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RESEARCH ARTICLE

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Microplastic sampling strategies in urban drainage systems for quantification of urban emissions based on transport pathways

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Abstract

Tracking waterborne microplastic (MP) in urban areas is a challenging task because of the various sources and transport pathways involved. Since MP occurs in low concentrations in most wastewater and stormwater streams, large sample volumes need to be captured, prepared, and carefully analyzed. The recent research in urban areas focused mainly on MP emissions at wastewater treatment plants (WWTPs), as obvious entry points into receiving waters. However, important transport pathways under wet-weather conditions are yet not been investigated thoroughly. In addition, the lack of comprehensive and comparable sampling strategies complicated the attempts for a deeper understanding of occurrence and sources. The goal of this paper is to (i) introduce and describe sampling strategies for MP at different locations in a municipal catchment area under dry and wet-weather conditions, (ii) quantify MP emissions from the entire catchment and two other smaller ones within the bigger catchment, and (iii) compare the emissions under dry and wet-weather conditions. WWTP has a high removal rate of MP (>96%), with an estimated emission rate of 189 kg/a or 0.94 g/[population equivalents (PEQ · a)], and polyethylene (PE) as the most abundant MP. The specific dry-weather emissions at a subcatchment were ≈30 g/(PEQ · a) higher than in the influent of WWTP with 23 g/(PEQ · a). Specific wet-weather emissions from large sub-catchment with higher traffic and population densities were 1952 g/(ha · a) higher than the emissions from smaller catchment (796 g/[ha · a]) with less population and traffic. The results suggest that wet-weather transport pathways are likely responsible for 2–4 times more MP emissions into receiving waters compared to dry-weather ones due to tire abrasion entered from streets through gullies. However, more investigations of wet-weather MP need to be carried out considering additional catchment attributes and storm event characteristics.

KEYWORDS

combined sewer system, large volume samplers (LVSs), microplastic pollution, monitoring methods, separate sewer system, size fraction, stormwater retention tank (SRT)

INTRODUCTION

The presence of microplastics (MP) in the environment has been reported since the 1960s when small plastic particles were found inside decaying carcasses of Albatrosses along Hawaiian beaches. [1] Since then and till the 2010s, the majority of studies focused on the occurrence in the marine environment. [2] However, sources of MP in any environment are linked to anthropogenic activities close to the marine environment (e.g., the fishing industry) and to activities within urban areas (e.g., littering).

According to the latest definition adopted by the international organization for standardization (ISO), MP is a collective term for solid and insoluble materials containing high polymers and trace amounts of additives that improve their properties with a size ranging from 1 to 1000 µm. [3] In addition, the previous definition for MP does not include elastomers (e.g., natural and synthetic rubber). [3] However, synthetic rubber is considered in this paper as MP due to its similar behavior of particle formation in MP size dimensions. [4]

Previous studies focused predominantly on wastewater treatment plants (WWTP) as a main entry pathway for MP into receiving waters. [5–12] These studies showed, nevertheless, that municipal WWTPs can capture the vast majority of MP entering the plants to the highest extent (>97%) regardless of weather conditions and catchment area. These studies adopted different sampling and detection methods and quantified MP in terms of number per area or sample volume, or as mass per sample volume, [13] starting from visual identification, stereomicroscopic methods, nondestructive (i.e., Raman spectroscopy) and destructive chemical characterization (i.e., thermoextraction/desorption-gas chromatography/mass spectrometry named TED-GC/MS) to remote sensing methods. [14–16] This made the comparison of available results more complex.

Urban drainage systems (UDS) have historically known two main approaches; combined and separate drainage systems. A combined drainage system drains stormwater runoff along with domestic and industrial wastewater using only one sewer network. While a separate drainage system drains stormwater runoff and wastewater streams in separate sewer networks. [17–19] Each drainage system represents a distinct entry pathway for different MP types, both quantitively and qualitatively, depending on the diffuse and point sources connected to it. Hence, both systems should be monitored separately to understand their significance in transporting MP of urban origin. As a definition, dry-weather days were the days when the cumulative precipitation depth did not exceed 0.1 mm on the day of sampling and the previous day. The same definition is applied to the calculation of base flow using the nightly-minimum method. [20]

A sampling of the influent of WWTP provides wide-ranging information on MP emissions and types from large catchment areas. Thus, it is challenging to estimate sources and release patterns of the different MP types detected in the catchment. Furthermore, the composition of dry-weather flow and, therefore, MP fractions contained therein depend on many area-specific influencing factors (e.g., the number of inhabitants and their water consumption behavior, buildings, and land use, the proportions and types of commerce and industry), so that the heterogeneity of the settlement area has probably a direct effect on release patterns of MP. In addition, temporally and spatially varying base flow amounts in the WWTP catchment have an influence on MP concentrations in wastewater.

As of yet, few studies focused on combined flow during wet-weather or stormwater runoff in urban catchment areas. [21, 22] According to the coalition clean Baltic, [23] however, 80%–95% of plastic litter ends up in the Baltic sea along with untreated street runoff from roads and parking lots. In addition, street runoff, as a transport medium for tire wear and other anthropogenic depositions, [24], is seen as the main cause of the deterioration of the quality of inland receiving waters. [25] Thus, reliable sampling strategies for MP need to be developed and tested to include wet-weather pathways in the catchment area.

The sampling of MP in wastewater media is particularly challenging and time-demanding since large sample volumes are needed to guarantee representative monitoring. Strong flow fluctuations, high organic content, and elevated risk of blockage due to large objects in the wastewater stream limit the technical feasibility of automatic sampling methods. Therefore, to monitor wastewater and stormwater runoff effectively, sampling methods should be designed to keep a balance between the effort to be applied and the reliability of these methods. In particular, sampling of combined flow during wet-weather conditions is challenging because of random occurrence and high flow dynamics.

The goal of this paper is to introduce and describe sampling strategies to monitor wastewater and stormwater-borne MP within a UDS and quantify the MP abundance of selected catchment areas under different weather conditions, considering the characteristics of these catchment areas in terms of land use and size. For combined flow during wet-weather and stormwater runoff in particular, an adjusted sampling strategy for MP using large volume samplers (LVS) is introduced. Namely, the following wastewater streams were studied: *influent (L1) and effluent (L2) of a WWTP*, combined sewer flow during dry and wet weather conditions (L3), and stormwater runoff at stormwater retention tank (SRT) in a separate drainage system (L4) (Supporting Information: Figure S1). In addition, differentiation according to different polymer types; PE, polypropylene (PP), polystyrene (PS), polyamide 6 (PA), polyethylene terephthalate (PET), polymethyl methacrylate (PMMA), and styrene-butadiene rubber (SBR), and sizes (5–50, 50–100, 100–500, and 500–1000 µm) are suggested in accordance with Bannick et al. [26] and Goedecke et al. [27] In this paper, the method TED-GC/MS is deployed for the detection of the above-mentioned polymers only and not the additives that, with polymers, makeup MP materials.

SAMPLING LOCATIONS

Municipal WWTP (L1 and L2)

The municipal WWTP of Kaiserslautern, Germany (49°27′33.786"N,7°44′33"E) has a nominal capacity of 210,000 [PEQ][1] and treats domestic and industrial wastewaters from the city and

[1]The abbreviation PE is common for population equivalents. However, PEQ was adopted in this paper to avoid confusion with PE for Polyethylene

other neighboring districts with a total served area of ca. 3500 ha. The treated wastewater flows through maturation ponds into the bordering river Lauter. On average, and under dry-weather conditions, around 49,000 ± 7542 m^3/day or 570 L/s (median = 47,210 m^3/day) of wastewater volume reaches the WWTP to be fully treated (Supporting Information: Figure S2).

Two sampling points at the treatment plant were chosen to investigate the quantity of MP emissions released from its large catchment area and to assess the removal capacity of the WWTP. The first sampling point is in the effluent of four screening units with 3 mm Ø mesh size (L1), while the second one is in the effluent of the secondary settling tanks (L2) (Supporting Information: Figures S1 and S2).

SRT in combined drainage system (L3)

A small catchment with a combined sewer system was chosen to sample dry-weather, and afterward, wet-weather outflows of a large SRT (14,000 m^3) (49°25′37.8"N 7°44′53.6"E) directly in the outflow canal (Ø 800 mm). The SRT serves a catchment with a *total area* of 67.22 ha [A_T] and an *effective impervious area* of 32.79 ha [A_{EIA}]. The traffic density on the two main streets is as high as 4200 [28] and 6300 [29] vehicle/day, respectively.

SRT in a separate drainage system (L4)

A location in a residential district in the city catchment was chosen for sampling the runoff of a separate drainage system. The catchment has a *total area* of 16.93 ha [A_T] and an *effective impervious area* of 6.67 ha [A_{EIA}]. The traffic density in the area is not recorded, but very low traffic was observed during sampling activities. The sampling took place at the outlet point of the catchment in the inflow canal (Ø 800 mm) of the SRT (4700 m^3) (49°25′00.2″N 7°41′44.7″E).

MATERIALS AND METHODS

At an early stage of sampling, less information on specific MP release patterns was available. Therefore, universal sampling guidelines of wastewater and stormwater runoff were used as a baseline to design monitoring strategies and guarantee basic safety and quality requirements.[30–34] To ensure harvesting enough solids from samples, volume-proportional approaches were adopted for sampling activities, both in dry and wet-weather streams. While the upper size limit of 1000 µm was set to focus on smaller MP particles, the lower limit was set at 5 µm due to technical limitations of the smallest available stainless-steel mesh size and the huge effort associated with extracting such small particles using filter cascade.

A combination of manual and automatic methods was applied at the sampling locations. Total solids (TS) measurements were performed on candidate wastewater and stormwater runoff streams to estimate the required sample volumes and to guarantee harvesting enough particulate matter for the MP detection using TED-GC/MS and the parameters (TS, COD, Loss on ignition) (Supporting Information: Table S1). Therefore, for all samples taken throughout the sampling campaigns, the entire particulate matter from the samples was harvested for the analysis, except for a small sample portion preserved for other parameters mentioned above (≈1 L). Another factor behind the choice of sample volumes was maintaining representativity along the entire sampling duration, that is, available sample volume was counted to represent whole or multiple stormwater runoff events.

The thermal detection with TED-GC/MS was conducted on each size fraction individually, and for each size fraction, detected polymers were measured as the weight fraction of TSs within respective size faction in µg/mg. Then, the mean concentration in the sample was calculated as a sum of concentrations of all detected MP according to sample volumes (Equations 1, 2, and 3).

$$C_{m,\mathrm{total}} = \sum_{x=1}^{k} C_{m,\mathrm{MP},x}, \tag{1}$$

$$C_{m,x} = C_{m,x,y1(\mu m)} + \cdots + C_{m,x,yn(\mu m),} \tag{2}$$

$$C_{m,x,y} = \frac{m_{x,y} \times m_{s,y}}{V_s} \tag{3}$$

where:

- k = number of detected polymers,
- $C_{m,\mathrm{total}}$ = mean concentration of all detected MP in the sample in [µg/L],
- $C_{m,x}$: mean concentration of MP × in sample in [µg/L],
- $C_{m,x,y}$: mean concentration of MP × in size fraction y (µm) in [µg/L],
- $m_{x,y}$: mass concentration of MP × in analyzed subsample of size fraction y (µm) in [µg/mg],
- $m_{s,y}$: total mass of sediments of size fraction y (µm) in [µg],
- V_s: sample volume in [L].

Table 1 demonstrates all sampling locations and gives descriptions of their catchment areas, intended sampling strategy, and specific goals and motivations behind choosing this location.

Sampling and estimation of MP loads in dry-weather flow

The sampling of wastewater streams under dry-weather conditions aims to track urban MP emissions, which are released during indoor anthropogenic activities and transported separately or combined to a treatment facility. Following the definition of dry weather days in (1) and by analyzing the inflow data of the WWTP, only 422 days of 1087 measurement days from January 2018 to December 2020 were eligible for sampling.

TABLE 1 Overview of sampling strategies and motivation of each sampling location

Location	Catchment/source	Strategy	Sampling volume (L)	Goal/motivation
Influent of wastewater treatment plant (WWTP) (L1)	City 210,000 [PEQ]	24 h-composite samples	25	• Microplastic (MP) characterization (particle size distribution [PSD], type, and concentrations). • Quantifying of per capita emission rate (g/PEQ · a). • Removal rate of different MP
Effluent of WWTP (L2)	City 210,000 [PEQ]	4 h-composite samples	1000	
Stormwater runoff tank (SRT) in combined drainage system (L3) (dry-weather)	Residential district 5000 [PEQ]	12 h-composite sample	22–25	• MP characterization (PSD, type, and concentrations) • Comparison of per capita emission rate (g/PEQ · a) to one at WWTP • Comparison between emissions during dry and wet weather.
SRT in combined drainage system (L3) (wet-weather)	Residential district 5000 [PEQ] and (67 ha)	Composite samples, LVS	1000	• Designing and adjusting sampling strategy using LVS. • MP characterization (PSD, type, and concentrations). • Quantification of MP emissions from catchment area (g/ha_b · a) (ha_b: impervious area). • Comparison between wet-weather emissions of two catchment areas different in size and land use.
SRT in separate system (L4)	Residential district (17 ha)	Composite samples, LVS	1000	

Influent of a municipal WWTP (L1)

Under dry-weather conditions, a stationary automatic sampler (AS) (ASP Station 2000, Endress+Hauser) was deployed to sample the effluent of the screening units at a suction depth of around 40 cm below the water surface; the length of the suction pipe was less than 2.5 m (Supporting Information: Figure S3). The sampling location behind the screens made automatic sampling without mechanical interruptions possible. Samples were taken time-proportionally (100 ml shot every 6 min) and preserved in 24 glass bottles at 4°C, hence each bottle represented 1 sampling hour. With a total volume of around 23 L (24h-composite sample), samples were then transported to the laboratory to be homogenized volume-proportionally (direct volume-proportional sampling was not possible due to technical issues in syncing the analog signal from the flow meter to AS).

The extrapolation of average daily MP concentration into per capita yearly loads ($B_{\mathrm{MP},a}$) in [g/PEQ·a] was conducted based on yearly median daily flow values under dry-weather conditions (Equation 4), that is, <0.1 mm precipitation or 628 days from 2018 to 2020, with $Q_{\mathrm{T,aM}}$ (median) = 47,819 m^3/day. The number of PEQ was retrieved from our own estimations of WWTP operators. [35]

$$B_{\mathrm{MP},a} = \frac{C_{m,\mathrm{total}} \times [Q_{T,aM} \times 365]}{\mathrm{PEQ}}. \tag{4}$$

Effluent of municipal WWTP (L2)

While concentrations of suspended solids under dry-weather conditions in the influent of WWTP might fluctuate heavily according to release patterns in the catchment within a day, the concentrations in the effluent are less sensitive to these diurnal fluctuations due to continues mixing in several treatment steps. Hence, 4 h composite samples were taken from the effluent stream, usually from 10 a.m. to 2 p.m., of secondary settlings tanks to evaluate the removal efficiency of the treatment processes and characterize MP emissions in terms of PSD and polymer type.

The sampling of the effluent stream was carried out in the effluent canal of the secondary settling tank using a stainless-steel garden pump (flora-best 1100 W) and, due to the low concentration of TSs in the effluent (2–10 mg/L), a relatively large sampling stainless-steel tank of 1000 L. According to the estimation of WWTP operators, the total residence time of wastewater in the treatment plant is 36 h. However, dry-weather conditions could not be guaranteed for long periods, thus, an intended 36 h lag between sampling in the influent stream and the effluent one could not be maintained. To avoid transporting large water volumes to the laboratory, the samples were initially sieved using a sieve cascade (1000–500, 500–100, 100–50 µm) in situ. Then, a subsample of 10–20 L was vacuum filtered with a 5 µm stainless-steel weave. Further sample preparation steps were conducted in the laboratory similar to influent samples.

In analogy to measurements in influent of the treatment plant (see Section Influent of a municipal WWTP [L1]), median daily flow of $Q_{T,\mathrm{aM}}$ (median) = 47,819 m^3/day was adopted to calculate per capita yearly emissions into receiving waters using Equation (4). The removal efficiency of WWTP for total MP, specific MP types, or size fractions was derived by comparing the concentrations in influent and effluent resulting from Equations (1), (2), and (3), respectively.

Dry-weather flow of the SRT in a combined drainage system (L3)

Based on own long-term flow measurements from May to December 2019, a flow pattern could be identified with two flow peaks at around 06:30 and 18:00, and two recessions at around 14:00 and 03:00 (Supporting Information: Figure S4). According to a nightly minimum method for base flow estimation (BW), [20] minimum flow at 03:00 is nearly equal to the base flow in the catchment. Hence, the sampling at this time window of the day was not considered. Only three grab samples per day were taken at 06:30 (≈10 L), at 14:00 (≈5 L), and at 18:30 (≈10 L) to represent the entire day.

The previous approach, however, does not capture sudden changes during the peak flow time from 06:30 to 18:30 but considers a linear change in released MP amounts. To overcome this limitation, a modified approach was introduced to mimic automatic sampling devices; volume-proportionally, and sample the whole duration from 06:30 till 18:30 in a 30-min-sequence. The modified approach was developed to estimate the subsample volume every 30 min based on the dry-weather flow pattern mentioned earlier, real-time flow measurements at the sampling site, and aiming at a total daily sample volume of around 30 L. The sampled volume was set to ensure sufficient sediment amount for the thermal analysis and to reduce the sample preparation time. The previous long-term flow measurements showed that, on average, 472 m^3 of dry-weather flow is recorded from 06:30 to 18:30. Hence, for each 16 m^3 of dry-weather flow, 1 L sample was taken (Equation 5). Before each sampling activity, the real-time flow was retrieved and Equation (6) was used to calculate representative subsample volumes for the time interval. The actual composite sample volume is calculated using Equation (7).

$$\frac{V_{S.ref}}{Q_{d.ref}} = \frac{30\ \text{L}}{472\ \text{m}^3} = 6.36 \times 10^{-5}, \tag{5}$$

$$V_i = (Q_{RT,i} \times t_i) \times \frac{V_{S.ref}}{Q_{d.ref}}, \tag{6}$$

$$V_{sample} = \sum_{i=06:30}^{18:30} V_i, \tag{7}$$

where:

- $V_{S.ref}$: targeted daily sample volume [L] from 06:30 till 18:30,
- $Q_{d.ref}$: reference median dry-weather flow for the time interval from 06:00, to 18:00 based on long-term flow measurements [m^3],
- V_i: sample volume representing the time interval i [L],
- $Q_{RT,i}$: real-time flow measurement at the sampling point [L/s],
- t_i: time interval between each sampling [s],
- V_{sample}: summation of all subsamples representing the day [L].

Sampling and estimation of MP loads of wet-weather flows (L3 and L4)

Stormwater runoff represents, by far, the largest share of wastewater managed within the UDS. With a total storage volume of about 410.000 m^3 stretched over almost 150 stormwater retention structures, the UDS of Kaiserslautern manages huge quantities of stormwater runoff generated from its 3500-ha catchment area.

The two stormwater retention tanks in combined (L3) and separate systems (L4) manage two distinct wastewater flows; combined flow with dry-weather portion and pure stormwater runoff generated from catchment areas with different sizes and traffic densities (see Sections SRT in combined drainage system [L3] and SRT in separate drainage system [L4]).

Since representative sampling of wet-weather flows imposes capturing entire or multiple runoff events with as many solids as possible, LVSs were deployed in the two monitoring campaigns. LVSs allow for long-term, and event-based monitoring of specific pollutants in terms of event mean concentrations (EMC), provide adequate amounts of particulate matter for analysis, and are suitable for deriving reliable particle size distributions (PSD) of solids in samples. [36] For MP monitoring and as mentioned in Equation (3), entire sample volumes were homogenized and prepared (100–1100 L).

Development of a sampling concept

The main element in a sampling system using LVSs is the control unit. Therefore, to maintain the goals of representative wet-weather flow sampling, the control unit was designed to respond automatically to runoff events, and operational parameters were set to fulfill the following aspects:

- Controlled and fully automatic sequence of sampling.
- Event-dynamic sampling, which accounts for the temporal variability of stormwater runoff.
- Consideration of catchment-specific lag times after the end of rainfall.
- Sampling of as many rain events as possible (event diversity).
- Volume-proportional sampling as a composite sample, for obtaining up to 1000 L sample volume.

The requirements described above shaped the performance criteria for the technical equipment and fittings needed at each sampling location, which is briefly described below.

At both sampling locations, flow measurement systems (Nivu-Flow 750, NIVUS GmbH) were installed in the inlet canal of STR in a separate system and outlet canal of a combined system, which, after continuous data aggregation, send digital switching signals volume-proportionally to a programmable logic controller (PLC) (Siemens LOGO). In parallel, an external rain sensor (REGME, B + B

Thermo-Technik GmbH) was used to sense the precipitation event. In the case of rain, it sends a signal to the control unit to activate the entire sampling apparatus.

The sampling itself is controlled by a setup for the sampling pump adapted to the boundary conditions of the respective sampling location. In a separate system (L4), a peristaltic pump (Ponndorf P-Classic 3) (Supporting Information: Figure S5), and in a combined system (L3) a submersible pump (Ebara Optima). The sampling cycles last between 15 and 30 s per switching signal and are executed either until the end of the stormwater-runoff event or until the maximum level in the stainless-steel collection tank (~1100 L) is reached. Details of the sampling algorithm and associated parameterizations can be found in supplementary materials (Supporting Information: Figure S6).

A drawback of using an LVS system is the lack of time allocation of sampling cycles. [36] Therefore, the sampling areas were monitored continuously using a security camera (blink mini, LLC) equipped with a motion detector to capture every sampling cycle and to test the effectiveness of sampling parameters. Hence, it makes it to allocate the exact sampling cycles within runoff hydrographs.

The volume of the sample was measured using the known geometry of LVS and an average of four freeboard measurements above water level. Further sample preparation steps are described in (Section Samples preparation).

The estimation of average MP concentration ($C_{m,\text{total}}$) in samples was performed in analogy to dry-weather samples according to Equations (1), (2), and (3). (see Section Materials and Methods). In addition, specific MP emissions from the effective impervious area (EIA) in the catchment (A_{EIA}), were estimated (extrapolated) according to Equation (8).

$$B_{\text{MP,EIA},a} = \frac{C_{m,\text{total}} \times V_{R,a}}{A_{\text{EIA}}}, \qquad (8)$$

where:

- $B_{\text{MP,EIA},a}$: specific yearly load of MP in [g/(ha · a],
- $V_{R,a}$: total runoff volume in a year, based on flow measurements in [m^3],
- A_{EIA}: effective impervious area in catchment in [ha].

Samples preparation

After each sampling event, samples were collected and prepared within 48 h. Depending on the size of each sample, some samples were first partially sieved in situ (1000–50 µm) and then transported to the laboratory for further preparation. A universal handling method (Supporting Information: Figure S7) was developed to deal with the different types of samples from the different wastewater streams.

Samples were mixed either manually, for small volumes (<30 L) using aluminum rods in a stainless-steel container, or using a mobile mixing device (Atika RL 1000, max. 1000 rpm) for large sample volumes (>30 L). Afterward, samples were wet-sieved using a sieve-cascade of four sieves (1000, 500, 100, and 50 µm, Retsch). In addition, Ø200 mm sieves were used for dry-weather samples and Ø400 mm sieves for stormwater runoff samples. Then, a subsample of the filtrate 1–2 L was vacuum filtered using Combisart® stainless steel filtration system connected to a vacuum pump (Microsart® e.jet Sartorius AG), and 5 µm stainless-steel weaves (Ø5 mm, GKD - Gebr. Kufferath AG). To minimize the drying time in the coming step, the volume of each wet fraction did not exceed 300 ml. Then, samples were sterilized in (VARIOKLAV 75 S, HP Labortechnik GmbH) with a slow-cooling program and dried in Teflon® plates at 105°C in a compartment drier. To avoid cross-contamination, all samples are preserved and transported in plastic-free instruments (Teflon or glass). Each dry sample was then divided into three portions; for MP analysis, Loss on ignition (LoI) analysis, and a third portion was saved for potential future analysis (e.g., MP particle morphology, heavy metals in samples).

The fraction with the size of 50–5 µm from runoff samples was extracted from 2 L samples at the BAM laboratory using a stainless-steel vacuum apparatus (Whatman plc). Filters with a mesh size of 5 µm and 50 mm in diameter (GKD—Gebr. Kufferath AG) collected the solid residue. For further analysis, the filter cake was dried overnight in the oven at a temperature of 50°C.

RESULTS AND DISCUSSION

Dry-weather MP emissions

Influent of a municipal WWTP (L1)

During the period from November 2018 to March 2019, four 24 h composite samples were thermally analyzed in the laboratory to characterize MP emissions in terms of PSD and polymer type from the entire catchment area of the WWTP. The summary of the analytical results shows that, on average, MP occurred with a concentration of 286 µg/L. PE is dominant in size fractions 50–1000 µm and with an average concentration of 244 µg/L in all size fractions (Figure 1). In contrast, the remaining MP types represent only 14.7% of the total load. The approximate total MP loads in the influent of the WWTP during dry-weather conditions are shown in Table 2.

Effluent of municipal WWTP (L2)

The results of this sampling campaign (Figure 2) show that a drastic reduction of MP concentrations occurred within the WWTP. So that the average daily concentration of all size fractions in the effluent of WWTP is only 11 µg/L. Table 2 gives a rough projection of yearly MP emissions of ~189 kg/a or a yearly PEQ load of 0.94 g/(PEQ · a). These results suggest an overall elimination rate of about 96% of all MP entering the WWTP. However, sampling at the inflow of WWTP (L1) was carried out behind screens (Ø3 mm) (see Section Influent of a municipal WWTP [L1]). Hence, the actual removal rate is likely higher than 96%.

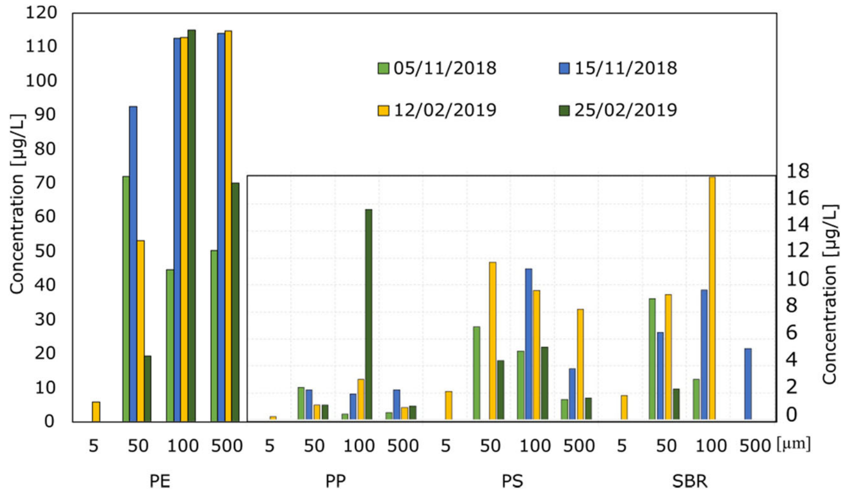

FIGURE 1 Polymer concentrations in different sieve fractions in the influent of wastewater treatment plant (L1, $n = 4$)

TABLE 2 Summary of estimated yearly microplastic (MP) loads in influent and effluent of wastewater treatment plant (WWTP) (L1 and L2) and at the small catchment (L3) under dry weather conditions based on concentrations of all size fractions

	L1 ($n = 4$)	L2 ($n = 3$)	L3 ($n = 8$)	
Average daily concentration of polymer	286	11	794	µg/L
Total annual polymer load	4996	189	n.a	kg/a
Annual polymer loads	23.8	0.94	30.1	g/(PEQ · a)
Average polyethylene loads	20.1	0.73	27.2	g/(PEQ · a)
Average polypropylene emissions	1.6	0.19	1.9	g/(PEQ · a)
Average polystyrene emissions	0.7	0.01	1	g/(PEQ · a)
Average styrene-butadiene rubber emissions	[-]	0.01	[-]	g/(PEQ · a)

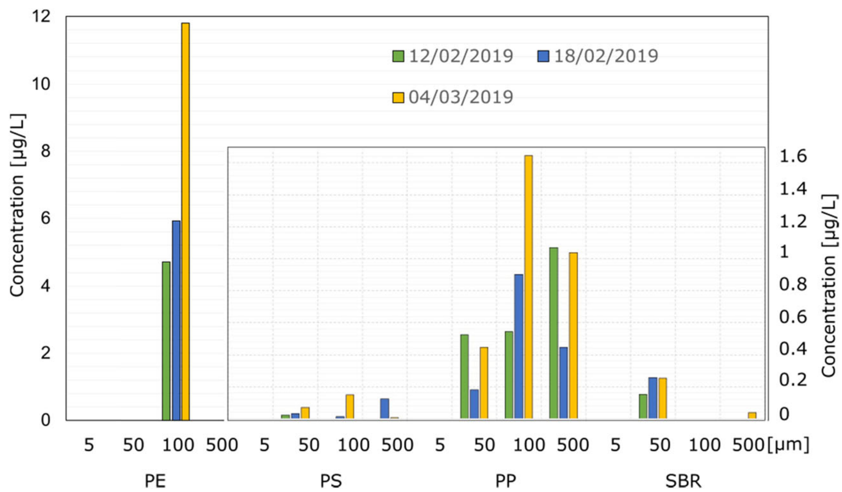

FIGURE 2 Polymer concentrations in size fractions (5–1000 µm) in the effluent of wastewater treatment plant, 4 h composite samples (L2, $n = 3$)

In view of the different polymer types, PE was still dominant, with a high share of about 77% of all detected polymers or 147 kg/a. However, mainly the size fraction 100–500 µm is yet present, while smaller fractions <100 µm are not detected. PP and PS showed no considerable change in PSD, with a slightly higher removal rate for PS. The traces of SBR found in influent at L1 of WWTP were efficiently removed, with the fractions greater than 100 µm completely eliminated.

Dry-weather flow of the SRT in a combined drainage system (L3)

SBR, for example, a possible marker molecule and component of tire abrasion, is clearly a stormwater-borne MP, since it is washed off the traffic surfaces into the sewage system. Nevertheless, it is detected in the dry-weather influent of the WWTP, from which obvious intermediate storage and release effects occur in the sewer system along the

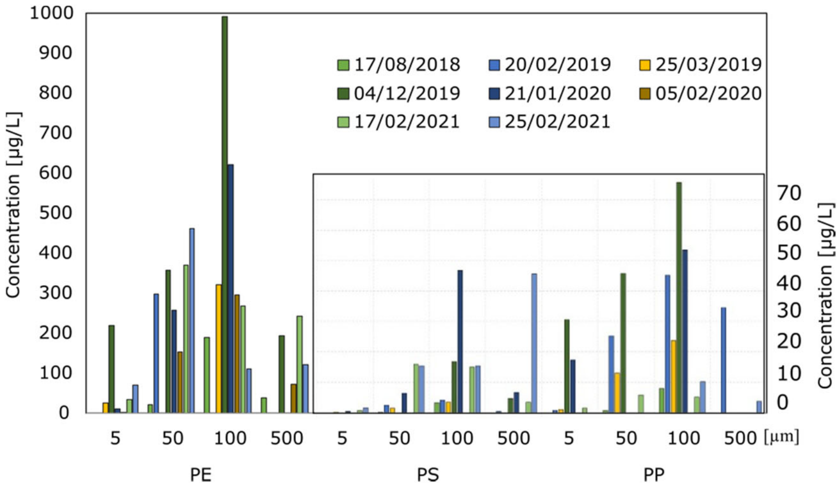

FIGURE 3 Polymer concentrations in dry-weather flow of stormwater runoff tank in combined system at L3, Approach I ($n = 6$) from August 2018 till February 2020, Approach II ($n = 2$) on 17th and 25th of February 2021

transport pathway. Retention tanks in the combined sewer system, in which the wastewater flow is temporarily delayed during wet-weather conditions, and reduced flow velocities favor the sedimentation of solids, including MP. Therefore, stormwater tanks, which are incidentally numerous in the urban sewer network of Kaiserslautern, temporarily represent both potential sinks (through accumulation) and time-delayed sources (through remobilization) for MP.

During this research, two sampling approaches were adopted to sample the dry-weather effluent of the combined system. In total, eight samples were taken using approaches I and II (see Section Dry-weather flow of the SRT in combined drainage system [L3]), six samples with approach I (three grab samples per day) and two with approach II (volume-proportional/24 grab samples over 12 h).

The results in Figure 3 suggest an average daily concentration of 794 µg/L in this wastewater stream. In addition, high average concentrations of PE with 717 µg/L (all size fractions), compared to a lower concentration of PS and PP with about 26 µg/L and 50 µg/L were detected. In contrast to dry-weather samples from the influent of the WWTP, SBR remained undetected (not existing or below the detection limit). In addition, about 51% of polymers were detected in the size fraction 100–500 µm, while only 22% were detected in the same size fraction in the influent of the WWTP (L1) at the end of the catchment area. This suggests that changes in PSD are expected to occur during the residence and transport of MP particles in the sewage network due to the extensive interaction with sewage particles, especially organic content, and also low flow velocities at some locations.

Based on daily flow measurements at the sampling location, exact daily yields of dry-weather flow were calculated and used to estimate yearly per-capita MP emissions from the subcatchment in the combined system at L3. In comparison to per-PEQ MP loads of the whole catchment area, higher values appear at the subcatchment area upstream of the WWTP; (28.9 g/PEQ · a) with sampling approach I and 33.4 g/(PEQ · a) with approach II, or an average of 30 g/(PEQ · a) at L3 and 23 g/(PEQ · a) at L1. However, these values maintain a comparable order of magnitude considering the uncertainties involved with flow measurements at both locations and with actual PEQ numbers, as well as analytical uncertainties. In addition, sampling behind the screens plays here, similar to (Section Influent of a municipal WWTP (L1)), a potential role in underestimating actual MP loads entering the WWTP.

The results from the two sampling approaches are comparable to one another. Thus, monitoring dry-weather flow with only three daily grab samples is more favorable. With such, more samples can be retrieved with less effort. However, reliable flow measurements and accurate dry-weather flow analysis are necessary requirements to guarantee representative sampling.

Table 2 summarizes the results from the three dry-weather samplings at L1, L2, and L3.

Wet-weather MP emissions

Stormwater runoff in a separate system at L4

The SRT in a separate system was the first location to test the novel sampling system described in (Section Development of a sampling concept). During the period from July 2020 to April 2021, seven runoff events were sampled and analyzed for their MP content.

The thermal detection revealed that PE is the most abundant polymer in stormwater runoff (Figure 4, Supporting Information: Table S2) followed by SBR. PE concentrations occurred in all samples in relatively high concentrations (44–249 µg/L) regardless of runoff characteristics and the number of dry days before the rain event. In contrast, SBR concentrations showed a strong dependency on the number of dry-weather days before the rain event (Supporting Information: Table S2), with average concentrations of all size fractions ranging between 9 and 89 µg/L (Figure 4). Yet, both, PE and SBR, were abundant the most in the size fraction of 100–500 µm, counting for 58% and 45% of the total polymer load, respectively. This can be seen as consistent with the findings of a study on tire particle abundance which found that the median value of size distributions of tire abrasion from urban areas is 140 µm. [37]

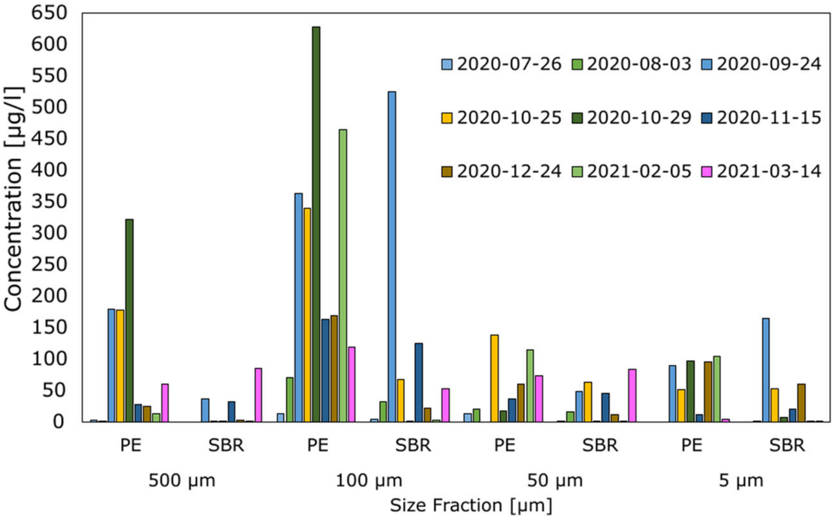

FIGURE 4 Concentrations of polyethylene and styrene-butadiene rubber in stormwater runoff in a separate system (L4, *n* = 7)

While PE and SBR were the most abundant polymers in runoff samples, PS and PP represented only 2.3% and 2.4% of all MP load. In addition, polymers in the size fraction 100–500 µm counted for 55% of the total polymer load. The rest was distributed in the remaining size fractions as follows; 5–50 µm (12%), 50–100 µm (17%), and 500–1000 µm (18%).

Based on the sampling campaign, it can be estimated that at least 558 g_{MP}/ha_{EIA} (n = 7) were transported through the separate drainage system with an average concentration of 122 µg/L. However, the sampled stormwater runoffs from September 2020 till March 2021 represent only 71% (≈35,155 m^3) of the yearly total stormwater runoff of ≈50,154 m^3 measured from September 2020 till September 2021. Using the average concentration from the sampling campaign, one can estimate a yearly polymer emission of about 796 $g_{MP}/(ha \cdot a)$ from this catchment area.

Wet-weather flow in the combined system at L3

The results of the sampling campaign in the combined system confirm the findings already obtained in the separate system that mainly PE and SBR occur in the stormwater-borne MP emissions. These two polymers represent about 96% of all MP identified in all runoff samples, which illustrates the high relevance of their urban occurrence. Figure 5 shows the concentrations of the detected polymers in the size fractions of 5–1000 µm. The average minimum and maximum concentrations over all fractions and stormwater runoff events range from 41 to 440 µg/L for PE and 1–724 µg/L for SBR. The majority (60%–85%) of all MP loads are linked to fractions larger than 50 µm. Based on average MP concentrations in dry-weather flow from Section Dry-weather flow of the SRT in combined drainage system [L3] and typical dry-weather flow portion (2%–7%) during the sampling of runoff events, polymers emissions related to dry-weather flow are negligible, and we assume that most polymers are transported with the stormwater runoff.

Comparison between MP emissions from the two catchment areas

With a closer look at the two main MP types in stormwater runoff; PE and SBR, the specific yearly emission of PE in the larger catchment is slightly higher (Table 3), probably because of higher population density. In contrast, the size and high traffic density in the larger catchment are reflected in the high specific load of SBR compared to the specific load of the separate system.

Finally, the city of Kaiserslautern has a unique UDS that consists mainly of stormwater retention structures, which concentrates the pathway for MP to the central WWTP. However, specific emissions from urban catchment areas are comparable to other catchments with different drainage schemes.

Therefore, to understand the significance of the wet-weather pathway compared to the dry-weather one, we choose to extrapolate the emissions from the studied catchments L3 (1952 g/[ha · a]), as the maximum specific emission, and L4 (796 g/[ha · a]), as the minimum specific emission, to an entire catchment of the studied drainage system in Kaiserslautern with a total catchment size of about 2343 ha. [38] Assuming, however, an impervious active catchment of about 56% of the total area, only half of the year combined wet-weather flow or stormwater runoff is managed by either combined sewer overflows (CSOs) or stormwater treatment tanks and a 40% average TSs removal efficiency at these structures. The results from this simple example suggest that at least 314–770 kg_{MP}/a from the entire catchment area of the drainage system will find its way to receiving waters. Compared to 189 kg_{MP}/a (see Section Effluent of municipal WWTP [L2]) that are expected to reach receiving waters along the dry-weather pathways, wet-weather emissions are two to four times higher. In addition, the studied catchment areas are located at the edge of the city with lower traffic compared to catchments near the city center where more commercial activities and traffic are expected, and varying population densities. Hence, higher emission rates are expected from the entire catchment area.

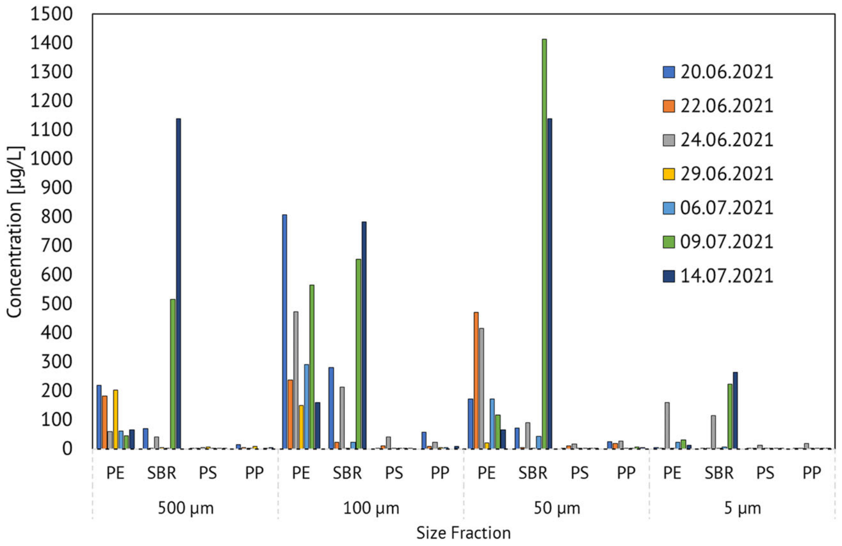

FIGURE 5 Polymer concentrations in combined wet-weather flow at stormwater runoff tank in the combined system (L3, wet weather, $n = 7$)

TABLE 3 Comparison between specific yearly emissions of polyethylene (PE) and styrene-butadiene rubber (SBR) in the two catchment areas

Polymer	Large catchment at L3		Small catchment at L4	
	Total load	Specific load	Total load	Specific load
	g/a	g/(ha · a)	g/a	g/(ha · a)
PE	23,061	703	3965	517
SBR	39,317	1199	1942	253

CONCLUSION AND OVERVIEW

The experience gained during polymer analysis (sampling, sample preparation, detection) suggests that one week should be planned per intended sampling. Accordingly, an amount of ~10–12 24 h-composite samples per year is recommended for sampling campaigns under dry-weather conditions. While for sampling under wet-weather conditions, all available events during a chosen time frame should be aimed at. The sample volume to be obtained must be aligned with the requirements of sample preparation and considering analytical detection limits. Thus, for each subsample or size fraction taken, there should be a sufficiently high solid yield to ensure the representativeness of the analysis and to exceed the detection limit by at least five orders of magnitude. Therefore, a minimum of 300 mg solids per size fraction is recommended for MP analysis with TED-GC/MS, from which 10 to 50 mg aliquots of the homogeneous sample can be measured directly without further sample preparation. For dry-weather media with an SS range of 200–400 mg/L, a sample volume of 30 L is recommended, also to reduce the preparation time. Whilst for wet-weather media, sample volumes between 200 and 800 L are recommended for representative MP and PSD analysis.

The results of sampling at the central WWTP show that at least 96% of MP entering the plant can be removed. PE is the most abundant MP in the influent and effluent of the WWTP, with a share of 86% and 77% of the total detected MP load, respectively. SBR was also detected in the influent (5.8%) and effluent (≈1%) under dry-weather conditions. PP represented about 6.6% of the MP load in the influent and 21% in the effluent of WWTP. The previous results from the WWTP suggest that WWTP is a less-significant entry pathway for MP in urban areas.

Away from the WWTP at the edge of the catchment area, MP emissions in dry-weather flow showed different characteristics in terms of quantity and PSD. While the average daily MP concentration at the WWTP influent was 286 µg/L (L1), the average daily concentration at (L3) was 794 µg/L almost three times higher. The reason behind that can be linked to the high base-water portion at the WWTP (47%) compared to a low portion (<10%) at the small catchment. Also, changes in the PSD of MP occur along the journey from the source to the WWTP.

The novel results obtained from sampling campaigns of wet-weather flow in separate and combined systems show the high relevance and dominance of the two polymers PE and SBR, the effect of catchment size, and traffic density on specific emission rates. On the one hand, PE occurred in high concentrations in both catchments regardless of weather conditions before the rain event, which suggests the constant release of this polymer from urban areas and the high variety of sources behind. On the other hand, SBR concentration is affected by the number of dry weather days before the rain event and the traffic density. Thus, the specific SBR emission from the larger catchment with higher traffic density was almost five times higher than the one from the small catchment.

While dry-weather emissions at WWTP are seen as less significant in magnitude to overall MP pollution of urban origin, wet-weather emissions play a much more significant role due to low treatment possibilities in these urban areas. The simplified example in (Section Comparison between MP emissions from the two catchment areas) with extrapolating MP emissions to the entire city catchment showed that two to four times more MP is likely reaching receiving waters over wet-weather pathways. However, the efficient treatment of MP at WWTP suggests that the ongoing and increasing treatment capacities of wet-weather flow will decrease the emissions from this pathway drastically.

Sampling within the sewer system implies dealing with a set of variables and requirements. Flow fluctuations, accessibility at selected sampling points, large objects in the wastewater stream, residence time, and safety considerations represent many challenges while designing a reliable monitoring program. In particular, monitoring of wet-weather flow is more complex and labor-intensive since more effort is required for designing, installing, and operating sampling equipment. The intermittent occurrence of rainfall events and the high temporal dynamics of the precipitation quantity create complex planning conditions for sampling, which require *adjusted-to-catchment* strategies. The mechanism of flushing and accumulation of MP particles from the catchment area has a potential effect on the quality of data acquired, as well as the existence of sinks, where MP is possibly released into the environment prior to reaching the endpoint of the drainage system. Thus, stormwater-borne SBR from tire abrasion is still latently detected in the dry-weather influent of the WWTP.

To establish a holistic view of MP release from urban areas through the drainage system, relevant sampling locations upstream of the WWTP need to be systematically investigated with differentiation between dry and wet-weather transport pathways. A sampling at the treatment plant alone "end of pipe" allows merely for acquiring data on the elimination performance of the many treatment aggregates.

Finally, to acquire more reliable estimates of emission loads, a much larger sampling effort over longer periods of time is required, in which a sufficient variety of rainfall events and accumulation phases are represented. This is also seen as a need for further research, for example, by linking automated sampling systems with nowcasting information on the immediately upcoming rainfall event.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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SUPPORTING INFORMATION

Additional supporting information can be found online in the Supporting Information section at the end of this article.

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