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Quantitative analysis of microplastics in wastewater during selected treatment processes

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ABSTRACT

Microplastics are plastic particles with a diameter of 1 μm–5 mm. In recent decades, contamination by plastic microparticles has become a serious environmental problem of a global nature. Wastewater treatment plants (WWTPs) are recognized as an important source of these micropollutants in surface waters – conventional methods of wastewater treatment do not completely remove plastic microparticles. The present research aimed to evaluate quantity and quality as well as estimate the removal efficiency of microplastics in the large and medium WWTP. The research carried out in two WWTPs (large and medium) showed the presence of microparticles of plastics in various forms – microfragments of foil, microfibers, and microgranules. The total concentration of microplastics in the grit chamber was 4,700–7,200 particles/m^3 in the medium WWTP and 2,140–2,220 particles/m^3 in the large one. In effluent, the concentration was not exceeded 600 and 780 particles/m^3, respectively. Both WWTPs showed high removal efficiency of fibers and microgranules removal (over 90%). Microfoils were removed in less than 50%.

Keywords: Microplastic; WWTP; Wastewater

1. Introduction

1.1. Definition of microplastics

Most materials belonging to the collection of plastics are often colloquially called plastic. The term "plastic" comes from the Greek word "plastikos," which means forged, easily formable material [1]. Other characteristic features of plastics include low weight, durability, resistance to corrosion, chemical, and physical factors [2,3].

Global plastic production reached almost 350 million tons in 2017, increasing by 3.9% over the previous year. European production amounted to 64.4 million tons, which is 7.3% higher than in 2016 [4]. Most plastics are produced in Asia, but the demand for plastic per capita is higher in developed countries. Nowadays plastics are used in almost every walk of life. The features that make plastic so useful, make it a very problematic waste at the same time. The lifetime of products made of plastics can vary widely. In some cases, this time may significantly exceed 50 y, but unfortunately in most cases, it is much shorter than 1 y. Plastic packaging, representing the largest percentage of the European processing market, is usually used once. This situation leads to the formation of a huge amount of waste. In 2016, the amount of plastic waste collected in Europe was estimated at around 27.1 Mt [4]. Although the percentage of plastic landfilled for the first time in European history was less

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than the percentage of plastic waste recycled, 7.40 Mt of plastic that went to landfill is still a big problem. It needs to be highlighted there are wastes thrown away illegally every year and the fact that the percentages presented earlier relate to Europe, while, for example, in Asian countries, the situation is much worse and in China, the percentage of recycled plastic waste is less than 10% [5].

Special cases of this problem are microplastic particles – those created in microscopic sizes, as well as those that have reached small sizes due to decay. Microplastic is present in the whole surrounding environment. The presence of microscopic-sizes plastic particles in the water of the Atlantic Ocean was first noticed and described by Edward J. Carpenter in the early 1970s [6] and since then, the problem has been gradually explored. The term "microplastics" was used first to describe these microscopic plastic particles only in 2004 by Richard C. Thompson of the University of Plymouth [7]. Thompson's definition of the word is "very small plastic particles or fibers." Sizes allowing to classify the particle as microplastics were not fully unified and depending on the author they can be: >1.6 μm (Jeff Obbard), <1 mm (Mark Browne), <2 mm (Peter Ryan), 2–6 mm (Jose Derraik), <5 mm (Michael Barnes, Kellyn Betts) [7], but recently the most common value is <5 mm. The lower limit of the microplastic size is not clearly defined, but usually, particles with dimensions <1 μm are called nanoplastics [8]. To clarify the definition, the report prepared by Verschoor [9], in which the researcher proposes the following criteria defining as a microplastic particle: synthetic material with a high content of polymers, solid particles, <5 mm, insoluble in water and not degradable.

Considering the origin of the molecules, that is, how they get into the environment, two main groups can be distinguished: primary microplastics and secondary microplastics. Primary microplastics are particles that gain dimensions <5 mm already at the stage of their production [7]. Within this group, two main forms of particles can be distinguished, including microgranules and pellets [10]. Microgranules are used in cosmetic products, such as, for example, body and face washing gels, toothpaste, or peels [11]. The second group, that is, secondary microplastics are particles that reach their microscopic dimensions due to the disintegration of larger plastic elements in the environment. Disintegration can occur both on the surface of the land and in water, and they are responsible for various physical and chemical factors. The different origins of the groups mean that they can be distinguished by a separate structure. Primary microplastic particles have regular, smooth edges, most often take an oval or spherical form, unlike secondary microplastic particles, which are much more diverse in shapes, colors, and sizes, and have irregular and jagged edges [12].

It should be noted that the primary microplastic is only a small fraction of the total amount of microplastic in the environment [13], and its further emission can be relatively easily referred to and reduced. A much bigger problem is particles coming from uncontrolled decays in the environment, which at the same time constitute the vast majority of pollutants. Over 50% of plastics contain hazardous monomers, fillers, and chemicals [11]. The whole set of materials, such as PS, PVC, and PC release toxic monomers that are identified with cancer and reproductive abnormalities [7]. Some auxiliary substances, for example, dyes or flame retardants, can accumulate in the human body, while some of them are endocrine-active compounds, that is, those affecting the functioning of the endocrine system [14]. Plastic components such as phthalates, bisphenol A (BPA), and triclosan are need of great concern [15,7]. BPA, that is, the main monomer used in the production of polycarbonate, can cause changes in liver function, changes in insulin resistance, fetal development disorders, brain function, and cause cardiovascular disease [7]. Some plasticizers, stabilizers, and dyes contained in plastics are produced by heavy metals such as chromium, cadmium, and lead [14].

Some researchers distinguish the third – separate – category of microplastics – synthetic fibers [10].

1.2. Removal of microplastics in wastewater treatment plants

Research shows that wastewater treatment plants (WWTPs) are one of the important sources of microplastics discharged into the environment [5,7,10,11,14,16–22]. This phenomenon takes place even though WWTPs can effectively remove a significant number of microplastic fragments present in the influent. For example, the levels of microplastic removal noted by researchers in some WWTPs amounted to 98.41% after the second stage treatment in the Scottish sewage treatment plant, over 95% in the American treatment plant using a three-stage treatment process and 99% in the Swedish treatment plant with PE equal to 45,000. In the Canadian treatment plant serving Vancouver, the percentage of microplastic removal was 99% [7]. Despite such high removal rates, due to the very large amounts of wastewater flowing through the treatment plants, the amounts of microplastic in effluents are still significant. Researchers have recorded quantities such as 4.9 fibers and 8.6 particles/L, 1,770 particles/h or 65 million particles/d at various facilities around the world [11]. The previously mentioned Canadian sewage treatment plant, despite 99% efficiency of microplastic removal, annually releases about 30 billion particles of these pollutants into the environment [7].

There are also treatment plants that do not achieve such high removal efficiency. One of the objects cited by Novotna et al. [23] showed MP removability at the level of only 72%. The different removability on different objects is mainly caused by different technological processes used in the treatment plants. Some researchers believe that relatively large quantities of microplastics are removed already in the first stage of treatment (also called mechanical treatment), but this may be because in their work they focused on larger impurities (>250 μm) [23]. Ziajahromi et al. [16] measured the content of microplastics after the first, second, and possibly the third stage of treatment on various objects. In the WWTP, where aeration, sedimentation, and UV disinfection were used as the second stage, the effluent after the second stage contained 66% fewer microplastics than those after the first one [16].

An even greater increase in effectiveness between the first and last stage (90%) was obtained in the treatment plant using 3-stage treatment technology, which consisted of biological treatment, flocculation, disinfection/dechlorination, ultrafiltration, reverse osmosis, and decarbonization [16]. The effectiveness of membrane processes and some processes used in the third stage of treatment was demonstrated by Talvitie et al. [17] and in these tests, a membrane bioreactor (post-treatment effluent after the first treatment stage), disc filter, fast sand filter, and flotator (serving as the third treatment stage) were tested separately. The removal rate of microplastics from effluent greater than 95% has been achieved by using each of these technologies.

The data on quantification and qualification of microplastics in WWTPs worldwide are still incomplete and there is a need to make more research in this area. The present research aimed to evaluate quantity and quality as well as estimate the removal efficiency of microplastics in a large and medium WWTP. Also loads of these pollutants discharged into the water environment were estimated.

2. Materials and methods

The tests were carried out on 2 WWTPs of the city in Silesian Agglomeration (Poland). The average daily load of WWTP A is approximately 45,000 m^3/d (municipal wastewater, PE equal to 410,000), while WWTP B approximately 1,500 m^3/d (up to a maximum of 3,400 m^3/d and PE equal to 17,000). There is a mixed sewerage system in the WWTP A catchment area (about 150 km^2) – the new sewage system is mainly distributive, while the old ones – combined. WWTP B is used to treat urban wastewater, but industrial wastewater from industrial plants located in the catchment area (about 15 km^2) also inflows the treatment plant. All sewage is collected in the collective sanitary sewage system.

The flow diagrams of the treatment plants are presented in Fig. 1. Both WWTPs are mechanical–biological installations.

The research consisted of the quantification of microplastics at several different characteristic points on various treatment stages of both WWTPs. At WWTP A, samples were taken: at the WWTP influent, below the grid chamber, in the primary sedimentation tank, in the secondary sedimentation tank, at the WWTP effluent. At WWTP B, due to the difficulties with access to some devices (and consequently also great difficulties with sampling), samples from the WWTPs influent were not collected. Difficult sampling associated with clogging of the plankton network with sediment occurred also in the primary sedimentation tank. Because of this sampling points at WWTP B were: the grit chamber, secondary sedimentation tank, and WWTP effluent.

Simultaneous measurement series were carried out in both WWTPs in the spring and summer of 2019.

50 L of wastewater samples were taken at both WWTPs at the abovementioned sampling points. The wastewater samples were then passed through the plankton net (0.25 mm mesh size). To clarify the sample and decompose organic matter, hydrogen peroxide (30%, 25 cm^3/sample) and iron sulfate (1 g/sample) were added to the samples (iron acts as a catalyst here) and heated at a temperature not exceeding the boiling point of water. The heating process was conducted at 70°C. After heating to identify microplastic particles, the samples were examined under a Delta Optical SZH – 650 B/T microscope. Thanks to the use of a camera coupled with a microscope, the observations were carried out on a computer screen using the ScopeImage 9.0 program. The performed activities consisted of counting microplastic particles, assisting at the same time with a scale in Thom's chamber.

3. Results

Three main groups of microplastics were analyzed in the study:

- foil fragments – plastic waste visually visible also to the naked eye (Fig. 2), originating from shredded plastic bags, carrier bags, etc. These fragments were of different colors and were characterized by jagged edges. During the research, it was found that these are the largest fractions of microplastics, occurring in wastewater with a size of 1–5 mm (Fig. 3).
- fibers – oblong, threadlike (Fig. 4). Their occurrence in wastewater is related to the fact that individual fabric fibers go to the effluent from washing machines, which then go along with domestic wastewater to the WWTP.
- granules – round or elliptical. They come mainly from toothpaste, peels, and other cosmetic products [3].

The tests were carried out for the quantitative determination of microplastics contained in wastewater and its grouping due to the form in which they occur. At each point where the tests were carried out, micropollutants were observed in various amounts and forms. Table 1 summarizes the test results for samples of sewage samples taken from WWTP A.

Table 2 summarizes the test results for samples taken from WWTP B.

Average concentrations of microplastics with SD values, as well as percent shares of particles of various shapes, are presented in Figs. 5 and 6. Microplastics in various forms were found in the wastewater from both treatment plants – both larger (microfoils) and smaller (microfibers).

Compared to the literature data, these are the amounts within the lower limit of standards detected in other treatment plants, much smaller than in case of the tested Scandinavian treatment plants in Finland and Sweden [19,24], where the highest results were recorded at around 15,000–20,000 particles/m^3 wastewater, therefore at least 50% more than in our research. However, it should be emphasized that in various plants concentration of microplastics may vary in time and it is difficult to point out the expected values for influent. Based on literature reports [25], it can be stated that, for example, adverse weather conditions may have an impact on the occurrence of microplastics in the environment, and probably also wastewater – after passing through violent storms a few or even several-fold increase in the concentration of microplastics in the area covered by the storm was found. These fragments could be leached to combined sewer systems.

The concentration of microplastics at the beginning of the treatment process of WWTP A and WWTP was statistically different. Higher concentrations of these pollutants

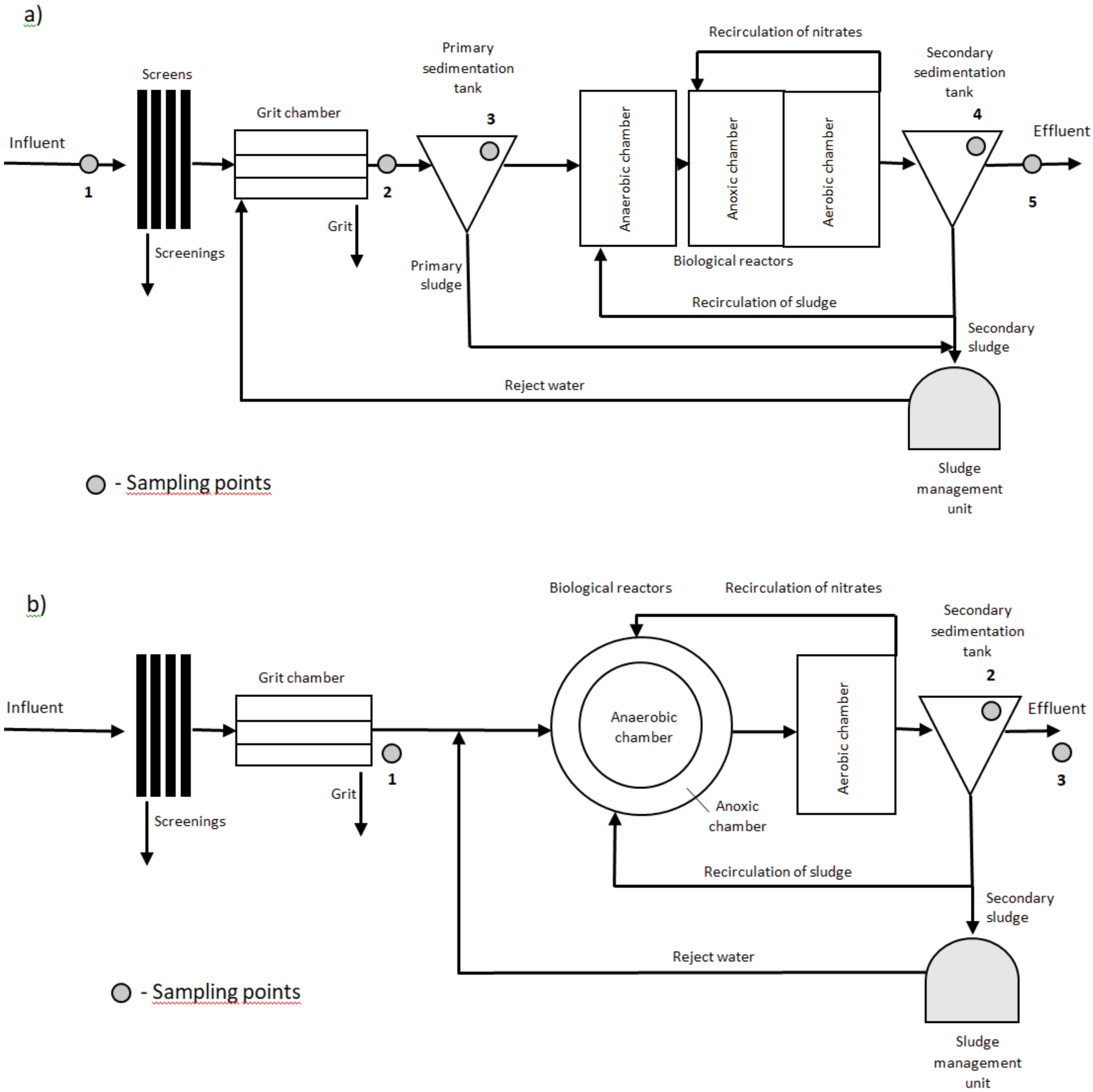

Fig. 1. Flow diagrams of WWTPs with sampling points (a) WWTP A and (b) WWTP B.

have been observed in smaller WWTP (inflow 1,500 m^3/d) than in the bigger one (inflow 45,000 m^3/d). It supports the thesis that there is no pattern for the expected concentration of microplastics in raw wastewater. A similar result has been obtained by us previously for WWTPs in Silesia region [26].

The majority of microfibers are noticeable in both treatment plants, as well as the negligible presence of microgranules (Figs. 7 and 8). The results were the most comparable to the WWTP at the Danube River in Germany [27–29] – similar values were noted for both foil fragments (100–2,000 in 1 m^3) and microfibers (100–4,800/m^3 wastewater). It is also noticeable that the vast majority of plastic microparticles are microfibers – in some stages of treatment (secondary sedimentation tank and effluent from WWTP B) they constitute up to 100% of their presence in wastewater. According to the research of Magnusson et al. [24] and Taltivie et al. [30], the majority of microfibers in wastewater are the norm – in their research, fibers accounted for 68%–71% of the total microplastics flowing into the treatment plant.

In case both of WWTP A and WWTP B, there was a significant percent share of foil particles after the biological treatment. Domination of the foils in secondary sedimentation tank and effluent could be caused by the fact that foil fragment floating on the surface of the wastewater and do not aggregate such easily as fibers. Fibers more easily are built-in sludge flocs, and therefore they are effectively removed from the water phase. Then, the problem with their presence in sewage sludge occurs [24].

Microfoils are primarily crushed plastic bags, which is a large number of macroplastics were found in the influent to the treatment plant (they confirm the results of tests also carried out at treatment plants in the Silesian

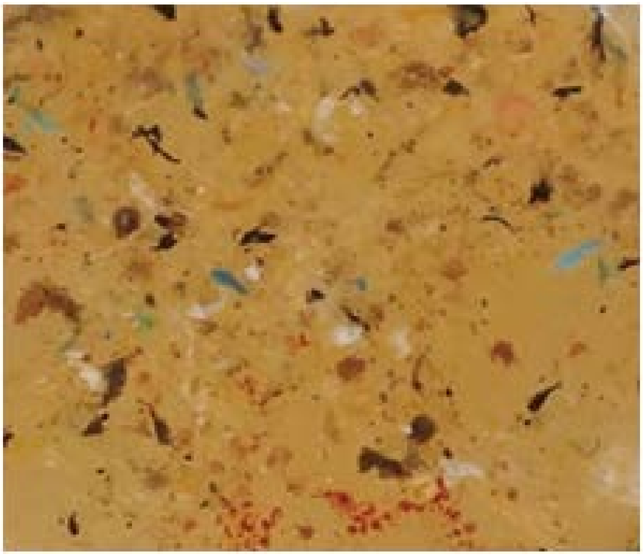

Fig. 2. Visible different colored elements of the microplastic in wastewater samples from WWTP (photo: M. Zyguła).

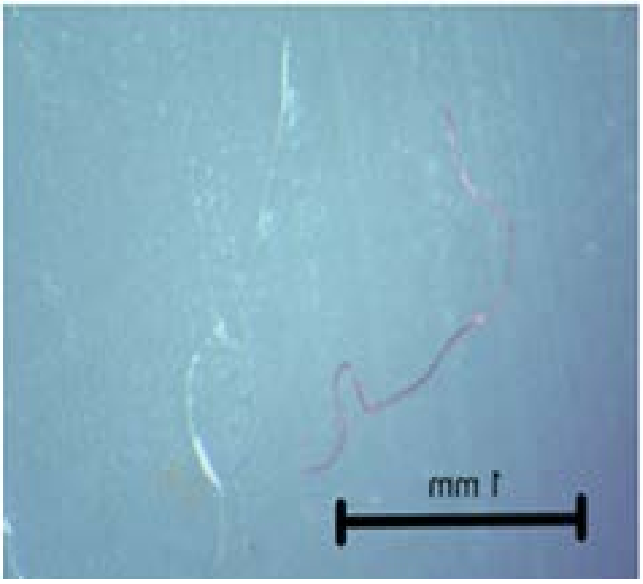

Fig. 4. Microfibers found in wastewater (photo: M. Zyguła).

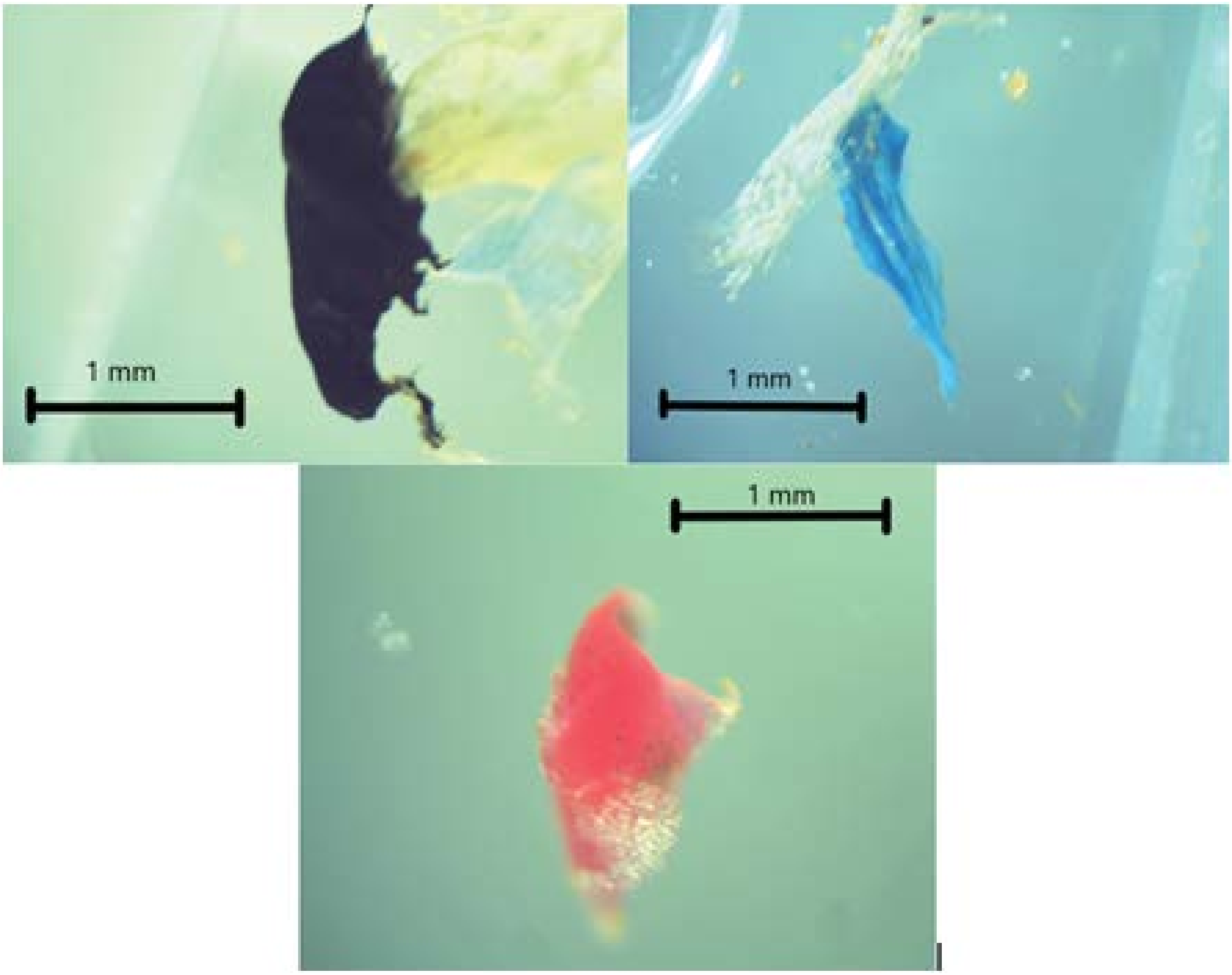

Fig. 3. Microfoils found in wastewater (photo: M. Zyguła).

Table 1
Microplastic in samples taken from WWTP A

Place of sampling	Particles in 1 m³	Total microplastics in 1 m³
WWTP influent	Microfibers – 1,900–2,060 Microfoils – 560–600 Microgranules – 240–480	2,700–3,140
Grit chamber	microfibers – 980–1,100 microfoils – 1,060–1,120 Microgranules – 40–60	2,140–2,220
Primary sedimentation tank	Microfibers – 4,000–10,000 Microfoils – 560–700 Microgranules – 0	4,700–10,560
Secondary sedimentation tank	Microfibers – 600 Microfoils – 800–1,300 Microgranules – 20–60	1,420–1,900
WWTP effluent	Microfibers – 40–60 Microfoils – 600–720 Microgranules – 0	640–780

Table 2
Microplastic in samples taken from WWTP B

Place of sampling	Particles in 1 m³	Total microplastics in 1 m³
Grit chamber	Microfibres – 4,400–7,000 Microfoils – 200–300 Microgranules – 0	4,700–7,200
Secondary sedimentation tank	Microfibres – 800–1,000 Microfoils – 0 Microgranules – 0	800–1,000
WWTP effluent	Microfibres – 400–600 Microfoils – 0–100 Microgranules – 0	500–600

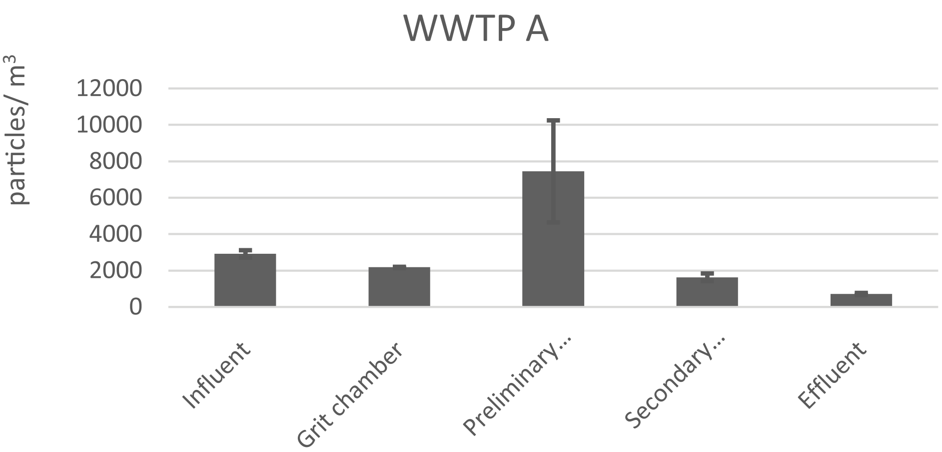

Fig. 5. Average concentrations with SD values of total microplastics in WWTP A.

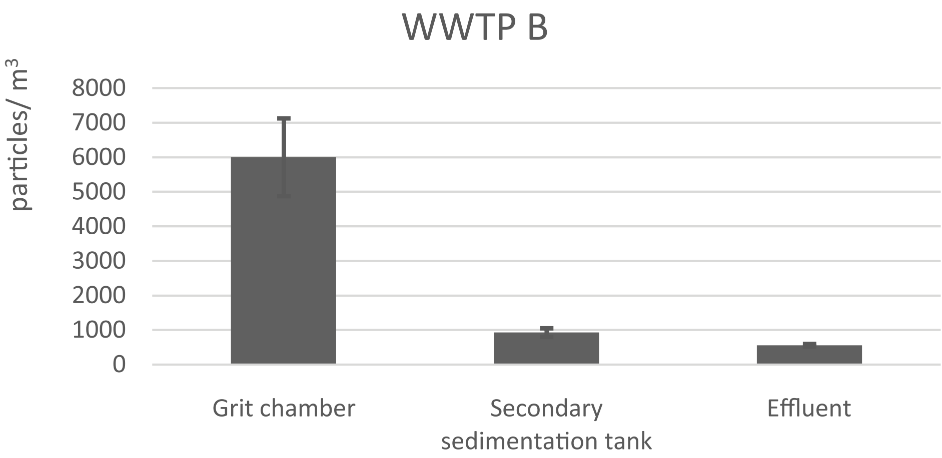

Fig. 6. Average concentrations with SD values of total microplastics in WWTP B.

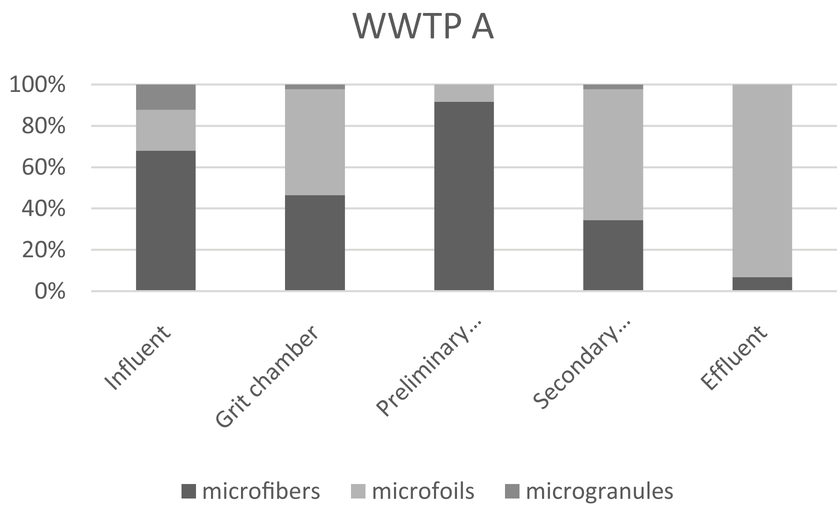

Fig. 7. Percent shares of various shape microplastic particles at different stages of wastewater treatment in WWTP A.

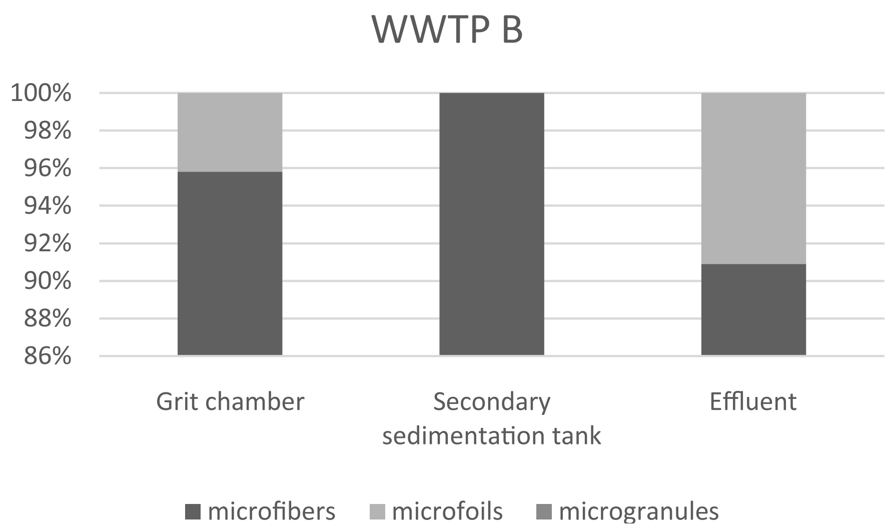

Fig. 8. Percent shares of various shape microplastic particles at different stages of wastewater treatment in WWTP B.

agglomeration [26]). The increased presence of foil particles is crushed in the mechanical part of the treatment (especially on the pumps) and further flowing with wastewater already occurs in micrometer sizes, thus belonging to the microplastic. This phenomenon is confirmed in the literature – waste on a macro scale is crushed, at some point reaching micrometric dimensions – then becoming so-called secondary microplastics [18]. Remarkably, a large and efficient WWTP does not cope well with the removal of the largest microplastic fractions, without having a problem with the effective removal of much smaller microfibers and microgranules. The more, according to research, the efficiency of removing microparticles of plastic increases with the increase in particle size – fractions larger than 1 mm are removed with an efficiency of over 90% in a conventional WWTP, while smaller particles are not removed so effectively and often require additional techniques [21,22]. This phenomenon is connected with all shapes of microplastics, however, microfoils, the largest of the examined microplastics, could be not removed relatively easily because they tend to float at the surface of wastewater, as mentioned above.

Based on the test results obtained in the study, the effectiveness of microplastic removal in the wastewater treatment process was also estimated.

Table 3 summarizes the amounts of microplastic particles by type and percentage removal in the wastewater treatment process at WWTP A. For reliable comparison, the results obtained in the samples after the grit chamber and from the effluent, that is, the first and last point from which sampling was possible from both tested WWTPs.

It was found that WWTP A copes best with removing some of the granules. Comparing the amount of all identified microparticles, it can be stated that the granules were the least numerous group occurring in the wastewater of WWTP A – 40–60 pieces/1 m^3 of wastewater in the grit chamber samples, while there were many more fibers and foils. In the samples taken from the WWTP A effluent microgranules weren't found, which means 100% removal of this type of microplastic in the wastewater treatment process. Over 90% removal efficiency was noted in the case of microfibers. In contrast, the removal of microfoils did not exceed 50%, thus departing from the results of other studies, which indicate the effectiveness of removal in the range of 50%–90% [19,24]. The general removal of microplastics in wastewater from WWTP A was 65%–70%. The result could be much higher were it not for significant amounts of microfilms in the tested wastewater.

Table 4 presents data for WWTP B – as above – numerical values and the percentage removal of microplastics including their type. The results obtained in the grit chamber and from the effluent were compared, that is, the first and last points from which it was possible to collect samples from both tested WWTPs.

WWTP B was more effective at removing microplastics from wastewater than WWTP A. Significant is the high degree of removal of microparticles of fibers and foils compared to the results obtained at the WWTP A. The total removal of microplastics at the WWTP B fluctuates around 90% and this result is comparable with the data available in the literature from Germany or Sweden, where the results of microplastic removal were 96% and 97%, respectively [24,27,28].

Based on the concentrations of microplastics in effluents also loads of these pollutants can be estimated. In the case of WWTP A, the load can be estimated at the level of 11×10^9 particles/y, in the case of WWTP B 30×10^7 particles/y.

4. Summary and conclusions

Pollution of the environment with microplastics discharged with effluents seems to be a neglected problem. Both in Poland and the world there are no legal regulations concerning the allowable amount of microplastics in treated wastewater.

Table 3
Removal of microplastics during the wastewater treatment process in WWTP A

Type of microplastics	Grit chamber (particles/m^3)	Effluent (particles/m^3)	Removal of microplastics (%)
Fibres	980–1,100	40–60	94.5–96
Foils	1,060–1,120	600–720	32–46.5
Granules	40–60	0	100
Total	2,140–2,200	640–780	65–70

Table 4
Removal of microplastics during the wastewater treatment process in WWTP B

Type of microplastics	Grit chamber (particles/m^3)	Effluent (particles/m^3)	Removal of microplastics (%)
Fibres	4,400–7,000	400–600	90.9–91.5
Foils	200–300	0–100	66.7–100
Granules	Not detected	Not detected	–
TOTAL	4,700–7,200	500–600	89.4–91.7

Discharge of treated wastewater from treatment plants is one of the reasons for the occurrence and spread of plastic microparticles in the water environment.

The real problem associated with the occurrence of microplastics in WWTPs is the fact that microparticles, even if they are removed from wastewater, are still deposited in sewage sludge and then during the sludge management process, microplastics can go to the environment again. The microplastic particles present in the stored wastewater sludge and then used as fertilizer in agriculture may end up with surface runoff into water reservoirs, followed by seas and oceans. The available literature shows that many microplastic particles, such as polyethylene or polypropylene, do not undergo biochemical degradation processes at all, so it is not surprising that biological treatment did not fully remove microfoils and microfibers from wastewater. In the case of microgranules, the highest amount was recorded at the influent to the treatment plant, while they were removed already at the initial stage of wastewater treatment. To improve the efficiency of removing microplastics from wastewater, it is recommended to use the third stage of purification, for example, filtration or the use of membrane reactors.

It is also suggested that to limit the process of microcontaminants formation in the WWTP, the wastewater sludge should be subjected to prior thermal treatment allowing the breakdown of microparticles and then their easier degradation.

Despite general considerations above the results obtained during the study permit the following conclusions:

- The removal efficiency of total microplastics in large WWTP A was 65%–70%, in the smaller one WWTP B it was 89%–92%.
- The lower removal efficiency of microplastics in WWTP A was connected with a higher abundance of microfoils in the effluent. The removal of microfoils was lower than 50% and as a result, decreased the total efficiency.
- Both WWTPs showed high efficiency in the removal of fibers and microgranules from influents, in WWTP A it was over 94% and in WWTP B over 90%.
- Both tested WWTPs did not contribute to the introduction of microgranules into the environment within the period of the studies, and very well deal with the removal of this type of micro-pollution.

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