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Original Research Article

OCCURRENCE AND CHARACTERISTICS OF MICROPLASTICS IN LEACHATE AT A LARGE MUNICIPAL WASTEWATER TREATMENT PLANT

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A b s t r a c t

Due to the widespread occurrence and the potential threat to the environment, plastic materials are currently a growing problem of environmental protection in the world. Plastics whit dimensions not exceeding 5 mm are called microplastics. One of the main sources of microplastics in the aquatic environment are municipal wastewater treatment plants. The paper presents the results of research on the presence of microplastics in leachate from sludge processing at a large municipal wastewater treatment plant. The leachate was divided into the leachate produced in the processes of sludge thickening and dewatering. The analysis of the isolated microplastics included a physical analysis, which focuses on determining size, shape, and color of the isolated material. The next step was the chemical identification of the microplastic, where the type of polymer of the tested material was confirmed by means of Attenuated Total Reflection Fourier Transform Infrared. Among the isolated microplastic particles, almost a half was identified as fragments, and a smaller amount was confirmed for the presence of foil, fibers, foams and granules. The identified particles were plastic materials, including polypropylene, polyethylene, polystyrene or poly(terephthalate). Based on the results of the conducted research, the mass of microplastics in leachate was characterized and determined.

Keywords: microplastic, wastewater treatment plant, leachate from wastewater treatment plant

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1. INTRODUCTION

Due to the enormous social benefits of plastics, world plastics production has grown from 2 million tons in 1950 to 335 million tons in 2016. In 2017, the number of plastics produced reached 349 million tons, and currently (2020) has been produced 367 million tons of plastics [1, 19]. Plastic waste in the environment has been classified according to its size. There are macro- (more than 25 mm), meso- (between 5 to 25 mm), micro- (between 1 μm to 5 mm) and nanoplastic (less than 1 μm) [5]. However, taking into account the proposed classification of nanoplastic by the European Union, nanoplastic particles are defined as particles with a size of up to 100 nm [9].

Small fragments or particles with a size of less than 5 mm, are name microplastics (MPs). Due to the source of origin MPs are divided into primary and secondary. Primary MPs is produced in microscopic sizes and can be found in domestic products such as facial cleansers, body scrubs, or powder [11]. The presence of primary MPs has also been confirmed in medical products and pharmaceuticals [7]. Secondary MPs arise from breakdown of larger pieces of plastic. The breakdowns are caused by many processes, including physical, chemical, and biological processes [15]. The constant degradation of primary and secondary microplastics causes changes in their properties (e.g. color, morphology, shape) and may additionally affect their chemical and physical properties [22].

The presence of MPs has been confirmed in rivers, air, soils, and other environmental media [3, 6, 10, 18]. The occurrence of MPs has also been confirmed in wastewater treatment plants (WWTPs). It is estimated to be one of the main sources of environmental pollution with MPs [11, 13]. The global number of MPs in sewage sludge ranges from 7.91 to 496×10^3 particles/kg dry solids (DS) [8]. Other sources say that the amount of MPs in sewage sludge ranges from 4.40×10^3 to 2.40×10^5 particles/kg DS. The amount of MP in raw wastewater varies from 0.28 to 3.14×10^3 particles/L, when in treated wastewater this range is reduced to the level of 0.01-2.97×10^2 particles/L [16]. Some research shows that wastewater treatment plants are unable to completely remove plastics from wastewater. Moreover, large amounts of MPs are emitted to the environment by both treated wastewater and sewage sludge applied to soils. WWTPs remove from about 40 to 99.99% of MPs, depending on the technology used. It is estimated that from 63 to 98% of MPs is removed in the primary treatment and from 7 to 20% in the secondary treatment [23].

This study was carried out to determine the presence of MPs in leachate from sewage sludge treatment processes in a municipal WWTP. The characteristics of the isolated MPs include the determination of their quantity and mass, as well as their morphological and chemical characteristics.

2. METHODOLOGY

2.1. Study side

Samples of leachate from the treatment of sewage sludge come from one of the large municipal mechanical-biological WWTP in southern Poland. The leachate was divided into leachate produced in the process of thickening of excessive sludge and in the process of dewatering of digested sludge. The samples were collected from July to September 2022.

2.2. Microplastics separation

First, a sample was filtered through metal sieves 5 mm and 45 μm in diameter. The fraction found on the 5 mm sieve was discarded. The sieve fraction with a mesh size of 45 μm was poured several times with distilled water and further processed.

Fraction collected from the sieve was processed according to the typical methods presented in the literature [14, 17, 20, 21]. The fraction was dried at 90°C for 24 hours. In order to separate the MPs from the environmental samples, the interfering matter was removed. The organic matter was removed using the Fenton reaction (30% H_2O_2 and 0.05M $FeSO_4$). The mixture was heated to 75°C and mixed for 30 min. This process was repeated until the organic matter was completely removed. In order to isolate inorganic matter from MP, density separation was used with the use of an aqueous NaI solution with a density of about 1.6 g/cm^3. The samples with a saturated solution were mixed and leave for 24 h. The top layer with floating pieces has been filtered through a cellulose filter. Filter residue with deionized water. The isolated material was dried at 90°C and subjected to physical and chemical characteristics.

2.3. Characteristics of microplastics

The particle morphology was initially examined using stereomicroscope (Leica EZ4 W ×35). With its help, the tested material was classified in terms of shape, color, and particle size. Attenuated Total Reflection Fourier Transform Infrared (FTIR-ATR) spectroscopy was used to record IR absorption spectra of MPs and to identify functional groups specific to them, and thus making it possible to confirm the type of MP (FTIR, PerkinElmer Spectrum Two). The FTIR spectra were recorded in the wavenumber range of 450–4000 cm^{-1} and averaged 64 scans per spectrum at room temperature, with attenuated total reflection (ATR) accessory.

3. RESULTS AND DISCUSSION

3.1. Leachate from thickening of excessive sludge

The average amount of MPs in the leachate from thickening of excessive sludge samples was 98.67±3.21 particles MP per liter of leachate. The amount of MP in the tested samples was by weight 0.55±0.21 mg MP per liter of leachate. The most frequently observed MP morphotypes were fragments (47%) and foils (24%), followed fibers (11%), foams (9%) and beads (8%) (Fig. 1A). Colorless particles constituted the largest percentage of MPs (36%). Then yellow (15%), red (14%), black and colored (11%), white (5%), blue (4%), brown (3%), and green (1%) (Fig. 1B).

The size distribution analysis indicated that the largest amount of MPs was in range 500-1000 µm (32%) and 250-500 µm (30%). MPs with a size greater than 1 mm are 23% of the total. The smallest isolated fraction was 15% (250-45 µm).

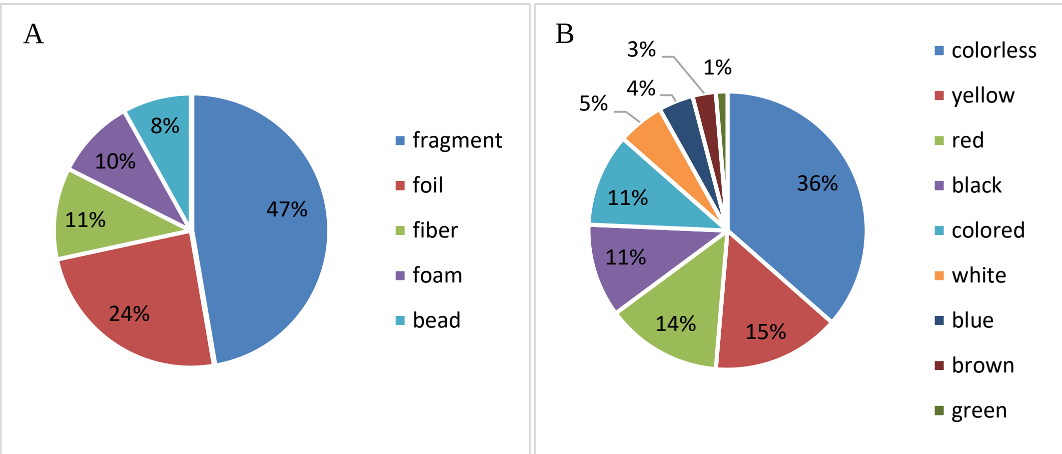

Fig. 1. Average percentages of different shape (A) and color (B) of MP particles in leachate from thickening of sewage sludge

Interpretation of the obtained FTIR-ATR spectra allowed to determine the type of polymer from which the isolated MP consist. Polyethylene (PE) constituted the highest percentage of all identified polymers (53%). The high content of PE may be due to the widespread use of PE plastics. The greatest demand for plastics is estimated in the packaging segment, where PE is used the most [19]. Most likely, they were created as a result of the breakdown of larger plastics (e.g. food containers or food foils) into smaller ones. Then polypropylene (PP) (20%), elastomer/rubber (12%), polystyrene (PS) (8%), polyethylene terephthalate (PET) (3%), polyacrylate (3%), and polyisobutylene (PIB) (1%) were identified (Fig. 2).

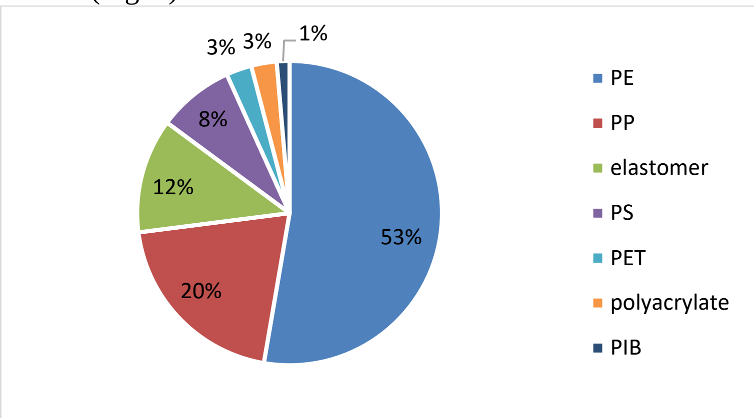

Fig. 2. Average percentages of MP particles depending on the type of polymer in leachate from thickening of sewage sludge

FTIR-ATR spectroscopy confirmed that the greatest number of fragment particles belonged to PE (46%). PE and other polymers identified as fragments most likely come breakdown of larger plastic particles. The most studies showed that the fragments of plastic were PE and PP, which was also confirmed in this work. PE is an easily formed and flexible material, as well as relatively cheap to produce. Due to its properties, it is the main material used in the production of foil, moreover, shopping bags and general packaging are made of it [2]. Probably due to the properties, use and widespread use of PE, most foil particles have been identified as PE (72%). Elastomers are group of plastics which include among others rubber. Rubber is a material from with many everyday objects are made, e.g. car tires, shoe soles, toys for children [12]. The widespread use of this material results in its high content in the form of foils (22%) and fragments (14%). Polyethylene fibers probably come from the decay of textile products. In fishing, sailing and climbing, polyethylene lines or ropes made of PE fibers are often used [2]. PP fibers (38% of all identified fibers) are widely used in construction. Due to its properties, it is increasingly used in many areas of life. FTIR analysis confirm that the most of foam particles belonged to PE (57%). Bead PS is estimated to be 33% of all identified bead. The disintegration of the foam composed of many pre-expanded PS bead results in a high concentration of this polymer in the environment [4] (Fig. 3).

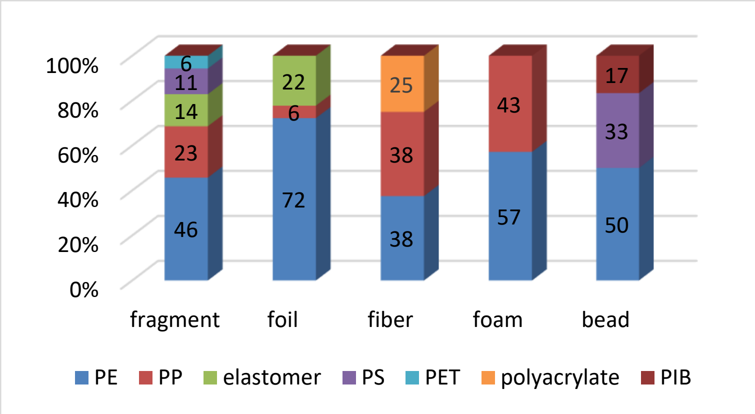

Fig. 3. Average percentages of the dependence of the type of polymer on the shape in leachate from thickening of sewage sludge

3.2. Leachate from dewatering of digested sludge

The average amount of MPs in the leachate from dewatering of digested sludge samples was 114.67±2.31 particles MP per liter of leachate (0.86±0.085 mg MP per liter of leachate). The highest content of fragments (32%) was found, followed by foil (31%), fibers (22%), foams (8%) and beads (7%) (Fig. 4A). The particle color analysis showed that most MP particles were colorless (34%). Then 20% of the particles of yellow color, 14% blue, 9% red, 8% black, 5% brown, 4% white, 4% colored, and only 1% green were identified (Fig. 4B).

Analysis of the fraction size showed that 39% of the MP particles were in the range from 5 mm to 1 mm. The particles whose size was in the range of 1000 µm to 500 µm was 23%. Then it was later determined that 22% of the particles ranged from 500 µm to 250 µm. The smallest fraction was 16% of the total (particles smaller than 250 µm).

Based on the obtained FTIR-ATR spectra, the type of polymers of all isolated MP particles was confirmed. PE (49%) was the highest amount of isolated MP. Then PET (19%), PP (18%), polyacrylonitrile (PAN) (5%), elastomer/rubber (5%), polyamide (nylon) (3%) and polyvinyl chloride (PVC) (1%) were interpreted (Fig. 5).

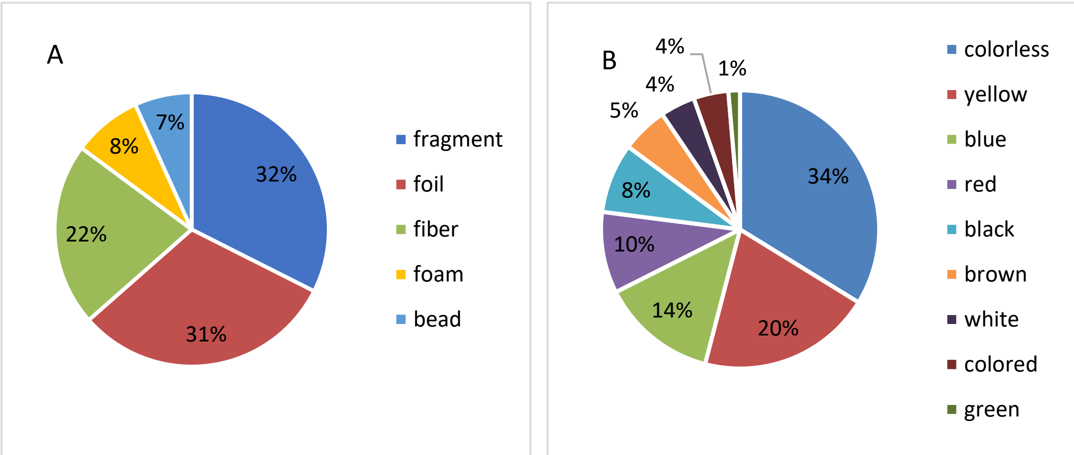

Fig. 4. Average percentages of different shape (A) and color (B) of MP particles in leachate from dewatering of sewage sludge

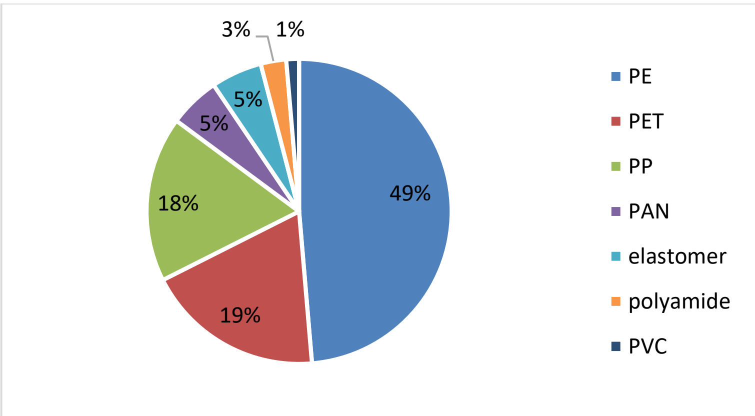

Fig. 5. Average percentage of MP particles depending on the type of polymer in leachate from dewatering of sewage sludge

The comparative analysis of the polymer type and shape shows that as much as 91% of the film particles were identified as PE. This is most likely due to the wide use of PE in the production of plastic films and bags [2]. Additionally, a large amount of PE may be caused by the low density of this polymer. Higher-density polymers are largely removed in the initial wastewater treatment processes. Particles of small size and low density get into further stages of wastewater treatment [23]. The most diverse group in terms of the types of polymers are fragments. The fragments come from the breakdown of larger plastics. The variety of polymers in the form of fragments is due to the large amount of use of plastics in every area of life. 69% of the fibers were identified as

PET polyester. Polyester fibers are used in the production of fabrics for outerwear or decorative fabrics, which can be confirmed by their high number [2]. 13% of all identified fibers are nylon fibers. Nylon is used in the production of ropes, cords, belts, fabrics, underwear and tights [2]. Over 60% of all foams were identified as polypropylene foams. The presence of PE (17%) and rubber (17%) foams was also confirmed. The content of granules in relation to all identified shapes was low. However, among them, 40% PAN, 40% PE and 20% PP were identified (Fig. 6).

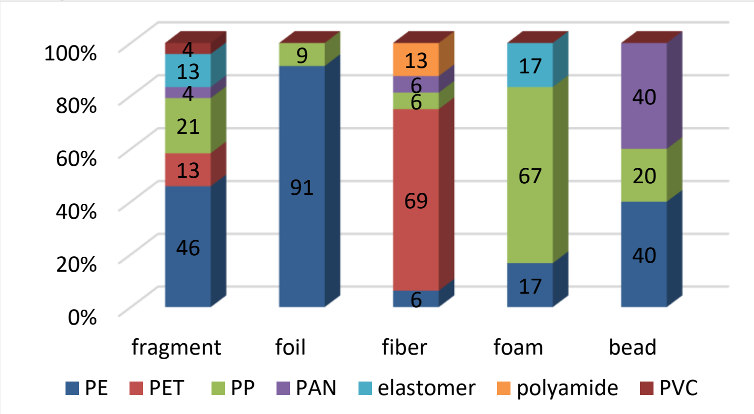

Fig. 6. Average percentages of the dependence of the type of polymer on the shape in leachate from dewatering of sewage sludge

Fig. 7 shows a representation of the isolated MPs due to their shape. The division takes into account the most frequently used system of dividing MPs into fragments, films, fibers, foams and beads.

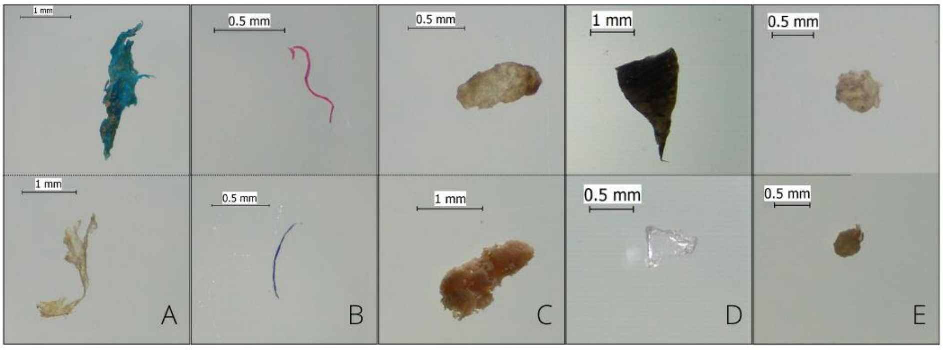

Fig. 7. Represented analysis of MPs with stereomicroscope: A – foil, B – fiber, C – foam, D – fragment, E – bead

The selected representative FTIR-ATR spectra are shown in Fig. 8. On the basis of the reference library, the polymers that are part of the isolated MP were confirmed. The percentage of the search result using the FTIR spectra reference library is shown in parentheses. The results were additionally confirmed by own analysis by confirming the range of occurrence of characteristic functional groups of individual polymers.

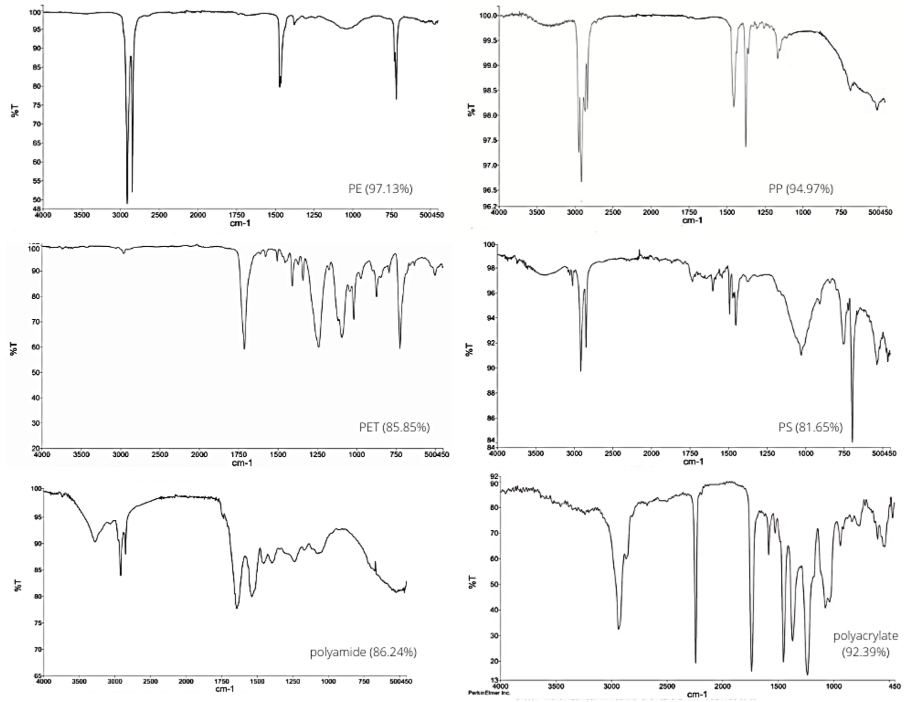

Fig. 3. Represented analysis of MPs with FTIR-ATR

4. SUMMARY AND CONCLUSIONS

This study focused on the content and characteristics of MP in leachate from thickening and dewatering sewage sludge processes. The widespread use of plastics in everyday life contributes to a large amount of MPs in WWTPs. The above analysis confirmed the occurrence of MP in leachate from the processing of sewage sludge in the municipal WWTP. The average amount of MPs in the leachate from thickening and dewatering of sewage sludge were 98.67±3.21 particles MP/L and 114.67±2.31 particles MP/L, respectively.

The morphology and type of polymer of isolated MPs were discussed and characterized. In both cases, it was noted that PE accounts for nearly 50% of all identified MPs. The low density of PE does not allow it to be removed in the initial wastewater treatment processes, therefore it is returned with the leachate back to the wastewater treatment process. In both cases, 5 basic types of MP shape were noticed. In the case of the leachate from thickening of sewage sludge, nearly 50% was identified as fragments. The analysis of leachate from the sludge dewatering processes confirmed the presence of almost the same number of fragments and foil.

The lack of standardization of MPs test methods makes it difficult to compare test results from different studies. Therefore, there is an urgent need to standardize the methodology for the detection of MPs in samples derived from WWTPs.

REFERENCES

1. Borrelle, SB et al. 2020. Predicted growth in plastic waste exceeds efforts to mitigate plastic pollution. *Science* **369** (6510), 1515-1518.
2. Charles, E and Carraher, J 2013. Introduction to polymer chemistry (3rd ed.). Boca Raton: CRC Press.
3. Choong, WS et al. 2021. Characterization of microplastics in the water and sediment of Bram River estuary, Borneo Island. *Marine Pollution Bulletin* **172**, 112880.
4. Crawford, CB and Quinn, B, 2017. Microplastic Pollutants. Oxford: Elsevier Inc.
5. da Costa, JP, Paço, A, Santos, PSM, Duarte, AC and Rocha-Santos, T 2018. Microplastics in soils: assessment, analytics and risks. *Environmental Chemistry* **16**, 18-30.
6. Ding, L, Mao, RF, Guo, X, Yang, X, Zhang, Q and Yang, C 2019. Microplastics in surface waters and sediments of the Wei River, in the northwest of China. *Science of The Total Environment* **667**, 427–434.
7. Duis, K and Coors, A 2016. Microplastics in the aquatic and terrestrial environment: sources (with a specific focus on personal care products), fate and effects. *Environmental Sciences Europe* **28**, 2.
8. El Hayany, B, Rumpel, C, Hafidi, M, El Fels, Loubna 2022. Occurrence, analysis of microplastics in sewage sludge and their fate during composting: A literature review. *Journal of Environmental Management* **317**, 115364.
9. EU Commission 2011. Commission recommendation of 18 October 2011 on the definition of nanomaterial (2011/696/EU). Official Journal of the European Union, no. L275 [verified 13.09.2022].
10. Goβmann, I, Süβmuth, R and Scholz-Böttcher, BM 2022. Plastic in the air?! – Spider webs as spatial and temporal mirror for microplastics including tire wear particles in urban air. *Science of The Total Environment* **832**, 155008.
11. Hamidian, AH, Ozumchelouei, EJ, Feizi, F, Wu, C, Zhang, Y and Yang, M 2021. A review on the characteristics of microplastics in wastewater treatment plants: A source for toxic chemicals. *Journal of Cleaner Production* **295**, 12658.

12. Harper, C 2002. Handbook of Plastics, Elastomers and Composites (4th ed.). McGraw-Hill Professional.
13. Kay, P, Hiscoe, R, Moberley, I, Bajic, L and McKenna, N 2018. Wastewater treatment plants as a source of microplastics in river catchments. *Environmental Science and Pollution Research* **25**, 20264-20267.
14. Lares, M, Ncibi, MC, Sillanpää, M and Sillanpää, M 2018. Occurrence, Identification and removal of microplastic particles and fibers in conventional activated sludge process and advanced MBR technology. *Water Research* **133**, 236–246.
15. Li, Y, Li, M, Li, Z, Yang, L, Liu, X 2019. Effects of particle size and solution chemistry on Triclosan sorption on polystyrene microplastic. *Chemosphere* **231**, 308-314.
16. Liu, W et al. 2021. A review of the removal of microplastics in global wastewater treatment plants: Characteristics and mechanisms. *Environmental International* **146**, 106277.
17. Lv, X, Dong, Q, Zuo, Z, Liu, Y, Huang, X, Wu, WM 2019. Microplastics in a municipal wastewater treatment plant: Fate, dynamic distribution, removal efficiencies, and control strategies. *Journal of Cleaner Production* **225**, 579–586.
18. Pastorino, P et al. 2021. First insights into plastic and microplastic occurrence in biotic and abiotic compartments, and snow from a high-mountain lake (Carnic Alps). *Chemosphere* **265**, 129121.
19. PlasticEurope (2021). Plastics – the Facts 2021. An analysis of European plastics production, demand and waste data, available at: https://plasticseurope.org/knowledge-hub/plastics-the-facts-2021/ [verified 13.09.2022].
20. Quinn, B, Murphy, F and Ewins, C 2016. Validation of density separation for the rapid recovery of microplastics from sediment. *Analytical Methods* **9**, 1491–1498.
21. Sun, J, Dai, X, Wang, Q, van Loosdrecht, MCM, Ni, BJ 2019. Microplastics in Wastewater Treatment Plants: Detection, Occurrence and Removal. *Water Research* **152**, 21–37.
22. Tirkey, A, Upadhyay, LSB 2021. Microplastics: An overview on separation, identification and characterization of microplastics. *Marine Pollution Bulletin***170**, 112604.
23. Turan, NB, Erkan, HS, Engine, GO 2021. Microplastics in wastewater treatment plants: Occurrence, fate and identification. *Process Safety and Environmental Protection* **146**, 77-84.

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