge was mostly bacterial and the sludge index averaged

53 with a sedimentation test score of 481, so the sludge showed good sedimentation properties.

### Impact of pharmaceuticals on the treatment plants

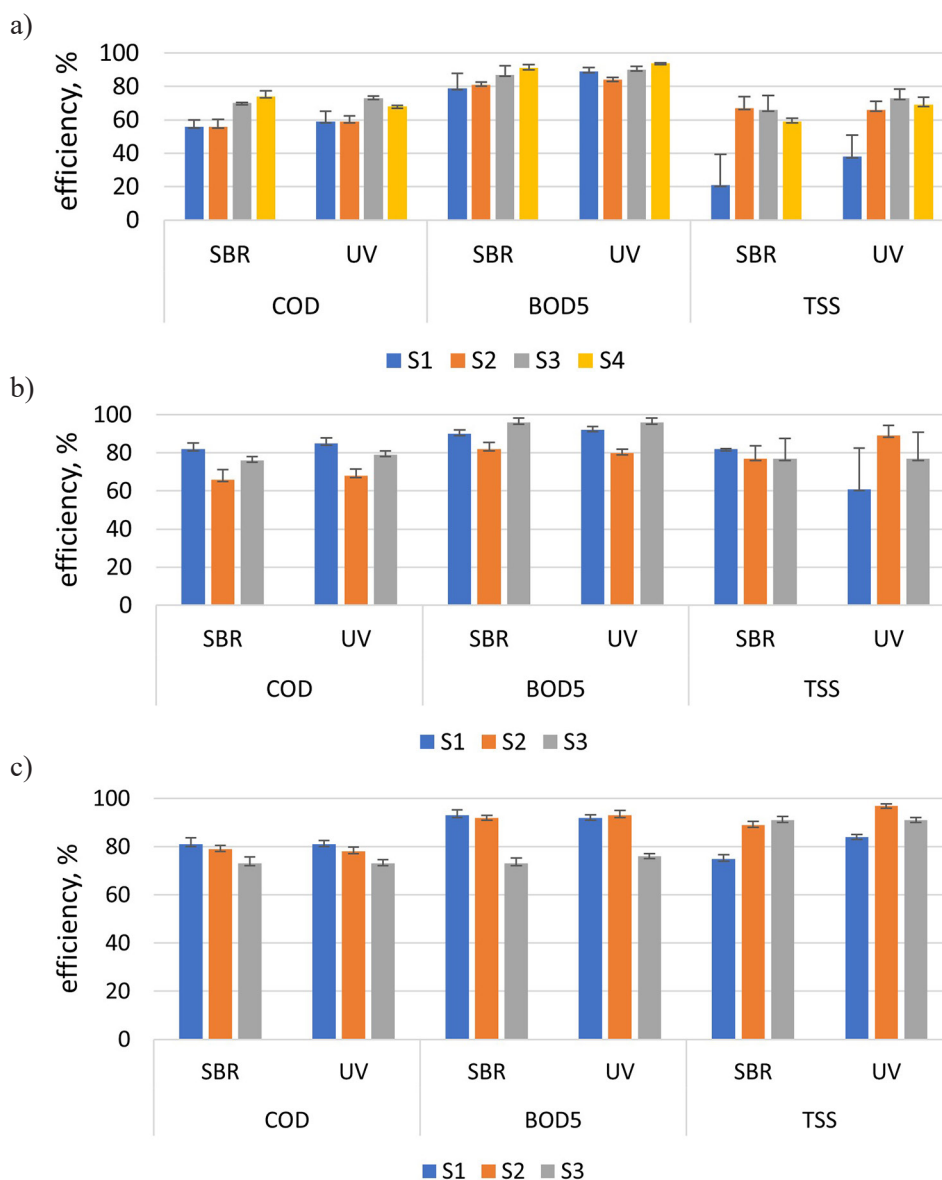
The main part of the study paid attention to possible changes in the removal efficiency of contaminants contained in the influent flowing into the treatment system and the properties of the activated sludge during pharmaceutical dosing. The assessment of the impact of pharmaceuticals on the treatment plant was based on an analysis of the efficiency of the system. Table 3 contains the values of the contamination indicators in the treated wastewater after the biological reactor and at the outlet from the treatment plant. The COD and BOD<sub>5</sub> values showing the content of organic compounds were within the limits set for the smallest treatment plants, except for three series of the first stage. It was assessed that the increased values were related to the higher indicators observed at the inlet to the treatment plant: COD then reached a maximum of 734 mg O<sub>2</sub>/dm<sup>3</sup>, while in the other series it ranged from 260 to 530 mg O<sub>2</sub>/dm<sup>3</sup>. This was important especially due to the type of diet followed by the household members. BOD<sub>5</sub>, although slightly higher than in the other stages, met the requirements for the smallest treatment plants. The high total suspended solids content

**Table 3.** Average values of contamination indicators after the biological reactor (\*) and at the outflow from the analysed treatment plant in successive stages of the study

Stage of research		COD	BOD <sub>5</sub>	TSS	N-NH <sub>4</sub>	P-PO <sub>4</sub>
		gO <sub>2</sub> /dm <sup>3</sup>	gO <sub>2</sub> /dm <sup>3</sup>	g/dm <sup>3</sup>	gN/dm <sup>3</sup>	gP/dm <sup>3</sup>
Stage 1	Series 1	288.75 ± 36.99* 274.33 ± 23.13	20.00 ± 5.00	219.90 ± 19.06	0.16 ± 0.03	9.17 ± 0.09
	Series 2	225.45 ± 11.24* 210.00 ± 9.67	23.75 ± 1.25	182.50 ± 24.66	0.13 ± 0.01	9.00 ± 0.24
	Series 3	155.77 ± 36.75* 169.33 ± 60.03	13.33 ± 3.33	173.60 ± 41.14	0.27 ± 0.06	9.77 ± 0.26
	Series 4	82.67 ± 4.18* 93.00 ± 11.00	5.00 ± 0.00	133.90 ± 34.50	0.40 ± 0.09	10.40 ± 0.10
Stage 2	Series 1	63.00 ± 11.22* 53.75 ± 9.72	8.75 ± 2.39	112.80 ± 61.48	0.49 ± 0.08	11.98 ± 0.49
	Series 2	111.25 ± 3.61* 106.75 ± 5.81	17.50 ± 4.33	49.35 ± 18.73	0.83 ± 0.14	13.50 ± 0.13
	Series 3	68.50 ± 2.72* 62.00 ± 1.83	3.75 ± 2.39	45.60 ± 21.90	0.43 ± 0.05	11.45 ± 0.37
Stage 3	Series 1	72.75 ± 1.65* 71.50 ± 3.80	8.50 ± 0.96	68.55 ± 11.78	0.49 ± 0.23	13.50 ± 0.30
	Series 2	77.50 ± 2.60* 78.00 ± 4.24	7.00 ± 1.00	32.60 ± 18.90	1.62 ± 0.51	13.53 ± 0.33
	Series 3	95.25 ± 12.96* 82.00 ± 2.71	7.50 ± 2.06	40.40 ± 22.88	2.64 ± 0.23	11.48 ± 0.21

indicated low sedimentation efficiency during this period. Ammoniacal nitrogen was oxidised with very high efficiency, while the high phosphate content in the outlet throughout the study period indicated insufficient chemical support for phosphorus removal with values in the inlet above 20 mg P/dm<sup>3</sup>. Due to the high efficiency of the biological stage, especially in terms of organic compound removal and ammoniacal nitrogen oxidation, the sand filter and UV lamp had very little effect on improving the quality of the treated wastewater, thus no separate analysis of the efficiency of these elements of the treatment plant was carried out. The sand filter reduced total suspended solids, but its efficiency was poor due to the high hydraulic load.

Indicators relevant to the capacity of the treatment plant were further analysed. Figure 3 shows the efficiency of removal of individual components from the wastewater at successive stages of the study, at the SBR and at the outlet from the treatment plant. The removal efficiencies for organic contaminants were high, in the case of BOD<sub>5</sub> even above 90%, except for stage 1, as explained earlier. The treatment system was the least stable in terms of suspended solids removal, as indicated by the relatively high standard deviations for this indicator compared to the others. The instability of suspended solids removal can be related to the length of the sedimentation phase and not to the influence of pharmaceuticals. The nitrification efficiency in all



**Fig. 3.** Removal efficiency of contaminants in successive stages of the study, average values; (a) stage 1, (b) stage 2, (c) stage 3; S1 – series 1, S2 – series 2, S2 – series 3, S4 – series 4

stages of the study exceeded 90%, demonstrating that the dosed pharmaceuticals did not have a negative impact on the population of nitrifiers. The efficiency of phosphate removal remained at a similar level throughout the study period for the reasons described earlier.

An attempt was also made to determine the dependence of the degree of contamination removal on the dose of individual pharmaceuticals. Table 4 contains the coefficients of determination obtained for these indicators. As reported by Szydłowski [1978], the correlation of the analysed quantities is significant in Stage 3 for COD at a significance level of 0.05 and for BOD<sub>5</sub> at a significance level of 0.1, and to a small extent in Stage 1 at a significance level of 0.1. Based on the presented results, it can be concluded that the treatment effect was influenced by dosing with paracetamol and, to a small extent, by dosing with diclofenac. The degree of organic substrate removal decreased by a few to several percent. The dependencies for which the best fit was found are shown in Fig. 4. The diclofenac particles are degraded relatively slowly by microorganisms due to their structure [Cirja et al. 2007]; the ability to eliminate this pharmaceutical has only been observed in some fungi [Wang and Wang, 2016, Santos et al. 2012, Popa et al. 2014]. The lack of a negative impact of diclofenac on the microorganisms may be related to sorption on activated sludge flocs [Gurung et al. 2019]. Paracetamol,

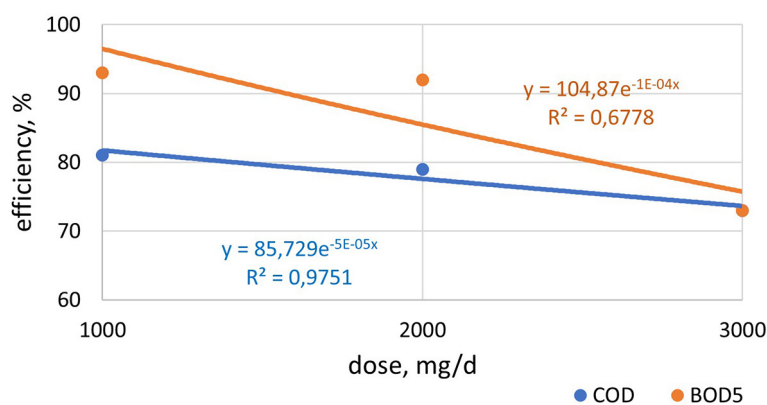
on the other hand, is more easily degraded by enzymes produced by microorganisms to less harmful intermediate forms, but the retention time of wastewater (HRT – hydraulic retention time) and of biomass (SRT – solids retention time) in typical activated sludge facilities may not be sufficient to achieve adequate efficiency of this process [Hatoum et al., 2019, Teharan et al. 2016, Goswami et al. 2018]. Although Joss et al. [2005] found no impact of either SRT or HRT on the removal of seven pharmaceuticals, they conducted their study on full-scale wastewater treatment plants. Goebel et al. [2007], in turn, suggest that differences in the removal rate of some pharmaceuticals from wastewater depend not on SRT or HRT, but on the substrate-to-pharmaceutical or substrate-to-biomass concentration ratio. The latter decreases with increasing SRT; an increase in the amount of active biomass leads to an increase in the intensity of contaminant removal processes.

#### Sedimentation properties of the activated sludge

The quality of the activated sludge in the reactor was also controlled during all series of the tests. The sludge concentration in the first stage was relatively high, reaching up to 9 g/dm<sup>3</sup>. The decrease in concentration in the second stage was caused by service works carried out at that time. The result of the sedimentation test indicated high

**Table 4.** Coefficients of determination for selected contamination indicators

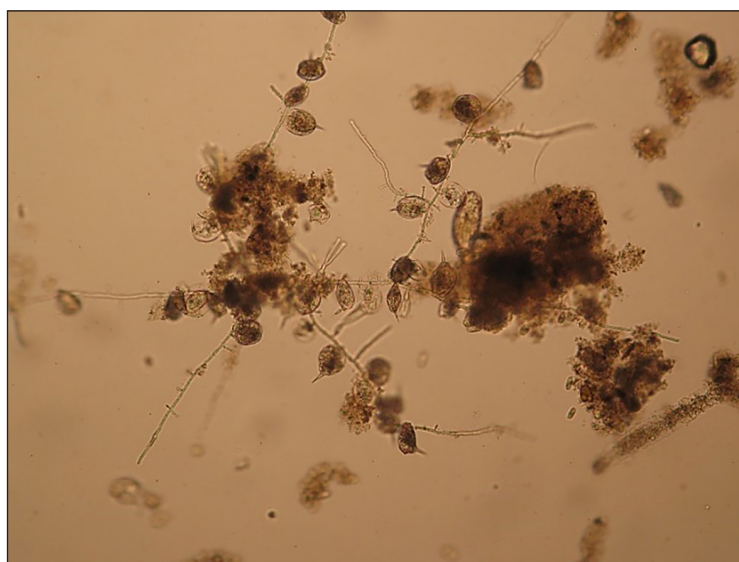
Indicator/stage	Stage 1	Stage 2	Stage 3
COD	0.5708	0.2813	0.9751
BOD <sub>5</sub>	0.5636	0.0028	0.6778
TSS	0.5766	0.4752	0.279



**Fig. 4.** Dependence of the degree of contaminant removal on the dose of paracetamol

**Table 5.** Characteristics of the activated sludge in successive stages of the study (average values); X – sludge concentration,  $V_{1/2}$  - volume of sludge after 1/2 hour of sedimentation, IO - Mohlman sludge index

Phase of research		X	$V_{1/2}$	IO
		g/dm <sup>3</sup>	cm <sup>3</sup>	cm <sup>3</sup> /g
Stage 1	Series 1	8.93 ± 1.65	176 ± 3	21.65 ± 3.05
	Series 2	5.12 ± 0.13	169 ± 5	33.01 ± 0.98
	Series 3	6.16 ± 1.29	182 ± 9	31.24 ± 4.17
	Series 4	7.32 ± 0.83	176 ± 7	25.30 ± 3.71
Stage 2	Series 1	5.39 ± 2.00	109 ± 5	29.16 ± 8.74
	Series 2	2.71 ± 0.04	130 ± 2	49.92 ± 1.37
	Series 3	2.78 ± 0.25	155 ± 5	56.89 ± 4.15
Stage 3	Series 1	3.96 ± 0.16	185 ± 4	46.92 ± 1.90
	Series 2	3.94 ± 0.21	210 ± 14	53.66 ± 4.11
	Series 3	5.53 ± 0.59	229 ± 1	42.96 ± 5.25

**Fig. 5.** Stalked ciliates in activated sludge microphotography

fragmentation of the flocs. The good sedimentation properties of the sludge are confirmed by the Mohlman Sludge Index values, which did not exceed 60 cm<sup>3</sup>/g (Table 5).

### Microscopic tests on the activated sludge

The microscopic analysis of the sludge allowed the identification of a relatively large number of stalked ciliates, shown in the figure below (Fig. 5). The abundant presence of stalked ciliates indicates a sludge working stably, effectively reducing organic matter as it is well absorbed on the surface of the flocs. An occasional occurrence of crawling ciliates was also observed at a later stage, which would suggest that the sludge had reached

an appropriate maturity. The share of rotifers and oligochaetes in the structure of the floc indicates high efficiency of the biological decomposition process with simultaneous proper oxygenation of the wastewater. In addition, oligochaetes are only found in well-oxygenated sludge with a high degree of BOD<sub>5</sub> reduction. The microscopic observations of the activated sludge confirm that the impact of the presence of pharmaceuticals in the influent was insignificant.

### CONCLUSIONS

The study on the impact of commonly used NSAID pharmaceuticals on the biological treatment of wastewater was carried out on an



individual system receiving domestic wastewater only. The amounts of pharmaceuticals introduced into the system were determined on the basis of the used therapeutic doses. Small treatment systems are usually more sensitive to changes in the composition of the influent, especially to the presence of toxic components. In the analysed cases, the doses of drugs used turned out to have no negative impact on the wastewater treatment biomass, which was confirmed by comparing the efficiency of the processes carried out without and with the addition of pharmaceuticals and by analysing the microscopic image of the activated sludge. The exception was the increased doses of paracetamol, which resulted in a slight reduction in the efficiency of removal of organic compounds from wastewater. With SRT ranging from a few to several days, usually occurring in small treatment systems, paracetamol might have been biodegraded to less toxic compounds to an insufficient extent. Although the amounts of added pharmaceuticals turned out to be non-toxic to the operation of the activated sludge, the problem may be residual drugs and their metabolites remaining in the treated wastewater and entering the environment with them. Joss et al. [2006] proposed a classification scheme for the biodegradability of pharmaceuticals based on their biodegradation rate constants. The authors concluded that current methods of treating municipal wastewater do not effectively remove micropollutants. It can be a good solution to protect against emissions of these components by using a stage to treat the wastewater of micropollutants in the form of, for example, an activated carbon bed filter [Pirvu et al., 2022].

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