IVM Institute for Environmental Studies

Microplastics in river suspended particulate matter and sewage treatment plants

S.H. Brandsma

P. Nijssen

M.J.M. van Velzen

H.A. Leslie

Report R14/02, version 1 14 November 2013

This report is released by: Prof. Dr. J. de Boer







This report was commissioned by: Mr. Frank Zijp Rijkswaterstaat Water, Verkeer en Leefomgeving

It was internally reviewed by: Bert van Hattum

IVM

Institute for Environmental Studies VU University Amsterdam De Boelelaan 1087 1081 HV AMSTERDAM The Netherlands T +31-20-598 9555 F +31-20-598 9553

E info.ivm@vu.nl

Mr. Bert Bellert
Rijkswaterstaat Water, Verkeer en
Leefomgeving
Postbus 17
8200 AA Lelystad
The Netherlands
T 06 - 53942815
E bert.bellert@rws.nl

Copyright © 2013, Institute for Environmental Studies

All rights reserved. No part of this publication may be reproduced, stored in a retrieval system or transmitted in any form or by any means, electronic, mechanical, photocopying, recording or otherwise without the prior written permission of the copyright holder

Contents

Abbre	eviations	5
Sumn	nary	7
1	Introduction	9
2	Sampling	11
2.1 2.2	Waste water treatment facilities Riverine suspended particulate matter	11 12
3	Analytical method	13
3.1 3.2	SPM and Sewage sludge Wastewater treatment plant influents and effluents	13 13
4	Results and discussion	15
4.1 4.2	Microplastics in SPM Microplastics in WWTPs	15 16
Refer	ences	19

Abbreviations

dw Dry weight

LIMS Laboratory Information Management System

NaCl Sodium chloride RWS Rijkswaterstaat

SPM Suspended particulate matter

WWTP Wastewater treatment plant



Summary

In this pilot study microplastics were analysed in suspended particulate matter (SPM) from the Maas River at Eijsden (NL) and two locations on the Rhine River, Lobith (NL) and Bimmen (DE). Additional microplastics analysis of influent, effluent and sewage sludge samples from wastewater treatment plants (WWTP) Amsterdam West, Amsterdam Westpoort and Heenvliet. In all samples microplastics were detected. The highest levels were found in the SPM from the Rhine River, location Lobith (average 4900 ± 540 particles/kg dw). The numbers of microplastics in SPM in the present study exceeded the dry-weight normalised numbers of microplastics observed in marine sediments from the Dutch coastal zones (Leslie et al. 2013).

Microplastic concentrations found in the effluent from Heenvliet and Amsterdam West (average 89 and 80 particles/kg ww, respectively) were comparable with the results of a previous study (average 48 ± 25 and 54 ± 42 particles/kg ww for Heenvliet and Amsterdam West, respectively) (Leslie et al. 2013). First indications were found that wastewater from industry (Westpoort) contained higher level of microplastic concentration than in wastewater from households (Amsterdam West and Heenvliet). However this indication is only based on one sample therefore more analyses would be needed to confirm this finding. Sewage sludge is rich in microplastic particles.

¹ Plastic particulates smaller than 5 mm in size.



1 Introduction

Microplastics are widespread in the Dutch marine environment (Leslie et al. 2011; 2012; 2013). Marine sediments tend to be rich in microplastics, as is sewage sludge. The treated effluents of sewage treatment plants are also known to contain microplastics. Rijkswaterstaat (RWS) contracted the Institute for Environmental Studies (IVM) of the VU University Amsterdam to perform analysis of microplastics in suspended particulate matter (SPM) from the Meuse and Rhine Rivers and in influent, effluent and sludge samples from three Dutch waste water treatment plants as part of a pilot study on microplastics in riverine systems and sewage treatment plants in the Netherlands.



2 Sampling

2.1 Waste water treatment facilities

Three WWTP locations were selected, Amsterdam West, Amsterdam Westpoort and Heenvliet. Amsterdam West and Heenvliet receive mainly household wastewater, while Amsterdam Westpoort receives mainly industrial wastewater (Table 2.1). Amsterdam Westpoort was added to the list of sampling locations in consultation with RWS contact person. The locations discharge directly to the North Sea Canal or to the Oude Maas River.

Table 2.1 Wastewater treatment facilities selected for sampling for microplastics analysis.

WWTP	Water body receiving effluents	Hydraulic capacity (m³/h)		
Amsterdam West	North Sea Canal	30000		
Amsterdam Westpoort	North Sea Canal	9000		
Heenvliet (conventional)	Oude Maas River	390		

An overview of all the samples for analysis is given in Table 2.2 below. Sample materials from the WWTPs in Amsterdam West, Amsterdam Westpoort and Heenvliet were collected by IVM. From Amsterdam West, sewage sludge and a 24-hour effluent sample were taken. Due to works at the facility, no influent sampling was possible during the project duration. At the Amsterdam Westpoort location, the influent sample was taken from the incoming wastewater at a discrete moment in time (not a 24-hour influent sample). In addition one 24-hour effluent sample and one sewage sludge sample were taken at Westpoort.

Table 2.2 Sample overview: wastewater influent, effluent and sewage sludge and river suspended particulate matter (SPM).

Sample type	N	Sampling date	Locations	description
24-hour effluent	1	06-11-2013	Amsterdam West	
Sewage sludge	1	06-11-2013	Amsterdam West	5 subsamples homogenized
* influent	1	06-11-2013	Westpoort	
24-hour effluent	1	06-11-2013	Westpoort	
Sewage sludge	1	06-11-2013	Westpoort	5 subsamples homogenized
24-hour influent	2	30-10-2013	Heenvliet	(duplicate)
24-hour effluent (conventional)	1	30-10-2013	Heenvliet	
Sewage sludge	2	30-10-2013	Heenvliet	5 subsamples homogenized (in duplicate)
River SPM	2	09-10-2013	Eijsden (Meuse)	(duplicate)
River SPM	2	08-10-2013	Lobith (Rhine)	(duplicate)
River SPM	2	07-10-2013	Bimmen (Rhine)	(duplicate)
Total samples	16			

^{* 24-}hour influent was not available at this location

At the Heenvliet WWTP samples of 24-hour effluent (n=2), sewage sludge (n=2) and 24-hour effluent (n=1) were taken. It should be noted that the membrane reactor effluent treatment is no longer in place at the Heenvliet facility, and conventional treatment is in place for all wastewater. All effluents in this study were conventionally treated. All WWTP effluents, influents and sewage sludge samples were collected in glass jars (ca. 2 liter for each sample) and stored in the dark at 4 °C until analysis. All samples were analysed before 13 November 2013.

2.2 Riverine suspended particulate matter

The river SPM was delivered to IVM by RWS. The SPM was delivered wet to avoid occlusion of microplastics. A total of three locations were sampled, representing the points of entry of the two main rivers in the Netherlands, the Rhine and the Meuse (Table 2.2). In Lobith (Rhine) 115 gram of SPM was collected, corresponding to 4595 liters of water; at the location Eijsden (Meuse) 393 gram of SPM was collected, corresponding to 24,956 liters of water; and at the location Bimmen (Rhine) 235 gram, corresponding to 7859 liters of water. The samples were stored in glass sample jars in the dark at 4 °C until analysis.

3 Analytical method

3.1 SPM and Sewage sludge

SPM samples were homogenized at the laboratory before taking subsamples for extraction and analysis and for dry weight determination so that concentrations could be expressed as number of particles per kg dry sample.

The five sewage sludge samples were homogenized before taking subsamples from each for extraction and analysis and for dry weight determination, so that concentrations could be expressed as number of particles per kg dry sample.

For the extraction of microplastics the method of Thompson et al. (2004) was followed, in a slightly adapted, miniaturized form. Briefly, 25 g of sample was added to an Erlenmeyer with pure analytical grade water (milliQ) to which NaCl had been added (saturated solution, 1.2 kg NaCl/L). The sample was allowed to settle to the bottom of the Erlenmeyer flask while the particles which were less dense than saturated salt solution were allowed to float at the top of the water layer. The top water layer was filtered over a $0.7\mu m$ Whatman glass filter (GF/F) and observed by light microscopy. The number of microplastic particles was counted and corrected for the low and stable blank (control chart mean value 2 microplastic particles per analysis; the blanks consistently consist of fibers only). The concentrations were expressed as number of particles per kg dry sample. Particles were classified into two broad size categories: particles between 1 and 300 μm and those between 300 and 5000 μm . (The latter corresponds to particle sizes commonly targeted in seawater surface microplastic sampling).

3.2 Wastewater treatment plant influents and effluents

The wastewater treatment plant influents and effluents were filtered similarly to the SPM and sewage sludge samples. The samples were well mixed immediately prior to taking aliquots from each effluent sample for extraction, since microplastics are not in solution and most can be assumed not to be neutrally buoyant.



4 Results and discussion

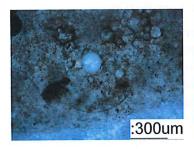
4.1 Microplastics in SPM

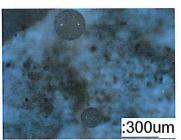
Microplastics were detected in all the SPM samples (Table 4.1). Procedural blanks, which are part of the quality control for background contamination during sample preparation, extraction and analysis, were low and stable; when calculating the total number of microplastics in a sample the background level was corrected for.

Table 4.1 Microplastic particle concentrations in SPM from two river locations in the Netherlands and one in Germany including average and standard deviation (s.d.)

IVM LIMS code	Location	Sample intake	Dry weight (%)	Fiber (n)	Foil (n)	Sphere (n)	Total particles (n)	% particles >300 μm	% particles <300 µm	Total particles per kg dry wt
13/0922A	Bimmen (Rhine)	24.23	46.05	20	0	2	22	63	37	1970
11/0922B	Bimmen (Rhine)	24.55		4	0	12	16	17	83	1420
	Average (s.d.)									1700 (390)
11/0923A	Eijsden (Meuse)	24.50	41.70	14	2	2	18	30	70	1760
11/0923B	Eijsden (Meuse)	23.26		7	3	0	10	50	50	1030
	Average (s.d.)									1400 (520)
12/0924A	Lobith (Rhine)	23.86	37.28	19	0	28	47	33	67	5280
12/0924B	Lobith (Rhine)	24.95		12	0	30	42	27	73	4520
	Average (s.d.)									4900 (540)

The highest numbers of particles were found in the Rhine River (Lobith) (average 4900 particles /kg dw). In the Meuse River (Eijsden) fewer of particles/kg dw were observed (average 1400 particles/kg dw), comparable with the number of particles in the Rhine at the Bimmen sampling location (1700 particles/kg dw). Most microplastics observed were in the size category <300 µm with the exception of one SPM samples from the Rhine (Bimmen 13/922A). The largest particle detected was not readily visible to the naked eye (fibres could be as long as a mm). Spheres, fibres and fragments were among the shapes detected (Fig 4.1). Spheres were dominant in three of the four SPM samples from the Rhine. In the Meuse the fibres were the most dominant shape (Table 4.1). On a dry weight basis, higher microplastic particle concentrations were observed in the SPM in this study compared to the marine sediments (100 - 3600 particles/kg dw) sampled on the Dutch coastal, offshore and estuarine locations (Leslie et al. 2013).





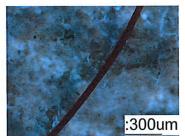


Figure 4.1 Examples of microplastics detected in SPM from the Rhine and Meuse Rivers.

4.2 Microplastics in WWTPs

Microplastics were detected in all influent, effluent and sewage sludge samples (Table 4.2). The analysis of duplicate influent samples from Heenvliet (13/1127 and 13/0971) resulted in 49 and 87 particles/ kg ww. The duplicate sewage sludge samples from Heenvliet (13/1132 and 13/1135) contained 370 and 950 particles/kg ww. Significant variation between the sludge samples from Heenvliet possibly suggests that not all sewage sludge samples are representative of the bulk (therefore multiple samples or pooling may be recommended).

Table 4.2 Measured concentrations of microplastics in WWTP influents, effluents and sewage sludge samples given per location.

IVM LIMS	Location	Sample	Sample intake (g)	Dry weight (%)	Fiber (n)	Foil (n)	Sphere (n)	Total particles (n)	% particles >300 µm	% particles <300 µm	Total particles per kg wet wt
13/1127	Heenvliet	24h- influent	103.63		9	0	0	9	45	55	87
13/0971	Heenvliet	24h- influent	101.17		5	0	0	5	71	29	49
	Average (s.d.)										68 (27)
13/1132	Heenvliet	Sewage sludge	21.57	0.32	8	0	0	8	70	30	370
13/1135	Heenvliet	Sewage sludge	19.96	0.32	19	0	0	19	62	38	950
	Average (s.d.)										660 (410)
13/0960	Heenvliet	24h- effluent	101.45		9	0	0	9	45	55	89
13/1193	Westpoort	**influent	10.95		10	0	0	10	67	33	910
13/1201	Westpoort	Sewage sludge	19.70	0.36	10	0	0	10	75	25	510
13/1184	Westpoort	24h- effluent	101.94		1	1	2	4	17	83	39
	Amsterdam	Sewage									
13/11/2	West	sludge	10.59	0.65	7	0	1	8	50	50	760
13/1169	Amsterdam West	24h- effluent	99.60		8	0	0	8	60	40	80

^{**24-}hour influent was not available at this location

The dry weights of the sewage sludge samples were all below 1% (Table 4.2) therefore the results are expressed in kg wet weight instead of dry weight. The concentrations of microplastics were high in the sewage sludge samples, indicating that microplastics are to a large degree retained in the sludge, as expected. In the 24h-influent and 24h-effluent samples from Heenvliet similar concentrations of microplastic were observed.

At the Westpoort WWTP the microplastics concentration in the influent was higher than in the sewage sludge and effluent from the same location. This may be explained by the fact that the influent was sampled at a single time point, which is less representative than a time integrated sample. This pilot study provides an initial basis for further investigations into the dynamics of microplastics in WWTPs.

Effluent samples from Heenvliet and Amsterdam West were analysed and reported by IVM previously (Leslie et al. 2013), with similar concentrations of microplastics in effluent from Heenvliet and Amsterdam West measured in October 2013 (average 89 and 80 particles/kg ww, respectively) and the previous winter (average 48 ± 25 and 54 ± 42 particles/ kg ww, respectively). Most microplastics counted were in the size category >300 μ m with the exception of 24h-effleunt form Westpoort (12/1184). This was also observed in the previous study of the same two household wastewater facilities (Leslie et al. 2013).

category >300 μ m with the exception of 24h-effleunt form Westpoort (12/1184). This was also observed in the previous study of the same two household wastewater facilities (Leslie et al. 2013).

Most of the particles detected in WWTPs were fibres, while microplastics in river SPM contained fibres but also a larger percentage of microspheres. Based on these preliminary data, it would be interesting to further explore if microplastic input into rivers is generally characterized by a greater assortment of plastic particle shapes than the microplastic input into wastewater.



References

- Leslie, H.A., Meulen, M.D. van der, Kleissens, F.M. & Vethaak, A.D. (2011). *Microplastic litter in the Dutch Marine Environment*. Deltares-IVM report 1203772-000. The Netherlands, 85 pp.
- Leslie, H.A., Moester, M., de Kreuk, M & Vethaak, A.D. (2012a). Exploratory study of the emission of microplastics from wastewater treatment plants. *H20*, (14/15), 45-47. In Dutch.
- Leslie, H.A. (2012b). *Microplastic in North Sea suspended particulate matter and cosmetics.* Institute for Environmental Studies (IVM), VU University, Report (W-12/01). In Dutch.
- Leslie, H.A., Velzen, M. van & Vethaak, A.D. (2013). *Microplastic survey of the Dutch environment: Novel data set of microplastics in North Sea sediments, treated wastewater effluents and marine biota*. IVM-VU University Report (R13-11), 27 pp.
- Thompson, R.C., Olsen, Y., Mitchell, R.P., Davis, A., Rowland, S.J., John, A.W.G., McGonigle, D. & Russell, A.E. (2004). Lost at sea: Where is all the plastic? *Science*, *304*, 838-838.

