FIRST INDICATIVE ANALYSIS OF WWTP SLUDGE SAMPLES IN HUNGARY AND ITS ANALYTICAL CHALLENGES

Gábor BORDÓS, Bence Prikler, Zoltán Palotai

WESSLING Hungary Ltd. 1045 Budapest, Anonymus u. 6.

corresponding author: bordos.gabor@wessling.hu

Introduction & objective

The current tasks of the EU Strategy for the Danube Region (EUSDR) "Water Quality" Priority Area (PA4) include "Encouraging the monitoring, prevention and reduction of water pollution caused by hazardous and emerging substances", such as microplastics (MPs). Therefore in cooperation with the Ministry of Foreign Affairs and Trade (the governmental body in charge of the Hungarian national coordination of the EUSDR) the effectiveness of the removal of microplastics at some wastewater treatment plants (WWTPs) has been investigated. This is the **first study in Hungary that is chemically identifying MPs in sewage sludge** further to effluent and influent water.

Materials & methods

- 2 large WWTPs assessed (>50,000 equivalent person)
- 2 sampling day, 14 days between samplings
- 1 kg sludge collected per sampling
- Density separation in MPSS and 1.3 g/cm^3 $CaCl_2$
- H_2O_2 oxidation and filtration on 25 mm Anodisc
- Analysis with Thermo Fisher iN10MX, point & shoot

Results & discussion

In the past years a common understanding has been formed, that WWTPs are a source of MPs in the aquatic environment. Still, unified methodology of sample collection from WWTPS, sample preparation and analysis are lacking, which means, results of different studies are hardly comparable as shown in **Table 1**.

Table 1: Parameters and results of MP analysis in European wastewater treatment plants (sludge samples).

Country	Sample treatment	Sampled particles size range (µm)	Analysis method	Type of detected microplastics	Material type of detected microplastics	Analysed sample mass	Concentration (particle per kg)	Reference
Sweden	n.d.	300–5000	VIS, FTIR spectroscopy	Fibres, fragments	n.d.	n.d.	16 700	Wagner et al., 2014
Netherlands	separation (NaCl 1.2 g/cm^3)	10–5000	VIS, FTIR spectroscopy	Fibres	n.d.	20 g	510–760	Leslie et al., 2017
Germany	alkaline treatment; separation (NaCl 1.14 g/cm^3)	<500	VIS; ATR-FT-IR; FTIR microscopy	Fibres	PE, PS, PP; PA	125 g	1 000 – 24 000	Mintenig et al., 2017
Finland	dry	250–1000	FTIR microscopy and Raman spectroscopy	Fibres	PA, PE, PES, PET, PP	0.1 g	23 000	Lares et al., 2018
Poland	n.d.	109–5000	n.d.	n.d.	n.d.	n.d.	6 700 – 62 600	Iyare et al., 2020
Italy	separation (NaCl 1.2 g/cm^3); O	10–5000	VIS; FTIR spectroscopy	Fibres, fragments	silicone; PU; PS; PP; PE; PA; PTFE; polyacrylates	n.d.	113 000	Magni et al., 2018
Denmark	ED, O; separation (1.7 g/cm^3)	11-95	FTIR microscopy	n.d.	PE-co-polymer, PP, PA/Nylon	0.1 g	169 000	Vollertsen et al., 2017
Hungary	separation (NaCl); O	0,45-5000	VIS	Fibres, fragments, spheres	n.d.	400	5490	Parrag & Kátai, 2020

The **collected sample amount is influencing the representativity of the sampling**, and in most cases only a low mass is collected. We aimed to collect high mass to enhance representativity and eliminate upscaling of results derived from low mass samples. In some cases we experienced, that high mass sample can cause **filter cake formation and overlapping particles** (as shown on **Figure 1**), that complicates analysis. As recommendation for future analysis, **upper size limit can be introduced** with a filter mesh size e.g. 1 mm. The high sample mass could be processed only in the high volume preparation device (MPSS). Due to cost efficiency reason, $CaCl_2$ was used. This is not the most dense solution but more dense than the **commonly used low density solutions** (NaCl with ~1.2 g/cm^3) and seems to be a viable option for sample preparation. MPs in the samples are ranging between 3-94 particles/kg, that is much less than the previously reported values. One reason can be the lack of upscaling (as high mass was applied). Also, due to an array detector discrepancy, **only point & shoot method could be applied, and t**he number of analysed particles seems to show a correlation with the MP results, as indicated in **Figure 1**. Our results highlight the uncertainty issues related to point & shoot analysis method, so it is recommended to scan the whole filter area. **As sewage sludge might be used in the agriculture for nutrient supply, it is very important to set up a standardised monitoring system to evaluate the potential load of MPs in soil and the related risks.**

Figure 1: Prepared samples on 25 mm Anodisc filters (sample codes and number of analysed particles) and the results of the point & shoot analysis.

References

Iyare, P. U., Ouki, S. K., & Bond, T. (2020). Microplastics removal in wastewater treatment plants: a critical review. Environmental Science: Water Research & Technology, 6 (10), 2664–2675. https://doi.org/10.1039/d0ew00397b

Lares, M., Ncibi, M. C., Sillanpää, M., & 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. https://doi.org/10.1016/j.watres.2018.01.049

Leslie, H. A., Brandsma, S. H., van Velzen, M. J. M., & Vethaak, A. D. (2017): Microplastics en route: Field measurements in the Dutch river delta and Amsterdam canals, wastewater treatment plants, North Sea sediments and biota. Environment International, 101, 133–142. https://doi.org/10.1016/j.envint.2017.01.018

Magni, S., Binelli, A., Pittura, L., Avio, C. G., Della Torre, C., Parenti, C. C., Regoli, F. (2018): The fate of microplastics in an Italian Wastewater Treatment Plant. Science of The Total Environment. https://doi.org/10.1016/j.scitotenv.2018.10.2

Mintenig, S. M., Int-Veen, I., Löder, M. G. J., Primpke, S., & Gerdts, G. (2017): Identification of microplastic in effluents of waste water treatment plants using focal plane array-based micro-Fourier-transform infrared imaging. Water Research, 108, 365–372. https://doi.org/10.1016/j.watres.2016.11.015

Parrag, K.T., Kátai L. (2020): Szennyvizek mikroszannyező és mikroműanyag tartalma. Iparbiztonsági és hatósági napok. Conference paper. https://www.researchgate.net/publication/341399488

Vollertsen, J., Hansen A. A. (2017): Microplastic in Danish wastewater. Sources, occurrences and fate. The Danish Environmental Protection Agency.

Wagner, M., Scherer, C., Alvarez-Muñoz, D., Brennholt, N., Bourrain, X., Buchinger, S., Reifferscheid, G. (2014): Microplastics in freshwater ecosystems: what we know and what we need to know. Environmental Sciences Europe, 26 (1). https://doi.org/10.1186/s12302-014-0012-7

This publication is based upon work from COST Action CA20101 Plastics monitoRIng detectiOn RemedIaTion recoverY – PRIORITY, supported by COST (European Cooperation in Science and Technology). www.cost.eu

The project was implemented with the support of the Hungarian Ministry of Foreign Affairs and Trade within the framework of the Water Sector Tender Program. The contents of this document do not constitute the official position of the Ministry of Foreign Affairs and Trade.