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Microplastics and Nanoplastics

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Microplastics in a highway stormwater system: concentrations and variability during rainfall

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Abstract

Microplastics (MP) have been reported in a wide range of environmental matrices, including seawater, freshwater, stormwater, sediments, soils, and air. Stormwater from urban areas has proven to be a major pathway for the transport of MP into aquatic environments, especially for tyre wear particles (TWP). This study investigates the presence of MP and tyre-related rubber polymers in stormwater collected during rainfall events at a Swedish highway. The specific objectives were to: quantify the concentrations of 13 MP polymers, including three tyre-related rubber polymers, in the particle size fraction 10–1000 µm in highway runoff; assess the relationships between MP concentrations, total suspended solids (TSS), volatile suspended solids (VSS) and metals. Total MP concentrations, including tyre-related polymers, were 2.4–4200 µg/L in the stormwater samples. The most frequently quantified polymers were polyethylene, polypropylene, and tyre-related polymers styrene-butadiene rubber and polyisoprene, in agreement with other studies on the occurrence of MP in runoff. The tyre-related polymers accounted for 41 ± 24% of the total MP concentrations in the samples where they were quantified, confirming TWP as an important source of MP pollution in this type of environment. The tyre-related polymers were quantified in lower concentrations than in other studies, indicating that concentrations could be underestimated due to the analytical size cut-off at 10 µm. No significant correlations were observed between MP concentrations and TSS, VSS or metals. This study contributes to the scientific understanding of MP emissions from road runoff and provides knowledge for the development of methods to treat road runoff from pollutants. Further research should address the development of standardised sampling techniques and sample preparation methodologies for pyrolysis-gas chromatography / mass spectrometry that can reliably identify and quantify smaller MP (< 10 µm) and are robust against interference from complex environmental matrices.

Keywords Automatic sampling, Intra-event variation, Microplastics, Pyrolysis-GC/MS, Runoff, Stormwater quality, Tire wear particles, Tyre wear particles

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Introduction

Microplastics (MP) have been found in a large number of environmental matrices, such as seawater, freshwater, stormwater, sediments, soils, and air [1]. MP are reported to be widespread in food and drinks, and have been found in multiple tissues and organs of humans, with emerging evidence of potential effects [2]. Large emissions, toxic additives and the presence of MP in the environment are of global concern. Recent studies reveal that over 16,000 plastic chemicals are incorporated into plastic materials, with at least 4200 of these being highly hazardous to both human health and the environment [3]. Specifically, tyre wear particles (TWP) contain many additives that provide strength and antioxidative properties to the rubber material [4]; however, many of these additives have been linked to harmful impacts in the environment [5–7], with recent studies suggesting that leachates from tyres are the main toxicity drivers [8].

In urban areas, stormwater has proven to be the major pathway for the transport of MP from land to aquatic environments [9–11]. Tyre and road wear particles from traffic have been identified as one of the most abundant sources of MP in runoff [12–14], together with air deposition and littering [15–17]. However, the concentrations of MP in stormwater have not been assessed to a large extent [17], particularly regarding the occurrence of MP < 20 µm [18, 19] with fine particles not addressed due to analytical challenges [20, 21]. Also, the dynamic behaviour of MP mobilisation and transport in runoff during rainfall have not been assessed to a large extent [10], with only a few studies addressing MP concentrations in stormwater during rainfall [22–24] and mostly focusing on MP sizes > 20 µm.

Spectroscopic methods, such as Fourier transform infrared (FTIR), Raman and Scanning electron microscopy (SEM), are among the most widely employed methods for MP analysis [25], and report MP concentrations as the number of particles per sample or as a calculated mass based on assumptions of particle size and density. Among these, FTIR and Raman are the most used techniques for identification of MP; however, these techniques are not suited for identification and quantification of TWP due to the high levels of carbon black [26, 27]. The high carbon black content of TWP strongly absorbs incident radiation and masks the characteristic spectral signatures of the constituent polymers, limiting the applicability of FTIR and Raman spectroscopy for TWP identification and quantification. Further, MP count in the finer size fractions < 20–40 µm can be underestimated due to deterioration of the spectra quality when using FTIR techniques [28]. Thermal decomposition methods, such as thermal extraction and desorption gas chromatography / mass spectrometry (TED-GC/MS) and pyrolysis-gas chromatography / mass spectrometry (Pyr-GC/MS), instead allow mass quantification of polymers present in TWP, as well as other MP polymers in environmental samples [27, 29, 30]. The discrepancies in analytical methods and reporting units between the methods make it difficult to compare MP concentrations across studies employing different analytical techniques. Additional challenges when comparing MP concentrations between studies arise from the differences in pre-treatment steps and size fractions investigated, as standardised collection, pre-treatment and analytical methods are still lacking for MP [31].

The Pyr-GC/MS technique does not have an inherent lower size limit, unless introduced from sample pre-treatment, but it has the disadvantages of not characterising the particles and destroying the samples upon analysis [27, 32], and accuracy can be affected by particle size [33]. It is an emerging technique for MP analysis that has been applied to complex sample matrices [30, 32, 34–40] making it useful for MP quantification for environmental monitoring [32]. In the literature, some studies can be found analysing TWP and related rubber polymers in stormwater using Pyr-GC/MS [14, 18, 41–43], whereas only a few studies have quantified other MP [44, 45]. Quantification of both MP and TWP polymers using Pyr-GC/MS (> 27 µm) has been performed in stormwater, street sweeping sand and snow samples [46–48].

This study aims to identify the presence of MP, including TWP polymers, in stormwater down to 10 µm, collected through automatic sequential sampling during rain events at a highway, and analysed with Pyr-GC/MS. The specific objectives were to: (i) quantify the concentrations of 13 MP polymers, including three TWP-related polymers, in highway runoff during rain events; (ii) relate the concentrations of different MP polymers to total suspended solids (TSS) and metal concentrations. The quantified MP concentrations are compared to data from other studies on stormwater in road environments, while discussing the effects of the particle sizes assessed, sample preparation methods, and analysis techniques. By combining sequential stormwater sampling during rain events with mass-based quantification of MP in size fraction 10–1000 µm, this study contributes to the limited body of research investigating occurrence and composition of MP, including TWP-related polymers, in highway runoff.

Methods

Study area

Sampling was carried out in the stormwater system at Testsite E18, a road research facility owned by the Swedish Transport Administration [49] and built in 2010, see Fig. 1. The highway E18 passes by the test site and has two lanes in each direction. The test site is located along the right lane heading towards Enköping. This part of the

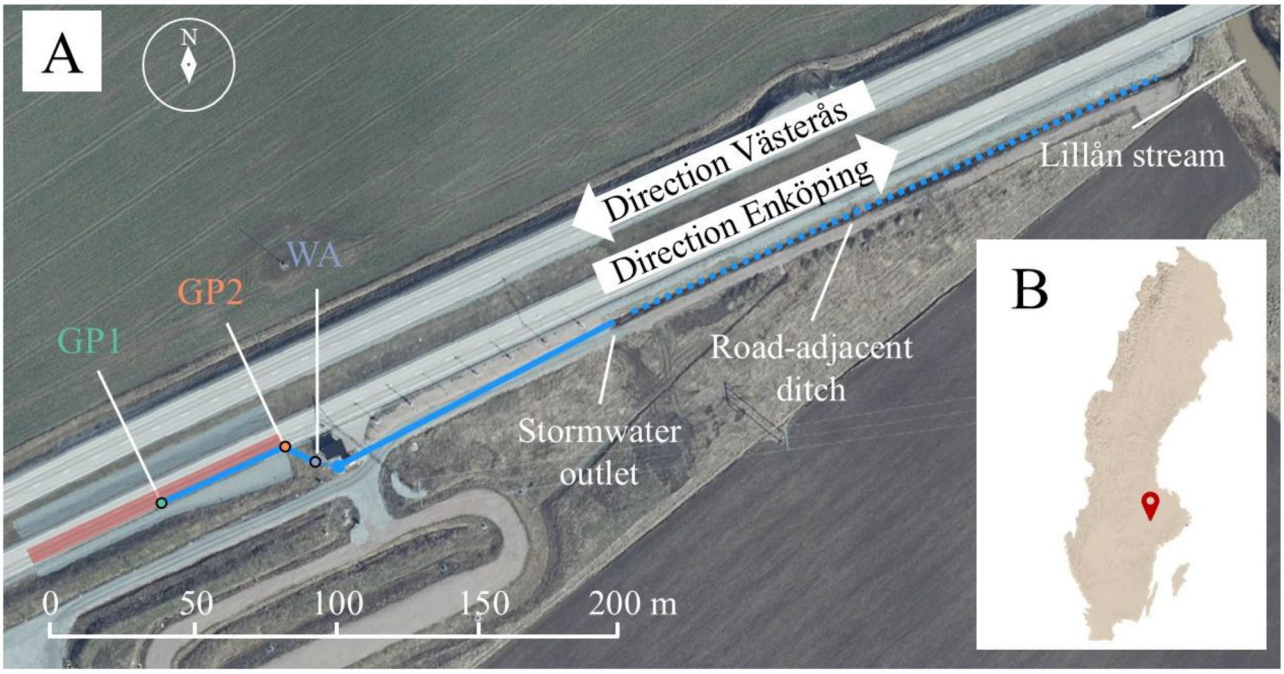


Fig. 1 (**A**) Map of Testsite E18 with illustration of the stormwater system by the highway. The red area represents the catchment of the stormwater system, and the blue lines represent the stormwater path through pipes. Circles represent the sampled gully pots (GP1 and GP2) and well (WA). (**B**) Location of Testsite E18 in Sweden. Maps retrieved and adapted from the Swedish Land Survey

highway is located in a rural area; approximately 12,000 annual average daily traffic (AADT), of which 10% are heavy-duty vehicles, pass by Testsite E18 [50], and the speed limit is 120 km/h. At the test site, a stormwater system has been installed. A kerb leads the water from the red road section in Fig. 1A towards two grate-covered gully pots (GP1 and GP2). Both gully pots, GP1 and GP2, dewater the road surface directly through inlet grates. Gully pot GP1 connects to gully pot GP2 through a pipe (diameter 15 cm), which then directs the water towards a collecting well WA (pipe diameter 17 cm). From well WA, the water is drained to a collection well (pipe diameter 20 cm) and then released into a road-adjacent ditch. The gully pots have a diameter of 40 cm and sand traps with depths of ca. 30 cm, resulting in a sedimentation volume of ca. 38 L each. Both gully pots contained sediments at the time of the sampling [18]. Well WA is equipped with a Thomson weir, which creates a standing water volume in part of the well of ca. 280 L [51].

Automatic sequential sampling during rain

As described in Gaggini et al., [51], a sampling campaign was carried out in the time period between 2023-03-28 and 2023-05-30. During this period, two rain events were sampled on 2023-04-29 and 2023-05-17. The weather conditions for the sampled rain events are described in Fig. 2. The antecedent dry period (< 2 mm/h) before 2023-04-29 began at 02:00 on 2023-04-25, during which a total of 32,717 vehicles passed in the rightmost lane crossing the catchment area. For the second rain event, the antecedent dry period before 2023-05-17 began at 11:20 on 2023-05-01, with a total traffic volume of 126,392 vehicles in the rightmost lane.

In total, three automatic sequential samplers were installed in the stormwater system at Testsite E18. In GP1, the sampler model was ISCO 6712c equipped with 24 polyethylene (PE) bottles of volume 0.5 L. In GP2, the sampler model was ISCO 6712 equipped with 24 polypropylene (PP) bottles of volume 1 L. In each gully pot, the inlet hoses to the samplers were installed in the standing water volumes, and a water level meter was installed in the water volume of each sand trap to trigger the samplers at a water level of 10 mm above the bottom of the outlet. In WA, the sampler model was ISCO 6712c equipped with 24 PE bottles of volume 0.5 L. The inlet hose was installed in the standing water volume of the well, with sampling triggered through a flowmeter installed approximately 20 cm into the outlet pipe. The flow meter was set to trigger the sampler at a measured flow of 1.5 L/s. When triggered, all samplers took 1 L samples every 2.5 min, thus filling two 0.5 L bottles or one 1 L bottle. Sampling at the three locations was successful on 2023-04-29 and 2023-05-17. The samples were retrieved from the field after 6 and 1 days, respectively. Retrieved samples were transferred to glass bottles and stored in fridges at a temperature of 4–8 °C prior to analysis.

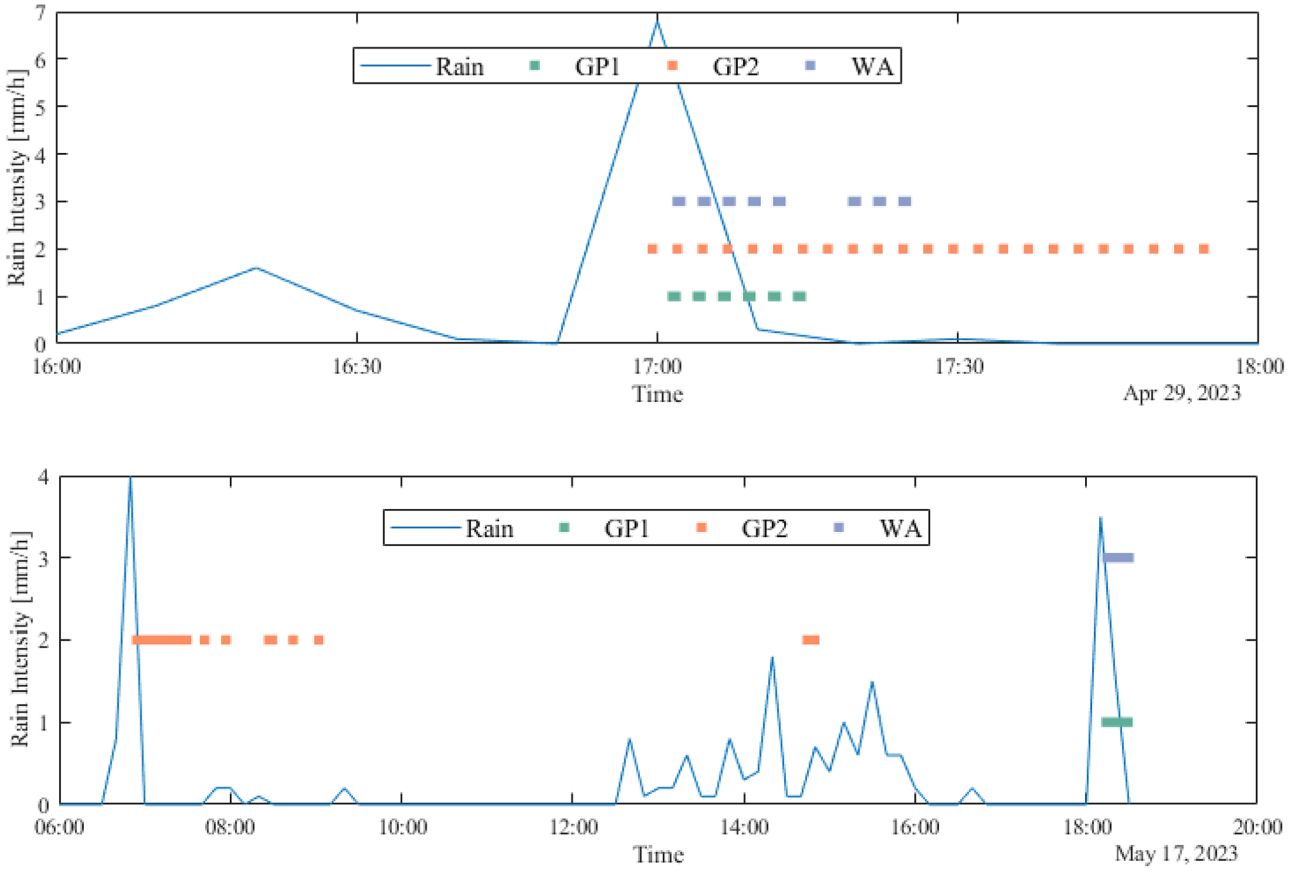


Fig. 2 Precipitation measured every 10 min on 2023-04-29 (top) and 2023-05-17 (bottom) at Testsite E18, with coloured dots marking the samples taken in the gully pots (GP1 and GP2) and the collection well (WA). Selected samples were aggregated and analysed (see Supporting Information SI 2)

The automatic ISCO samplers used for sampling contained several plastic components, including PP and PE bottles and a PVC pump hose, that could cause contamination of the samples. The samplers are covered by a lid in the field, which should prevent bottles from receiving contamination by air deposition of MP. To assess the potential contamination from the sampling equipment, two procedural blanks were prepared by pumping distilled water through the ISCO samplers and storing it in the plastic sampling bottles, before the sampling campaign. The blank samples were combined into a single composite sample, which was analysed in the same manner as the other samples.

Sample preparation and quantification of MP

Analysis of MP (>10 µm) was performed using Pyr-GC/MS by a commercial laboratory on selected aggregated samples of approximately 1 L from all locations and sampled rains. In total, 19 samples were analysed. The aggregated samples were prepared by combining volumes of homogenised samples from different bottles, see Table SI 2, and Figures SI 1 and SI 2 in the Supporting Information (SI). Thirteen MP polymers in the size fraction 10–1000 µm, were analysed in the stormwater samples with varying limits of quantification (LoQ see Table SI 2): acrylonitrile butadiene styrene (ABS), polyethylene (PE), polypropylene (PP), polystyrene (PS), polymethylmethacrylate (PMMA), polycarbonate (PC), polyvinyl chloride (PVC), polyethylene terephthalate (PET), polyamide 6 (PA6), polyamide-6,6 (PA66), and three tyre-related rubber polymers polyisoprene (PI), polybutadiene (PB), and styrene-butadiene rubber (SBR).

At the commercial laboratory, the stormwater samples were filtrated through stainless-steel filters (SSF) with a 10 µm pore size; these filters were then submerged into beakers of water and subjected to ultrasonication for 10 min. After rinsing and washing, the resulting liquids were mixed with a 10% potassium hydroxide (KOH) solution for oxidation and incubated overnight at 40 °C. If particle concentrations were low, the samples were decanted before being filtrated with a 1.6 µm GF/A filter, and thereafter added to the eco-cups for pyrolysis. In cases where the particle concentrations were high, the samples underwent an additional filtration through a 10 µm SSF after oxidation. These filters were then subjected to ultrasonication, and thereafter, calcium chloride

($CaCl_2$, density 1.4 g/mL) was added to the liquids for density separation. These samples were then decanted and filtrated on 1.6 µm GF/A filters, and the filters were added to eco-cups for pyrolysis. If the samples had high concentrations of particles after the density separation, they were filtrated again with a 10 µm SSF, and aliquots of particles from these filters were then added to eco-cups for pyrolysis. The aliquots were prepared by transferring the entire content from the dried filters into a mortar for homogenisation. The Pyr-GC/MS system for the MP polymer analysis included a multi-shot pyrolizer EGA/PY-3030D from Frontier Labs with an auto-shot sampler AS-1020E, an Agilent GC 8890, and an Agilent MS 5977b. Filters or aliquots of the samples were placed in eco-cups and loaded into the autosampler. Quality controls were manually assessed from a mixture of 13 polymers, using silicon dioxide (SiO_2) as a diluent, with the addition of internal polymer standards. Each polymer was identified and quantified through a specific pyrolysis marker compound, see Table SI 1. Plastic materials were present in the lab environment, but care was taken to use glass bottles for sample storage in the lab and to avoid the use of other plastic equipment during sample handling before shipping to the commercial laboratory.

Dataset on water quality and tyre-related polymers at Testsite E18

The samples analysed in the present study were also analysed in a previous study by Gaggini et al., [51] in terms of other water quality parameters and concentrations of TWP. The dataset published in Gaggini et al., [51] is only briefly described here and include: total suspended solids (TSS), volatile suspended solids (VSS), total (unfiltered) concentration of metals (Al, As, Ba, Ca, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, V and Zn) and TWP concentrations calculated from styrene butadiene rubber and butadiene rubber (SBR + BR). The dataset in Gaggini et al., [51] was used in the present study to assess possible correlations with MP concentrations. The samples analysed by Gaggini et al., [51] have a higher temporal resolution compared to the present study, where the individual samples of equal volumes had to be aggregated due to the larger volumes required for MP analysis (see Section Sample preparation and quantification of MP). Therefore, the results for TSS, VSS, and total metals by Gaggini et al., [51] were recalculated for the aggregated samples by averaging the values of the individual bottles; these results are included in SI 4 and Fig. 4.

The methodology employed in Gaggini et al., [51] to quantify tyre-related rubber polymers differs from the present study (see Section Sample preparation and quantification of MP). In Gaggini et al., [51], sample preparation consisted of wet-sieving, a centrifugation step and vacuum filtration onto glass fibre filters (size cut-off > 1.6 µm) This was followed by Pyr-GC/MS analyses to quantify styrene-butadiene rubber and butadiene rubber (SBR + BR) using a four-marker combination (m/z 78 Da benzene, m/z 118 Da α-methylstyrene, m/z 117 Da ethylstyrene, and m/z 91 Da for butadiene trimer). Some samples in Gaggini et al., [51] had high concentrations of benzene, and for these samples Gaggini et al., [51] excluded benzene as a marker in the SBR + BR quantification. These samples are indicated in Table SI 2. More details on the sample pre-treatment, Pyr-GC/MS methodology and settings, as well as the marker combination can be found in Rødland et al., [52].

Statistical analyses

Statistical analyses were conducted in IBM SPSS Statistics 28. Due to the non-normality of the data, Spearman's rank order correlation tests were used to investigate the relationship between MP concentrations, TSS, VSS, metal concentrations and SBR + BR. For MP polymer concentrations that were < LoQ, the values used in statistical analyses were set to one-tenth of the respective polymer-specific LoQ reported in Table SI 2.

Results and discussion

MP polymers in stormwater at testsite E18

The quantified MP concentrations for the 13 polymers found in the gully pots and the well of the highway system during the two sampled rain events are presented in Fig. 3 and Table SI 2. The quantified MP are in the size fraction 10–1000 µm (see Section Sample preparation and quantification of MP), and the total MP concentrations ranged from 2.4 to 4200 µg/L in the analysed samples. Of the 19 samples analysed, only a few polymer types were > LoQ in more than three samples. The polymers found in the samples were mainly PE, PP, SBR, and PI, followed by PB, PVC, and ABS, which occurred in a few samples. Relatively high concentrations of PE occurred (from < 0.10 up to 1400 µg/L); however, the polymer was quantified inconsistently between the samples, with only four samples with concentrations > LoQ. This observation may reflect an intermittent occurrence of relatively large PE particles that strongly influence the measured mass concentration. Relatively high concentrations were also found for PP from < 0.50–55 µg/L, and it was the most commonly occurring polymer in the samples, found at concentrations > LoQ in 15 of 19 samples. The synthetic rubber polymers SBR and PI were also quantified recurrently in the samples, in 6 and 8 samples, respectively. The concentrations were < 0.20–1800 µg/L for SBR and < 1.0–1000 µg/L for PI. The polymers occurring in fewer of the samples (only in one to three samples), i.e. PB, PVC, and ABS, were found in concentrations of < 1.0–3.6 µg/L, < 3.0–17 µg/L, and < 0.2–1.4 µg/L, respectively. None of the polymers PS, PMMA, PC, PET, and PA66

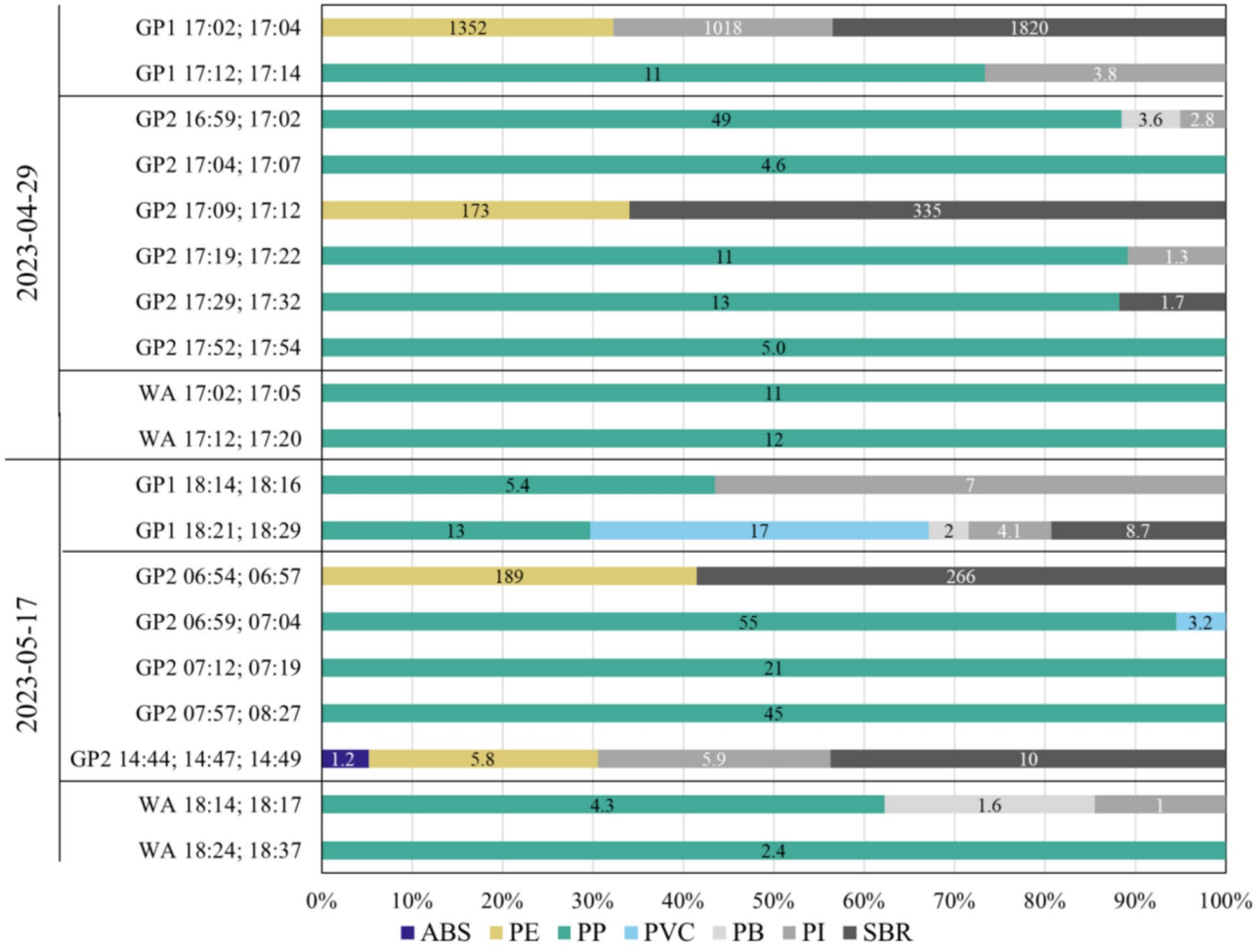


Fig. 3 Concentration (in µg/L, indicated by the values inside the bars) and relative proportion (%, indicated by the size of the bar along the horizontal axis) in the samples of MP polymers: acrylonitrile butadiene styrene (ABS), polyethylene (PE), polypropylene (PP), polyvinyl chloride (PVC), polybutadiene (PB), polyisoprene (PI) and styrene-butadiene rubber (SBR). Polymers analysed in concentrations < LoQ are not included in the figure

were quantified at concentrations > LoQ (from < 0.10 up to 3.0 µg/L) in any of the samples, and PA6 was only found in low concentrations in one sample.

Contamination from equipment or other external sources is always a concern in MP analysis. As mentioned in Section Sample preparation and quantification of MP, the levels of sample contamination were assessed by analysing a composite blank sample. The results showed measurable concentrations of ABS, PET, and PP. However, PP concentrations were on average 17 times lower than those found in the samples and 1.7 times lower than in the sample with the lowest measured concentration, see Table SI 2. The other MP polymers were < LoQ in the blank, indicating low levels of external contamination compared to the concentrations in the stormwater.

The most abundant polymers at Testsite E18 (PE, PI, SBR, and particularly PP, Fig. 3) are also among the most reported in the literature. The polymers PE and PP are among the most frequently identified in stormwater and sediments from gully pots [22, 24, 28, 41, 53–56]. Source appointment of polymer types is challenging [53], and there are many sources and pathways of MP into the environment. The Testsite E18 catchment consists only of the highway section (red area in Fig. 1), and is surrounded by agricultural land, which can narrow down the possible MP sources to most likely be high-speed traffic sources and littering, with some possible contribution from air deposition [17]. The polymers PE and PP are among the most widely produced polymers in the world and are also among the most frequently reported in urban areas [57], as well as the most common polymers in packaging materials, containers and disposable products [58, 59]. Their presence in the highway stormwater system at Testsite E18, which is located in a rural area, could therefore originate from littering or from air deposition. The polymers PE and PP, together with ABS and PVC, are also widely used in vehicle parts [60], and the MP in the runoff can also originate from wear of these components.

Up to 15% of a car's total weight is estimated to consist of plastic materials [78]. As plastic use in vehicle manufacturing increases to reduce weight, MP release to the environment from worn car components is also likely to rise. Other widely produced polymers, including PVC, PET, PS, and PUR (OECD Global Plastics Outlook Database, [61]), were found only at low concentrations or not at all in the present study. This may indicate that their specific sources were absent or limited within the highway catchment, although analytical challenges associated with quantifying certain polymers in environmental matrices cannot be excluded. As the highway by Testsite E18 is surrounded by agricultural land, agricultural activities like mulching and sewage sludge application may also contribute MP to the stormwater system, with PE being the most used plastic in agriculture [62]. In the present study, the agriculture practices employed on the land surrounding Testsite E18 are unknown. Since the studied catchment includes only the highway road surface, the contribution of agricultural MP to the stormwater system is likely limited to atmospheric deposition.

The rubber polymers found > LoQ in the samples (PB, PI and SBR) made up on average 41 ± 24% of all MP quantified in the samples in which they were found. Even if the rubber polymers were not quantified in concentrations > LoQ in all samples, they represent an important fraction of the MP measured, indicating that tyre wear is a major contributor of MP to stormwater in the near-road environment. This is also suggested by the high estimated emissions of TWP from Swedish highways [63], and other sampling studies of highway runoff have also reported that rubber was the most abundant MP [28, 41, 64].

Several limitations should be considered when interpreting the MP concentrations. The stormwater samples collected and analysed represent MP occurrence in the gully pots and wells of the stormwater system at discrete times during the rain events, providing temporal resolution. As shown in Fig. 2, the samplers were not triggered simultaneously and did not sample throughout the entire duration of the rain events. Therefore, the measured MP concentrations do not represent the total MP load in the stormwater system, which would require continuous sampling approaches, such as nets or installed filtration units. Also, only a limited number of rain events could be sampled due to weather conditions and time constraints, and a single highway stormwater system was investigated, restricting the extent to which the observed trends can be generalised. Furthermore, the samples were collected within the stormwater system. Coarser MP may not be mobilised from the road, and since the gully pots and the well are equipped with sand traps and standing water volumes, coarse MP may settle in the sand trap depths. At the same time, the measured concentrations may be influenced by resuspension of previously accumulated MP from the sediments in the stormwater system. The measured concentrations are therefore indicative of MP concentrations within the stormwater system (locations GP1 and GP2) and flowing out of the system (WA, Fig. 2), rather than of MP concentrations mobilised from the road surface. More research investigating the mobilisation of MP from road surfaces during rain events is necessary to assess MP loads into stormwater systems, as well as MP transport and retention processes within such systems. Additionally, the absence or low occurrence of some polymers should be interpreted with caution. Certain Pyr-GC/MS markers can be affected by matrix interferences, potentially causing underestimation of polymer concentrations. For example, quantification of PET with Pyr-GC/MS through benzoic acid may be influenced by iron oxides [65], and PVC quantification using marker naphthalene can be affected by other polymers, as well as organic matter [66]. More research is needed to improve the understanding of marker performance and analytical uncertainties associated with MP quantification in complex environmental matrices. As highlighted by Goßmann et al., [66], multi-method approaches combining thermal and spectroscopic techniques might be necessary to improve accuracy.

Comparison of MP concentrations in stormwater

In the literature, there are several reviews and other studies investigating MP in stormwater [10, 31, 67], but comparison of MP concentrations across studies is challenging due to differences in sample pre-treatment, particle sizes analysed and analytical methods employed, with only a limited number of studies reporting MP concentrations as mass per volume, rather than particle counts per volume. Studies investigating mass quantification of MP in stormwater, either with Pyr-GC/MS, TED-GC/MS or FTIR, have been summarised in Table 1.

These studies show considerable variation in both sample pre-treatment, analytical approaches and the types of stormwater investigated. Most studies have employed FTIR techniques to estimate MP concentrations, with fewer studies attempting MP quantification through Pyr-GC/MS. Sample pre-treatment steps applied in the studies include filtration, ultrasonication, digestion, and density separation. These procedures were not implemented consistently between studies, and different chemicals were employed in the pre-treatment steps (Table 1). The investigated stormwater matrices include road runoff from highways and parking lots, as well as influent and effluent waters from stormwater treatment systems such as bioretention filters and stormwater ponds. Below, the findings of the present study are evaluated in the context of the studies summarised in Table 1. The discussion first focuses on studies employing mass-based

Table 1 Overview of studies addressing quantification of MP in stormwater. Reported concentrations have been rounded to two significant figures

Study	Sampling	Number of samples	Particle size [µm]	Pre-treatment	Density separation	Analysis method	Polymer concentration
Present study Sweden, rural Highway	Highway runoff (AADT 12,000). 3 ISCO samplers, 2 rain events, automatic sequential sampling	19 samples	10–1000	Filtration, ultrasonication, KOH	$CaCl_2$	Pyr-GC/MS, 13 polymers[a]	SBR > PE> PI > PP > > PVC > PB> ABS Max (Mean[d]) µg/L: SBR 1800 (130), PI 1000 (55), PE 1400 (91), PP 55 (14), PVC 17 (< LoQ), PB 3.6 (< LoQ), ABS 1.4 (< LoQ)
Cho et al., [22] South Korea, Gumi	Stormwater from industrial and residential, 3 rain events, time- proportional sampling	6 L composite, 44 samples	> 20	Filtration, Fenton	Li_2WO_4	FTIR, 27 polymers	Estimated event mean concentration µg/L: industrial 35–776, residential 40–450; Industrial: PP > PE > PET/PES = polyacrylate Residential: PP > PE > polyacrylate
Criollo et al., [28] Norway, highway	Highway runoff drained to a tank (6000-20,000 vehicles/day).	Filtration device close to the outlets, 3 samples, volumes 9–42 L	> 10	Filtration, ultrasonication, SDS solution, Fenton, NaOH	Sodium polytungstate	µ-FTIR, Pyr-GC/MS (TWP based on SBR + BR)	Estimated total MP (without TWP) µg/L: 0.4–12 TWP µg/L: 270–1500 PP > PA> PE > PVC & acrylic
Gaggini et al., [18] Sweden, rural highway	Highway runoff (AADT 12,000). Grab samples from stormwater system	3 occasions, 30 samples	1.6–500	Ultrasonication, sieving	-	Pyr-GC/MS (SBR and BR)	SBR + BR: 63–1000 µg/L
Galfi et al., [47] Galfi and Carvalho Nejstgaard, [71] Sweden, urban stormwater	Runoff from inlet and outlet of bioretention filter. Mixed area (roads and construction sites, 12,000 vehicles per year). ISCO samplers, automatic flow proportional sampling, 3 rain events	4 composite, 10 discrete	> 10	Ultrasonication, Fenton	ZnCl	TED-GC/MS, 10 polymers[b]	PET > PVC > PS > PP=PA6 Max µg/L: PET 850, PVC 700, PS 100, PP ca. 5, PA6 3
Galfi et al., [47] Galfi and Carvalho Nejstgaard, [71] Sweden, urban stormwater	Runoff from inlet and outlet of bioretention filter. Mixed area (roads and construction sites, 12,000 vehicles per year). ISCO samplers, automatic flow proportional sampling, 3 rain events	10 composite, 10 discrete	> 27	Filtration, ultrasonication, KOH	$CaCl_2$	Pyr-GC/MS, 10 polymers[c]	PE > PI = PB > PP> PVC > PS> PET = PC Max µg/L: PE 201, PI 97, PB 92, PP 67, PVC ca. 28, PS ca. 16, PET ca.5, PC 2
Jakubowicz et al., [72] Poland, stormwater, Gdansk	Influent to sedimentation tank	Filtration device UFO	> 10	-	-	FPA-µFTIR + ATR-FTIR	Estimated µg/L: Total MP 0.390, 0.0065–0.105 each Alkyd > PE> Polyester > PP

Table 1 (continued)

Study	Sampling	Number of samples	Particle size [µm]	Pre-treatment	Density separation	Analysis method	Polymer concentration
Johansson et al., [44] Sweden, pre-settled urban stormwater	Mixed area including urban highway (AADT 105,000). Samples collected in ~1m^3 composite. Sampling May to August.	7 composite	10–1000	Filtration, ultrasonication, KOH	$CaCl_2$	Pyr-GC/MS, 10 polymers[c]	PE > PI> PVC > PB> PP > PS> PET > PC Max (Mean) µg/L: PE 180 (74), PI 150 (45), PVC 120 (26), PB 88 (24), PP 30 (7.7), PS 10 (2.2), PET 1.3 (0.19), PC 1.2 (0.17), PA6 < LoQ, PMMA < LoQ
Johansson et al., [45] Sweden, pre-settled urban stormwater	Mixed area including urban highway (AADT 105,000). Samples collected in ~1m^3 composite. Sampling January to March.	9 composite	10–1000	Filtration, ultrasonication, KOH	$CaCl_2$	Pyr-GC/MS, 13 polymers[a]	Max (Mean) µg/L: PI 460 (68) > PB 250 (37) > PP 95 (30) > PE 130 (28) > SBR 140 (21) > PVC 47 (15) > PA 66 9.2 (5.3) > PMMA 2.8 (1.7) > PS 1.7 (1.4) > PET 0.8, ABS 0.5, PA6 0.3
Lindfors et al., [41] Sweden, urban stormwater	Runoff from parking lot, road (AADT 6400) and roof. One rain event	3 samples	> 10	Filtration, SDS ultrasonication, enzymatic digestion, Fenton	$ZnCl_2$	µ-FTIR, Pyr-GC/MS (TWP based on SBR + BR)	Estimated MP (without TWP) µg/L: 0.0185–114 PP, PE, Polyester, Acrylate, PU, PU paints, PVC, PA, PS, ABS TWP µg/L: 0.03–2300
Liu et al., [55] Denmark, stormwater ponds	Industrial, residential, and highway runoff from ponds, filtrated 722–1139 L	7 ponds	10–2000	SDS solution, ultrasonication, enzymatic digestion, Fenton	$ZnCl_2$	µ-FTIR	Estimated µg/L: 0.085–1.14, mean 0.23 PP > PVC> polyester > PE> PS> others
Rasmussen et al., [42] Denmark, stormwater ponds	Residential and industrial, 4 stormwater ponds	Filtration device close to the outlets, 4 samples each 560–1700 L	10–500, > 500	Ultrasonication, enzymatic digestion, Fenton	Na_2WO_4	FPA-FTIR, ATR-FTIR, Pyr-GC/MS (TWP based on SBR + BR)	Size 10–500 µm, estimated MP (without TWP) µg/L: 0.2–420 Size > 500 µm, estimated MP (without TWP) µg/L: 0.020–3500 PP > PE> polyester > PS> PA > ABS~PVC > PU> cellulose acetate > PVAC (by particle number) TWP µg/L: 0–22
Rosso et al., [68] Italy, rural Highway. stormwater	Highway runoff (AADT 71,000), 8 rain events, influent from 3 drains, effluent from stormwater treatment solution	2 sites, 16 samples	5–100	H_2O_2+SSO (oleo-extraction)		µ-FTIR	Estimated MP µg/l: 1900–45,000 (influent); 210–4700 (effluent) Influent: PA6, acrylic, vinyl ester, PPA, PE, PARA, PTFE, PP, PES (by particle number)

Table 1 (continued)

Study	Sampling	Number of samples	Particle size [µm]	Pre-treatment	Density separation	Analysis method	Polymer concentration
Rosso et al., [69] Italy, rural Highway stormwater	Highway runoff (AADT 71,000), 4 rain events, 3 drains	4 samples	5–100	H_2O_2+SSO (oleo-extraction)		µ-FTIR	Estimated MP µg/L: 930 – 3500 Vinyl ester, PA6, fluorocarbon (FC), PES and PTFE predominant
Treilles et al., [24] France, suburban stormwater, Paris	Suburban, 4 rain events, 3–5 consecutive samples of stormwater collected at catchment outlet (80–100 L)	18 samples	80–1000; 1000–5000	SDS+H_2O_2, ultrasonication	NaI	µ-FTIR + stereo microscopy; ATR	Size 80–1000 µm estimated µg/L: 56 (median) interquartile range 194 Size 1000–5000 µm estimated µg/L: 31 (median) interquartile range 22 PE, PP, and PS predominant

[a]Polymers ABS, PA6, PA66, PC, PMMA, PE, PET, PP, PS, PVC, PB, PI, SBR; [b]Polymers PA6, PC, PMMA, PE, PET, PP, PS, PVC, PI, SBR; [c]Polymers PA6, PC, PMMA, PE, PET, PP, PS, PVC, PB, PI; [d]In the calculation of mean concentration per polymer, concentrations < LoQ were assigned a value equal to one-tenth of the respective polymer-specific LoQ indicated in Table SI 2.

analytical techniques (Pyr-GC/MS or TED-GC/MS), as these provide the most directly comparable data to the present study. Then the comparison is extended to studies employing FTIR techniques, starting from the ones investigating road runoff, followed by those addressing stormwater from residential and industrial catchments, as well as stormwater ponds. Finally, the concentrations of tyre-related rubber polymers are discussed.

The studies by Johansson et al., [44, 45] employed mass-based analytical techniques and used the same analysis method with the same particle sizes of 10–1000 µm as the present paper. Johansson et [44, 45] analysed stormwater from the urban area of Gårda in Gothenburg, Sweden. The stormwater, which included runoff from a highway with an AADT of 105,000, was sampled after initial sedimentation in a chamber. In the pre-settled stormwater samples, all 10 or 13 analysed polymers were quantified at least once in the 16 analysed samples (Table 1). Overall, the MP concentrations for the quantified polymers appear to be in the same order of magnitude at Gårda [44, 45] and in the present study. The major polymers found in Gårda during summer were PE, PI and PVC [44]. Compared to the present study, polymers PE and PI were higher in some samples from Testsite E18 than in Gårda during summer, whereas PB, PVC and PS were considerably lower in the present study. The tyre-related polymer SBR, which was one of the major polymers quantified in the present study, was not analysed by Johansson et al., [44]. The pre-settled stormwater from Gårda sampled during winter in Johansson et al., [45] showed higher concentrations of PB, PP and PVC than the present study, while PE and SBR concentrations were lower (Table 1). In Gårda [44, 45] more polymer types, including PA6, PA66, PC, PET, PMMA and PS, were found in concentrations > LoQ compared with the present study. This was expected due to higher traffic load and a more diverse urban catchment with a broader range of MP sources in Gårda compared to Testsite E18, which is located in a rural area.

The influent runoff and effluent from a bioretention filter in Gothenburg, Sweden, were analysed by Galfi et al., [47] for MP with the same Pyr-GC/MS method as Johansson et al., [44], but with larger particle sizes > 27 µm, as well as with a TED-GC/MS methodology for MP > 10 µm, where SBR was included but not PB (Table 1). The total MP concentration in the influent runoff was 115–496 µg/L and was lower than in the present study, likely due to fewer polymer types being targeted and the larger particle size range analysed by Pyr-GC/MS. Concentrations of polymers PE, PI, and PB were also lower in Galfi et al., [47] than in the present study. For the tyre-related rubber polymers PI and PB, this difference is likely explained by the higher traffic load at Testsite E18 compared to the more urban catchment area of the bioretention filter in the study by Galfi et al., [47]. The polymer PP concentrations were similar between the studies, whereas PET and PVC concentrations were higher in the influent and effluent of the bioretention filter. This possibly reflects the more diverse MP sources in the catchment studied by Galfi et al., [47], which also included a construction site, compared to the present study, where the stormwater originated from the highway.

Moving on to studies estimating MP concentrations using FTIR (Table 1), a study of highway stormwater in Italy (AADT 71,000) estimated MP concentrations < 100 µm to 1900–45,000 µg/L [68] and 930–3500 µg/L [69]. These concentrations exceed those quantified in the present study and in Johansson et al., [44, 45], which may be due to the higher traffic load on the Italian highway. On the other hand, a Norwegian highway catchment

with traffic load similar to the present study showed substantially lower MP concentrations of 0.40–12 µg/L, excluding tyre-related polymers [28]. The reported MP concentrations in highway stormwater show substantial variability among studies, which may be due to differences in sampling and analytical approaches, particle size fractions analysed, site-specific factors and MP sources.

Concentrations of MP in stormwater during rain from residential and industrial areas assessed using FTIR showed similar concentrations as in the present study (Table 1), with a median concentration of 56 µg/L (MP > 80 µm) in a suburban residential area in Paris [24] and event-mean concentration 35–776 µg/L in Korean residential and industrial areas [22]. Similarly, stormwater collected from a roof, a car park, and a road in the central area of Luleå, Sweden, had estimated total concentrations of MP > 10 µm ranging from 0.019 to 110 µg/L ([41]; Table 1). The MP concentrations from Luleå were lower than in the present study and in runoff in Italy [68, 69], potentially due to the lower traffic load per surface area. PP and PE were among the most abundant polymers in Lindfors et al., [41] and Treilles et al., [24].

Studies reporting MP concentrations in stormwater ponds generally found lower concentrations than those found in the present study (Table 1). Liu et al., [55] reported 0.085–1.14 µg/L (> 10 µm), while Rasmussen et al., [42] reported 0.2–420 µg/L (10–500 µm) and 0.02–7.1 µg/L (> 500 µm), except for one pond with one MP concentration of 3500 µg/L. Comparison of the polymer types shows that, as in the present study, PE and PP were among the most abundant in stormwater ponds, together with PVC and polyester. Direct comparison between stormwater ponds and highway runoff is challenging due to the different hydrodynamic conditions. However, given the limited availability of mass-based MP concentration data in stormwater, comparisons among different stormwater matrices remain valuable for improving the understanding of MP occurrence and distribution.

The combined concentrations of SBR, PI and PB, identified in the present study, can be compared to the calculated corresponding concentrations in the samples of SBR + BR quantified in Gaggini et al., [51] (see Section Dataset on water quality and tyre-related polymers at Testsite E18 and Table SI 2). The latter quantification was performed using a Pyr-GC/MS methodology developed specifically for SBR + BR quantification for estimation of tyre wear particle concentrations using a combination of four marker, except for samples with high benzene levels in which the latter was excluded as a marker (see Section Dataset on water quality and tyre-related polymers at Testsite E18; Table SI 2, Gaggini et al., [51]; Rødland et al., [52]. Due to the different pre-treatment methodologies, the resulting particle size fraction is 1.6–500 µm in Gaggini et al., [51]. The synthetic rubber concentrations from the present study were, on average, nearly a factor of 1000 lower than the corresponding SBR + BR concentrations identified in Gaggini et al., [51]. Such discrepancies were also seen in three stormwater samples from the Gårda study [45], where the mean SBR + BR concentration of 5300 µg/L using the Pyr-GC/MS method by Rødland et al., [52] was 90 times higher than SBR and PB concentrations analysed with the present method. The comparison of the methodologies is challenging due to many differences, including variations in sample pretreatment, sample volume analysed, size cut-off thresholds and marker selection. The SBR + BR quantified for TWP analysis in the same samples as the present study found that the fraction 1.6–20 µm contributed to 47–100% of the total SBR + BR concentrations [51], and the importance of including particles < 20 µm has also been stressed by other studies [19, 70]. As the size cut-off for the MP analysis in the present study was 10 µm, the discrepancies in rubber polymer concentrations between the Pyr-GC/MS methodologies could indicate that most of the rubber polymers are smaller than this. However, given the large discrepancies observed, several factors are likely at play, including differences in sample pre-treatment, pyrolysis procedures, and markers used to quantify the polymers. Additionally, it has been reported that ultrasonication pre-treatment of water samples can increase the proportion of SBR + BR quantified in the fraction 1.6–20 µm [18]. This could indicate that the ultrasonication pretreatment step in the present study, and in several other studies (Table 1), may have caused break-up of TWP into sizes < 10 µm and therefore caused additional loss of particles during sieving.

Transport behaviour of MP during rain compared to TSS, VSS and metals

When comparing the MP concentrations between the rain events, sampled wells and over time from the start of the sampling, there is high variation between both concentrations and the types of polymers found (Table SI 2). The rain event on 2023-05-17 had a longer antecedent dry period, which, due to the higher traffic load, could be expected to generate higher MP concentrations (Section Study area); however, this was not the case, with similar total MP concentrations across the rains, except for well GP1 on 2023-04-29 in samples 17:02 and 17:04, which had the highest concentrations. As the rain on 2023-04-29 had higher rain intensity, MP may have been mobilised from the road to a larger extent, or possibly MP on 2023-05-17 had already been washed off by a light rain (1.8 mm/h) occurring on 2023-05-16. Also, no consistent decrease in MP concentrations among consecutive samples was observed. This is not in line with previous results on TWP from the same samples, where a decrease in concentration was observed with time from the onset of

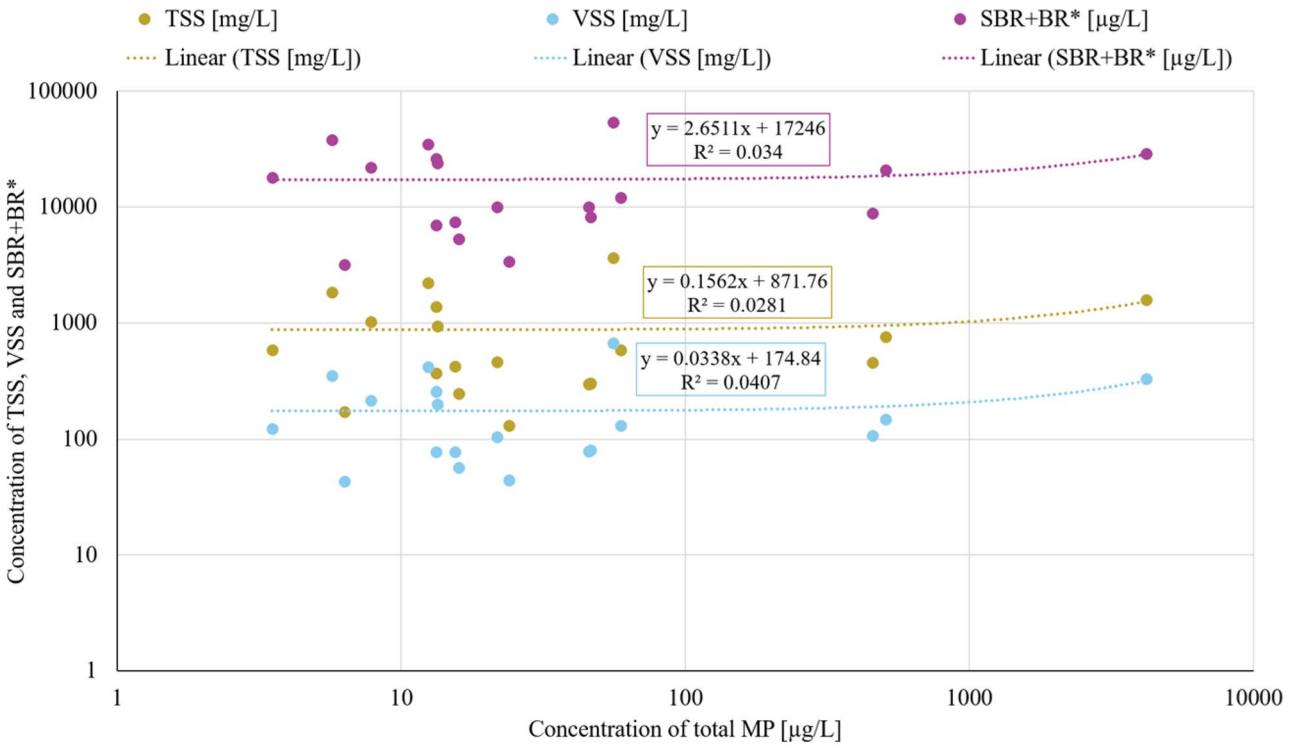


Fig. 4 Scatter plot of TSS, VSS and SBR + BR* concentrations (along the vertical axis) over the sum of MP concentrations (along the horizontal axis) for each sample, including rubber polymers, with their respective concentration units indicated in the legend. Dashed lines represent linear regression lines for each parameter, with the corresponding equation and R^2 value shown in the coloured boxes. (*) indicates calculated SBR + BR concentrations for the corresponding aggregated samples of the present study based on the results of Gaggini et al., [51]

rainfall at all locations and during both rain events [51]. Treilles et al., [24] also found that MP concentrations were highest just before the flow rate peak in a stormwater outlet, and that the concentrations were much lower at the end of the rainfall. However, no such pattern in MP concentrations was seen in stormwater from urban and suburban areas in the study by Ochoa et al., [73]. Large variations in MP concentrations during rain events were also reported by Galfi et al., [47], and overall high variability in MP concentrations in runoff from the same site across sampling occasions was seen in urban stormwater collected from a well [44]. The difference in behaviour can be due to the different topology of the area, rainfall characteristics, or particle properties.

The concentrations of MP quantified in the samples were compared to aggregated values from Gaggini et al., [51] for TSS and VSS. For location GP2, metal concentrations from rain on 2023-04-29 were also included. Total MP concentrations are shown alongside TSS and VSS in Fig. 4, with the corresponding linear regression results. No significant correlation was found between MP concentrations and TSS or VSS, neither for total MP concentrations nor for specific polymer types. Moreover, MP concentrations did not correlate with SBR + BR concentrations measured in Gaggini et al., [51] (see Section MP polymers in stormwater at Testsite E18). The metal concentrations in the aggregated samples decreased with time during the rain event (see Supporting Information Figure SI 3), consistent with samples analysed at higher temporal resolution measured in Gaggini et al., [51]. The metal concentrations most likely originate from traffic and have an impact on the stormwater quality, with high concentrations of Cu, Cr and Zn (exceeding guidelines for Gothenburg municipality at certain times [74], and of other traffic-related metals, such as Ba, which was also reported in roadside soil from the same location [75]. None of the quantified metals was found to correlate with total MP concentrations. The SBR + BR concentrations measured in the same samples from Gaggini et al., [51] showed strong correlations with TSS ($r > 0.9$), VSS ($r > 0.9$), and metals ($r > 0.7$), likely due to similar particle transport processes of solids and TWP.

The lack of correlations between the investigated parameters and the MP concentrations in the present study is potentially related to the greater variability in MP concentrations across samples and differences in MP characteristics (e.g. density). A strong correlation ($r > 0.8$) between MP and suspended solids was reported in stormwater in industrial and residential catchments [22], and between solids and SBR + BR concentrations in runoff [18], snow [76], and tunnel washwater [77]. On the other hand, no correlation with turbidity was observed

for MP in runoff from roads, parking lots, and roofs in the study by Lindfors et al., [41]. The inconsistency between findings could indicate that MP transport and mobilisation are heterogeneous and variable. It could also indicate that the transport characteristics are strongly site-specific. Furthermore, MP concentrations may be highly influenced by the occurrence of few, but relatively large plastic particles or fragments, which could substantially affect the measured mass-based concentration, increasing variability across samples. Additionally, current analytical limitations in MP quantification, such as the inability to quantify in fine size fractions and certain polymer types, may still hinder a comprehensive assessment of MP transport patterns.

Conclusions

This study provides valuable insights into the presence of MP > 10 µm, including tyre-related polymers, in runoff collected during rain events from a Swedish highway stormwater system. The results highlight a varying composition of MP in stormwater and emphasise the presence of certain polymers over others.

- Among the quantified polymers, PE, PP, SBR, and PI were most frequently identified, with concentrations ranging from < 0.1 to 1800 µg/L. The results suggest a significant presence of these materials, likely due to the high emissions of TWP and the widespread use of PE and PP in packaging materials, containers, and disposable products, as well as the growing use of plastic polymers in vehicle bodies.
- In contrast, polymers such as PS, PMMA, PC, PET, PA6, and PA66 were either quantified at minimal concentrations or remained < LoQ, indicating a lower contribution to stormwater in highways crossing rural areas. Additionally, ABS, PB, and PVC were quantified in only a few samples, further highlighting the variability in polymer presence in stormwater.
- The tyre-related polymers were quantified in considerably lower concentrations than those reported in other studies, with PB ranging from < 1.0 to 3.6 µg/L, PI from < 1.0 to 1000 µg/L, and SBR from < 0.2 to 1800 µg/L. The reported concentrations in the present study do not account for particles < 10 µm, which may result in an underestimation of TWP polymer concentrations in the samples. Improved methods are needed, both in terms of standardisation and harmonisation of pretreatment methods, analytical methodologies including markers for Pyr-GC/MS, as well as improved capability to analyse MP finer than 10 µm, since other studies suggest that TWP may be abundant in the finer fractions.
- Comparison with other studies showed that the findings were consistent with reported results on the occurrence and concentrations of MP in stormwater, while also highlighting the lack of studies quantifying mass concentrations of MP in environmental matrices.
- The MP concentrations showed high variability between samples, no correlations with TSS, VSS, and metals, as well as no temporal trend during the rain events, indicating that MP transport may be governed by multiple interacting factors, including rainfall characteristics, catchment conditions, and particle properties.

Overall, the findings contribute to the growing body of research on MP and TWP pollution in built environments, highlighting the need for continued development of monitoring and mitigation efforts. Understanding the distribution of these materials in stormwater can inform future environmental policies and infrastructure strategies to reduce plastic contamination in aquatic ecosystems. Further research should address the development of standardised sampling techniques and sample preparation methodologies for Pyr-GC/MS that can reliably detect the finest size fractions of MP and are robust against interference from complex environmental matrices. These advancements are needed for effective environmental monitoring and will support more consistent investigations into the transport dynamics and long-term ecological effects of MP, as well as the evaluation of effective stormwater remediation strategies.

Abbreviations

AADT	Annual average daily traffic
ABS	Acrylonitrile butadiene styrene
ATR-FTIR	Attenuated total reflectance Fourier transform infrared spectroscopy
BR	Butadiene rubber
$CaCl_2$	Calcium chloride
FTIR	Fourier transform infrared spectroscopy
GP	Gully pot
KOH	Potassium hydroxide
LoQ	Limit of quantification
MP	Microplastics
PA6	Polyamide 6
PA66	Polyamide 6,6
PB	Polybutadiene
PC	Polycarbonate
PE	Polyethylene
PET	Polyethylene terephthalate
PI	Polyisoprene
PMMA	Polymethyl methacrylate
PP	Polypropylene
PS	Polystyrene
PVC	Polyvinyl chloride
Pyr-GC/MS	Pyrolysis-gas chromatography / mass spectrometry
SBR	Styrene-butadiene rubber
SBR + BR	Combined styrene-butadiene rubber and butadiene rubber
SEM	Scanning electron microscopy
SiO_2	Silicon dioxide
TED-GC/MS	Thermal extraction desorption-gas chromatography/mass spectrometry

TSS	Total suspended solids
TWP	Tyre wear particles
VSS	Volatile suspended solids
WA	Collection well (sampling location)
μ-FTIR	Micro-Fourier transform infrared spectroscopy

Supplementary Information

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Supplementary Material 1

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Author contributions

E. L. G. conducted the formal analysis and investigation, developed the methodology, contributed to funding acquisition, and wrote the original draft. A-M. S. contributed to conceptualisation, methodology, and writing of the original draft. E. R. contributed to methodology and manuscript revision. E. S. contributed to conceptualisation, methodology, funding acquisition, project administration, supervision, and manuscript revision. Y. A-S. contributed to conceptualisation, methodology, supervision, and manuscript revision. M. B. contributed to conceptualisation, methodology, funding acquisition, project administration, supervision, and manuscript revision. All authors reviewed and approved the manuscript.

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Data availability

All data supporting the findings of this study are available within the paper and its Supplementary Information.

Declarations

Competing interests

The authors declare no competing interests.

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