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Research article

Quantification and characterization of additives, plasticizers, and small microplastics (5–100 μm) in highway stormwater runoff

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ABSTRACT

Highway stormwater (HSW) runoff is a significant pathway for transferring microplastics from land-based sources to the other surrounding environmental compartments. Small microplastics (SMPs, 5–100 μm), additives, plasticizers, natural, and nonplastic synthetic fibers, together with other components of micro-litter (APFs), were assessed in HSW samples via Micro-FTIR; oleo-extraction and purification procedures previously developed were optimized to accomplish this goal. The distribution of SMPs and APFs observed in distinct HSW runoff varied significantly since rainfall events may play a crucial role in the concentration and distribution of these pollutants. The SMPs' abundance varied from 11932 ± 151 to 18966 ± 191 SMPs/L. The dominating polymers were vinyl ester (VE), polyamide 6 (PA6), fluorocarbon, and polyester (PES). The APFs' concentrations ranged from 12825 ± 157 to 96425 ± 430 APFs/L. Most APFs originated from vehicle and tire wear (e.g., Dioctyl adipate or 5-Methyl-1H-benzotriazole). Other sources of these pollutants might be pipes, highway signs, packaging from garbage debris, road marking paints, atmospheric deposition, and other inputs. Assessing SMPs in HSW runoff can help evaluating the potential threat they may represent to receiving water bodies and air compartments. Besides, APFs in HSW runoff may be efficient proxies of macro- and microplastic pollution.

Author contribution

Beatrice Rosso: Methodology, Validation, Investigation, Data curation, Visualization, Writing – original draft; Writing - review & editing. Fabiana Corami: Conceptualization; Methodology; Validation; Formal Analysis, Visualization, Writing - original draft, Writing - review & editing, Supervision. Luca Vezzaro: Resources; Writing - review & editing. Stefano Biondi: Resources; Barbara Bravo: resources, writing—review & editing Carlo Barbante: Supervision, Writing - review & editing; Andrea Gambaro: Supervision, Resources, Writing - review & editing.

1. Introduction

Urban runoff and highway stormwater (HSW) runoff contribute significantly to water pollution and are widely recognized as significant contributors to the deterioration of receiving water quality (Müller et al., 2020). They are the most significant non-point source of pollution and a direct pathway from land-based sources to freshwaters and groundwater, posing risks to aquatic and terrestrial organisms (Koutnik et al., 2022; Liu et al., 2019; Monira et al., 2021). HSWs can collect different pollutants from road dust, consisting of a heterogeneous mixture of particles that are generated by the physical abrasion of organic and inorganic materials generated through various anthropogenic activities, especially traffic vehicles (Pateraki et al., 2019; Wang et al., 2022). Among the toxic chemicals and the hazardous pollutants in

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road dust and HSW, particles originating from tires, rubbers, and microplastics can be observed, together with trace elements (e.g., arsenic, chromium, mercury, etc.), oils, fuel and antifreeze additives (Brudler et al., 2019; Monira et al., 2021; Polukarova et al., 2020; Heidari et al., 2021). While the abundance and distribution of microplastics >100 μm have been often studied in the literature (Lange et al., 2021; Lutz et al., 2021; Braga Moruzzi et al., 2020; Werbowski et al., 2021), those in the range of 5–100 μm (small microplastics, SMPs) on highway HSW still appear to be poorly investigated and evaluated. Wastewater treatment plants (WWTPs) may not retain SMPs due to their sizes; hence, these pollutants can significantly affect the environment and the biota, as organisms can ingest them (e.g., invertebrates; Corami et al., 2020b; Hamidian et al., 2021; Ivyer et al., 2020; 'O'Connor et al., 2019).

Principal sources of MPs and SMPs in highway SW pollutants can come from vehicular traffic, tire wear, fluid leakage, polymers used to improve the bitumen, elastomers used in road and building marking paints, pavement degradation, roadway maintenance, littering, and atmospheric deposition. These sources of pollution can be temporary, seasonal, accidental, or chronic (Piñon-Colin et al., 2020; Monira et al., 2021; Roychand and Pramanik, 2020). The wear-out of tires during driving, acceleration, and braking can be washed away from highways and roads' impervious surfaces during a rainfall event and is among the most relevant sources of primary MPs in the environment, with pathways implication for aquatic, terrestrial, and atmospheric compartments (Knight et al., 2020; Roychand and Pramanik, 2020; Baensch-Baltruschat et al., 2020). A tire's composition depends on the brand and typically contains rubbers, e.g., polyisoprene, styrene-butadiene rubbers, plastic fibers, and other components, such as oils, plasticizers, vulcanization agents, antioxidants, and other several additives (Baensch-Baltruschat et al., 2020). Additives and plasticizers are added to polymers during their manufacturing and are used to convert or confer specific characteristics to the polymers. These substances can be released into the aquatic and terrestrial compartments when macroplastics (including tires) and SMPs are dispersed in the environment, causing potential adverse effects on humans and biota (Beiras et al., 2020; Turner and Filella, 2021). Additives and plasticizers can be employed as proxies for the presence of plastic polymers in the environment. These compounds and other microlitter components (e.g., nonplastic sìynthetic fibers, APFs) should be assessed together with SMPs (Corami et al., 2021; Rauert et al., 2022).

Because of the lack of a fully standardized method and harmonized techniques for sampling, pretreatment, contamination, quantification, and characterization of SMPs from highway SW runoff, there are significant differences in the data of these studies (Liu et al., 2019a; Müller et al., 2020; Olesen et al., 2019, Pramanik et al., 2020; Piñon-Colin et al., 2020; Shruti et al., 2021; Tan et al., 2022; Werbowski et al., 2021; Windsor et al., 2019; Ziajahromi et al., 2020). Most studies did not report the abundance of polymers, and plastic particles were randomly selected and identified (Shruti et al., 2021). Furthermore, MPs >100 μm were mainly investigated, while SMPs are overlooked entirely, and so are additives and plasticizers released into the environment by the breakage of macro-, meso- and large microplastics (Mammo et al., 2020). Thus, investigating the presence of SMPs and APFs in HSWs with standardized, robust, and reproducible methods is crucial for toxicological studies of these classes of pollutants, for risk assessment, but also for proposing future bills and regulatory thresholds (Khan et al., 2022; Stang et al., 2022; Winquist et al., 2021).

The first goal of this study is to investigate the abundance and distribution of SMPs (5–100 μm) additives, plasticizers, nonplastic synthetic and natural fibers, and other components of micro-litter (APFs) in HSW runoff. This is the first study to investigate the potential variability of the abundance and presence of SMPs (5–100 μm) and APFs in HSW runoff collected during distinctive rainfall events. The results will improve the knowledge of these pollutants HSWs, which is needed to design future sustainable treatment facilities and management, evaluating potential solutions in stormwater systems worldwide since the high presence and the use of the freeway network every day in different countries (e.g., total length of Europe freeway network is approximately 136,700 km, while in Italy the highway system extends for 6.942,7 km (EU 2020; ANISA 2019)).

Due to the lack of standardized methods for analyzing this environmental matrix, a pretreatment method (i.e., extraction and purification, Corami et al., 2021) to extract SMPs and APFs was optimized; these pollutants were simultaneously quantified and identified via Micro-FTIR. Particular attention has been focused on soft pretreatment procedures, i.e., procedures that have not used strong oxidizing agents, strong acids, and high temperatures because the identification and the quantification of SMPs can be impaired due to the complete loss of polymers such as polyamide or polyethylene, and phenomena of yellowing, denaturation, etc. (Al-Azzawi et al., 2020). Further degradation and denaturation, as well as the eventual loss of the polymers and compounds, should be prevented to avoid getting suboptimal recognizable IR spectra and consequently underestimates in the quantification (Corami et al., 2020a, 2021).

2. Materials and methods

2.1. Sampling

HSW runoff was collected during different rainfall events from January to March 2021 from a trafficked highway, namely Passante of Mestre on the mainland near Venice in Italy (Casale sul Sile, Treviso, Italy; Fig. S1a in the supplementary information). It is a section of the A4 motorway 32.3 km long, with average traffic of about 71,000 vehicles per day, including more than 20,000 heavy vehicles. This highway's sector is an essential connection between industrialized zones that cross environmentally sensitive areas; hence, it is a perfect area to study SMPs and APFs from a trafficked highway in SW runoff. Before sampling, numerous explorations had been performed to select the right equipment and position of sampling devices along the highway (slope, distances from the drains, and technical operations). The number and shape of the sampling devices employed were carefully tested to collect rainfall events greater than 5 mm, avoid potential overflow during the whole rainfall event, and collect the pollutants under exam. The sampling site was located in three different drains along the highway. The stormwater runoff fluxes discharge inside the drains connected with a first-flush SW treatment plant (SWTP). Then, the outlet of this SWTPs flows directly into a receiving surface water body. Three decontaminated glass flasks (1 L volume each flask) were located in each drain before a rainfall event to collect the stormwater runoff at each inlet and are a representative bulk sample. The flasks were hung inside the drains with clean iron wire (Fig. S1b). After the rainfall, the flasks were recovered and carefully transported to the laboratory. Technical operations, authorization access, and remote control are supported by StormWater Italia (SWI - Marghera-Venice) company and CAV (Concessioni Autostradali Venete) of Venice, Italy.

2.2. QA/QC

Several measures were taken to minimize contamination during sampling, transport of samples, pretreatment, and analysis. The operators wore cotton lab coats and nitrile gloves during sampling. The SW samples were collected carefully in previously decontaminated glass flasks covered with aluminum foil until they arrived at the plastic-free Clean Room ISO 7, a controlled laboratory made of inox steel and without plastic polymers even in the air filters. Pretreatment procedures (i.e., extraction, purification, and filtration) were carried out in the plastic-free Clean Room ISO 7 to minimize any contamination. All the pretreatment procedures and filtration were performed under a decontaminated steel fume hood, with the operators wearing cotton lab coats and nitrile gloves. All the steelware and glassware were previously

rinsed with UW, decontaminated with methanol, a 50% (v/v) solution of methanol and ethanol, and finally with ethanol. After filtration, all filters were stored in decontaminated glass Petri dishes covered with aluminum foil. Before the analysis, filters were transferred from the fume hood in the cleanroom to the Micro-FTIR laboratory, carefully covered with aluminum foil to avoid any external contamination. Reagent blanks (i.e., ultrapure water (UW), methanol, ethanol, hydrogen peroxide (H_2O_2)) and procedural blanks were tested for the presence of SMPs or APFs.

A recovery test was performed by spiking three further replicates of one of the HSW samples under exam with silver-grey PA 12 (polyamide 12) particles.

The list of all the reagents employed in the pretreatment and the decontamination is in the supplementary information (Table S1).

2.3. Oleo-extraction, purification, and filtration of HSW runoff

Density separation and flotation are the most employed pretreatment procedures for MPs in seawater, sediments, and stormwater samples (Nabi et al., 2022; Razeghi et al., 2022). This extraction technique exploits the difference in density between the polymers and the solution; however, several limitations can affect the estimation of SMPs, such as the limitation of the density polymer retrieved with the use of different density solutions, especially for the high-density polymers, the presence of the possible residues from the density solution may be cover the presence of SMPs on the filter (Corami et al., 2021), and the cost-safety used of some of these solutions (e.g., toxicity, and corrosiveness) with time-consuming limitations (Monteiro and Pinto da Costa 2022; Nabi et al., 2022).

In contrast, the oleo-extraction procedure allows a simple and safe method to extract SMPs from complex samples (such as water with a high presence of organic matter or sediment) based on the oleophilic properties of plastics. This extraction procedure allows retrieving polymers in a wide density range, from PE to fluorinated plastics (2,2 g/cm3), with the consequent accurate identification of polymers and other components of micro-litter with optimal identification. Then, the purification procedures allowed cleaning the retrieved particles and the filter from organic and biological impurities (including oil residues), avoiding degradation/denaturation of SMPs.

In this work, the pretreatment procedure, i.e., the oleo-extraction and purification procedure employed for HSW samples, was optimized from the procedure previously developed by Corami and coworkers at the Institute of Polar Sciences CNR-ISP (2021).

Three tests were performed, and each test was run in triplicate to achieve pretreatment optimization. First, HSW samples were thoroughly homogenized by inversion. In the first test, 200 ml of the SW sample were poured into a decontaminated flask. Then, 100 ml of UW and 5 ml of H_2O_2 were added to pseudo-digest the organic matter in the samples and avoid spectral interferents. The flasks were stirred for 3 h on a multipurpose orbital shaker at room temperature. The stirred aliquots were poured into 500 mL separating funnels, adding 7 ml of SSO to extract SMPs and APFs. After stirring the separatory funnels manually for 10 min, they were left to rest for at least 3 h for the complete separation of phases. After this time passed, the aqueous phase was collected in another decontaminated separating funnel. The oleo phase was recovered in a decontaminated Erlenmeyer flask with 20 mL of hexane and 20 mL of ethanol. In the second separating funnel, 5 mL of SSO were poured together with 5 mL of H_2O_2, and the oleo-extraction was repeated a second time. Then, the water was discarded, and the oleo phase was recovered again with 20 mL of hexane and 20 mL of ethanol and poured into the same Erlenmeyer flask.

In the second test, the volume of the HSW sample was 150 ml, to which were added 7 ml of SSO, 15 ml of H_2O_2, and 100 ml of UW; in the third test, the volume of the HSW was 100 ml, to which were added 7 ml of SSO, 30 ml of H_2O_2 and 100 ml of UW. The second oleo-extraction was performed as in the first test.

The oleo-extracts were then filtered under the fume hood using a decontaminated glass vacuum filtration system (VWR International, Milan, Italy) and aluminum oxide filters. The filters were purified during the filtration. Before the filtration, filters were rinsed following the procedure developed by Corami et al. (2020a), 2021), employing a 70% solution of ethanol-methanol, ethanol, and UW (ratio 2.5:1:5). The oleo-extracts were filtered alternating hexane, 70% solution of ethanol-methanol, and ethanol. Reagent and procedural blanks were pretreated and filtered in the same way.

After filtration, filters were left to dry in the cleanroom for at least 72 h at room temperature in previously decontaminated glass Petri dishes, which were covered with decontaminated aluminum foil. The filters were mounted on the Micro-FTIR stage and brought to the instrument for analysis, covered with decontaminated glass Petri dish halves coated with decontaminated aluminum foil.

2.4. Quantitative analysis, polymer identification, and data analysis of SMPs and APFs using Micro-FTIR

Quantification and simultaneous particle identification were performed using Micro-FTIR Nicolet™ iN™ 10 (Thermo Fisher Scientific), as previously described by Corami et al. (2020a), 2020b, 2021). The Micro-FTIR comprises an ultra-fast motorized stage and liquid nitrogen-cooled MCT detector (mercury cadmium telluride detector).

The analyses for quantification (microscopic counting) and simultaneous identification of SMPs and APFs were performed on transmittance mode employing the PARTICLE WIZARDS section of the Omnic™ Picta™ software. For microscopic counting (Corami et al., 2021, 2022), at least 20 known-sized areas (i.e., count fields 2000 μm × 1200 μm) were randomly chosen with no overlapping. Microscopic counting can be employed with any filter suitable for analysis using Micro-FTIR and Micro-Raman (in this work, alumina oxide filter). The software section PARTICLE WIZARDS detects the particles and fibers present on the surface of each count area of the filter in relation to the brightness value (the brightness ratio of each particle in contrast to the background) and the size of the particles retrieved.

Since measuring complete filters is significantly time-consuming (Corami et al., 2021; Huppertsberg and Knepper, 2018; Imohf et al., 2016), counting areas or counting fields need to represent the entire filter to avoid issues regarding representativeness and reproducibility. Appropriate microscope conversion (optical factor F) and volume or dilution factors must be applied to the total particles retrieved from all the counting fields to provide absolute abundance (Corami et al., 2021).

For robust quantification to occur, a significant number of particles on the filter must be selected on each counting field. The image intensity histogram of the PARTICLE WIZARDS allows the selection of particles. For each particle selected, a spectrum is retrieved and compared to spectra in several reference libraries (similarity mode); the complete list of reference libraries is in the supplementary information. The identification of each spectrum is expressed as match percentage (match %). Only spectra with a match percentage $\geq$65% (optimal identification) were considered as identified and then counted for quantification. Besides, the imaging of PARTICLE WIZARDS allowed retrieving the length and width of each particle analyzed. Spot punctual analyses were performed to double-check the identification of some SMPs or APFs, especially those with matches near the limit of 65%.

In this work, on each count field, an average of 300 particles was selected by employing the WIZARDS section; then, for each particle, 64 co-scans were collected (spatial resolution 100 μm, aperture 100 μm × 100 μm, spectral range 4000–1200 cm−1). Abundance (SMPs/L and APFs/L), particles' weight (μg/L), and aspect ratio (AR) were evaluated according to the equations in Corami et al. (2020a, 2021; see also supplementary information).

The precipitation intensity was retrieved from the Regional Agency for Environmental Protection in Veneto (ARPAV, 2022) networks from the Favaro Veneto station.

2.5. Statistical analyses

SMPs and APFs abundance data follow a Poisson distribution, and Poisson's confidence interval was calculated accordingly (Corami et al., 2020b, 2021). Statistical analyses were performed using STATISTICA software (TIBCO, Palo Alto, CA, USA). The homogeneity of the variances of SMPs and APFs abundances was tested (Fisher exact test, F test; $\alpha = 0.05$). After ascertaining the non-homogeneity of the variances, non-parametric statistical tests were performed to evaluate significant differences in the abundances of SMPs and APFs. While the Kruskal-Wallis test ($p < 0.05$) was employed for multiples comparison, the Mann-Whitney *U* test ($p < 0.05$) was used for pairwise comparisons.

3. Results and discussion

3.1. Optimization of the oleo-extraction and purification of SMPs and APFs in stormwater runoff

Once the efficiency of the sampling system was verified (representative volume collected), oleo-extraction and purification were then optimized from the pretreatment developed by Corami et al. (2021). Several tests were necessary before obtaining a filter, where the particles under investigation could be concurrently characterized and quantified without interferents. The first test resulted in a filter surface completely covered with black particles, e.g., asphalt, bitumen, and other organic and inorganic interferents, below which SMPs and APFS might have been present but were not analyzable. The second test allowed retrieving the spectra of a few particles (Fig. 1a); however, the match percentage was <65%, and the particles could not be adequately quantified. The last test filters could be analyzed using Micro-FTIR (Fig. 1b); the spectra of the particles were collected, with an average match percentage of 79% and a maximum match percentage of 90% (Fig. S2 in supplementary information).

Purification allowed removing any interferents. The optimized pretreatment reduced the imaging LOD (limit of detection) from 7-10 μm–5 μm and allowed a proper quantification, avoiding loss and/or further denaturation/degradation of polymers (Al-Azzawi et al., 2020; Corami et al., 2021).

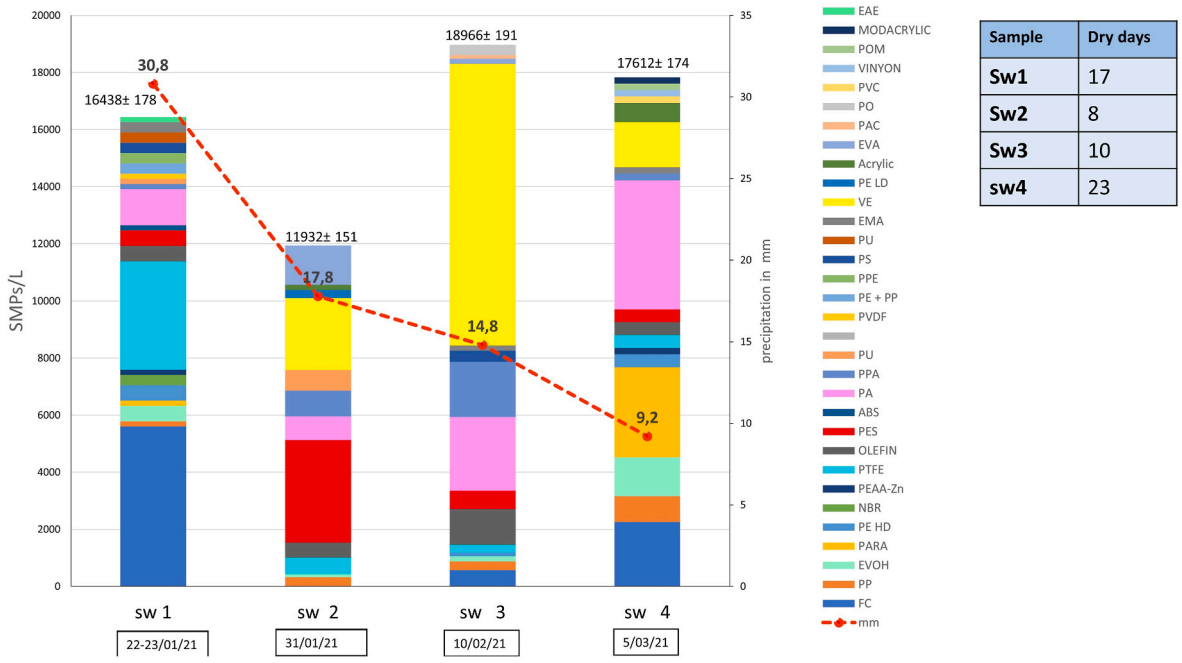
Validation of analytical methods for measurements of microplastics (MP) is crucial to assess the accuracy of the extraction, purification, quantification, and identification method to ensure the reliability of the results; however, there is a general lack of reference materials used in MPs and SMPs studies (Ivleva 2021; Yusuf et al., 2022). In this study, purchased particles of PA 12 were employed as a reference since polyamides are present in different environmental matrices, such as biota, sediments, water, and atmosphere (Abbasi et al., 2023; Corami et al., 2021; Kor et al., 2020, Sfriso et al., 2020). The average yield of the recovery test was 94%; hence, the pretreatment was accurate and replicable.

Tire wear particles had not been unambiguously identified due to the complex chemical composition of tires that varies between typologies and brands. A tire is commonly made up of several layers, including textiles, e.g., nylon (polyamide 6, PA 6), polyester (PES), and rayon, used for belt cords that reinforce the tire, which contributes to wear, handling, stability, and traction (Andersson-Sköld et al., 2020). Black carbon added to the tires as a filler, together with oils and silica residues, vulcanizing agents, softeners, plasticizers, and other additives, is absorbent throughout the infrared region, making spectrum acquisition challenging (Baensch-Baltruschat et al., 2020; Kole et al., 2017; Liu et al., 2019; Luo et al., 2021). Nevertheless, this study successfully identified and quantified several tire polymers, additives, and plasticizers. However, a suitable method to identify and quantify all the polymers, additives, and plasticizers in tires and employ them as markers of the presence of tire wear particles is needed; cross-validation techniques can be handy for the purpose and are the focus of our future study.

3.2. Quantificdation and identification of SMPs in highway stormwater runoff

The abundance of SMPs, found in all the collected HSW runoff is shown in Fig. 2; since they followed Poisson distribution, the confidence limit (error) for the abundance of each site is reported. The complete list of identified polymers and their acronyms is reported in Table 1.

Fig. 1. **a**First test of the oleo-extraction and purification procedure optimized from the pre-treatment developed by Corami et al. (2021). This test resulted in a filter surface completely covered with black particles, e.g., asphalt, bitumen, and other organic and inorganic interferents, below which SMPs and APFS might have been present but were not analyzable. b The last test filters could be analyzed using Micro-FTIR, and the spectra of SMPs and APFs were collected.



Sample	Dry days
Sw1	17
Sw2	8
Sw3	10
sw4	23

Fig. 2. Abundance (SMPs/L) and polymer distribution of SMPs in highway stormwater runoff. Abundances, errors (fiducial interval according to Poisson Distribution), mm of precipitations, and the number of dry days are reported.

The highest abundance was detected in SW3, while the lowest was observed in SW2. As regards the weight of the SMPs (Fig. S1 in supplementary information), the minimum weight was observed in SW3 (925.5 ± 42 μg/L), while SW4 showed the maximum value (3486.5 ± 82 μg/L). Particle weight is most influenced by the single polymer density and size (Fig. S3, supplementary information). VE, PA 6, FC, PES, and PTFE were predominant in all samples, with a few exceptions (e.g., SW1). AR (Fig. S4 in supplementary information) showed that ellipsoid (E) was the most common shape among SMPs in these HSW samples. According to our data, most SMPs present in HSW are smaller than 100 μm; the length ranged from 25,0 μm to 80,4 μm, while the width range varied from 14.5 μm to 43.4 μm, with very few exceptions (Table S3, supplementary information). According to their size, these particles may not be retained by SWTPs and may enter the environment, as already observed in WWTPs (Corami et al., 2020a, Iyare et al., 2020). Once released into the environment, these particles can pose a threat to terrestrial and aquatic organisms since they can enter the trophic web and be ingested by biota (Corami et al., 2020b; Iannilli et al., 2019; Mak et al., 2020; Olesen et al., 2019). It should be noted that some polymers were identified only in specific SW samples (e.g., SW4, POM, PVC, and VE). Before a rainfall event, the most prolonged dry period might have influenced the transport and/or the accumulation of different polymers in the road dust, which was washed away with the next rainfall.

The variances of the samples were not homogenous (F test, $\alpha = 0.05$); thus, non-parametrical statistical tests were applied. The Mann-Whitney U test ($\alpha = 0.05$) showed that the abundance of SMPs in the samples under exam was significantly different. These differences were highly statistically significant according to the Kruskall-Wallis test ($p < 0.01$).

The intensity in mm of rainfall was then compared with the SMP's abundance in the corresponding HSW runoff. In all the SW samples analyzed, the abundance of SMPs was high, corroborating that the SW runoff can be a primary contributor of SMPs to freshwater and groundwater bodies (Pinon Colin, 2020; Monira et al., 2021). The rain intensity of four consecutive events from the end of January to March 2021 decreased, being the highest in the first rainfall event (January 2021) that occurred after a long dry period.

SMP's abundance in HSW samples may be related to the abundance of these particles in the road dust and the atmospheric deposition. Extended dry periods may result in increased concentrations of pollutants and SMPs reaching the HSW runoff since they include atmospheric deposition, gravitational sedimentation of solid materials, micropollutants, and SMPs. The same is valid for road dust. The subsequent rainfall collects all the pollutants in road dust and the atmosphere, thereby increasing their concentration in the HSW runoff.

However, it should be noted that, besides the scavenging effect of rainfall, other meteorological parameters, e.g., wind intensity and direction, together with the intensity and quantity of vehicular traffic, may contribute to the increment of pollutants and SMPs in HSW runoff. The combination of all these factors together can account for the trend observed in the samples under examination.

Vehicular traffic is among the most significant sources of MPs and SMPs in SW runoff; plastic polymers, e.g., VE, PTFE, PU, ABS identified in the samples investigated, can be employed in various applications in vehicles, from tires to car chassis, seats, cooling systems, or even in engine and vehicle parts and gaskets.

Together with the wear and tear of vehicles and vehicular traffic, tire wear is another most relevant source of SMPs in SW runoff. Although styrene-butadiene rubber (SBR) is considered the most extensively used rubber in tires, it was not irrefutably identified; however, particles of other rubber polymers were identified (NBR, PS, and PU), potentially residues from the chemical mixture of tire wear released on the highway. As aforementioned, tire wear particles are difficult to be identified with FTIR techniques (3.2 section). These results show that a potential marker for analyzing tire wear particles is needed for future research.

All the sources listed above can be referred to as continuous because they are always available at any time and period of the year, just like the atmospheric deposition. The latter contributes substantially to a load of plastic particles, especially SMPs, in road dust and SWs in general (Mbachu et al., 2020). Besides vehicular traffic, which is the most relevant source of SMPs for road dust and SW runoff, other sources can also be significant. Road marking products consist of different plastic polymers (e.g., EVA, PA 6, and PU), pigments, fillers, and additives; they may result in road marking wear particles (RWPRMs) from weathering and traffic activities. RWPRMs are an overlooked component of MPs and SMPs in the environment; these particles may leach high levels of phthalates and other toxic additives and plasticizers, leading to the

Table 1
List of the complete names and their acronyms SMPs and the most abundant APFs observed in SW samples investigated.

SMPs	Abbreviations
Acrylonitrile butadiene styrene	ABS
Acrylic polymers	Acrylic polymers
Ethylene methyl acrylate	EMA
Ethylene methylacrylic elastomer	AEM
Ethylene-vinyl acetate	EVA
Ethylene vinyl alcohol	EVOH
Fluorocarbon	FC
Grylamid TR55	Grylamid TR55
MODACRYLIC	MODACRYLIC
Nitrile Butadiene Rubber	NBR
Nylon	PA
Olefin	OLEFIN
Zinc salt of ethylene acrylic acid copolymer	PEAA-Zn
Polyacetylene	PAC
Polyarylamide	PARA
Polyehtylene HD	PE HD
Polyehtylene low density	PE LD
Polyethylene and Polypropylene Copolymer	PE + PP
Polyester	PES
Polyolefin	PO
Polyoxymethylene	POM
Poly(p-phenylene oxide)	PPE
Polyphtalamide	PPA
Polypropylene	PP
Polystyrene	PS
Polytetrafluoroethylene	PTFE
Polyurethane	PU
Polyvinyl Chloride	PVC
Polyvynildenefluoride	PVDF
Vynil ester	VE
Vinyon	VY
APFs	**Abbreviations**
Butyl Vynil Ether	NBVE
butyl (Z)-octadec-9-enoate	MGD
Dicapryl phtalate	DCP
diester of 3-dodecylthio propionic acid and tetraethylene glycol, hydrated amorphous silica	3-DTPA-DE/TEG
Epoxidized glycol dioleate	EGD
Glyceryl triacetyl stearate	GTS
(N-(2-ethoxy phenyl)-N-(2-ethyl phenyl)-ethanediamide)	2E2ANI
polyester adipate	PEA
polyester sebacate	PES-S
Propylene glycol dipelargonate	PGD
Rayon	Rayon
5-Methyl-1H-benzotriazole	5-MBTR
2-(2-phenylpropan-2-ylperoxy)propan-2-ylbenzene (dicumyl peroxide)	VCP

suggestion that road marking paints are a new significant plastic source to study in-depth (Gaylarde et al., 2021; Vogelsang et al., 2019).

Other relevant sources of polymers (e.g., PP, PA6, LDPE, etc.) can be farming activities in the vineyards and farmlands near the highway due to mulching film and wires for plants; polymer-modified bitumen (PMB) in asphalt pavement, often underrated but recently received some attention (Järlskog et al., 2020; Rødland et al., 2022; Volgesang et al., 2019), and the tire and wear of plastic garbage deliberately abandoned at the curbside of the highway or dropped by some vehicle. The debris then enriches the road dust, SWs, and the atmosphere, as the lighter particles can be transported for long distances (Monira et al., 2021).

Because stormwater discharges are enriched with pollutants, including SMPs, it is crucial that there are treatment facilities for these waters and that these facilities are appropriately managed to monitor and reduce the loading of SMPs from highways to the environment. Currently, understanding remains poor, measurements are limited, and more investigations are needed (Koutnkin et al., 2022; Windsor et al., 2019b).

3.3. Quantification and identification of APFs in highway stormwater runoff

Simultaneously with the quantification and identification of SMPs, APFs were identified and quantified in all SW samples (Fig. S5). The complete list of identified APFs and their acronyms is reported in Table S2 in the supplementary information. The distribution of the most abundant APFs is shown in Fig. 3. APFs' abundance ranged from the minimum value in SW1 and the maximum value in SW2. APFs' weight ranged from 1735,0 ± 57 to 13317,6 ± 159 μg APFs/L (SW1 and SW4, respectively; Fig. S6 in supplementary information); SW4 showed the highest weight of both SMPs and APFs.

As already observed for SMPs, elliptical particles (E) were prevalent (Fig. S7 in the supplementary information). While APFs and SMPs were equally abundant in SW1 and SW3, APFs were dominant in SW2 and SW4. As already observed for SMPs, APF's sizes were >100 μm, being an average length of 88,0 μm and an average width of 46,4 μm (Table S3, supplementary information); these components of microlitter in SW runoff may be ingested by organisms and enter the trophic web, potentially exerting a toxicological effect on biota (Beiras et al., 2020). We ascertained that the variance of APFs was not homogeneous (F test, $\alpha = 0.05$), then non-parametrical statistical tests were applied. The abundance of APFs observed in the samples under study was markedly different (Mann- Whitney U test $p < 0.05$), and these differences were statistically significant (Kruskall-Wallis test, $p < 0.05$).

Comparing the trend of SMPs' abundance and the rainfall's intensity, APFs' abundance differed, with the maximum concentration of APFs observed in SW2, which registered the minimum abundance of SMPs. Additives and plasticizers can be leached from MPs, or they may still be attached to plastic particles during the fragmentation into smaller pieces; since they can be polymer specific, their presence can serve as a proxy for the presence of MPs and SMPs.

Additives and plasticizers can be present in the atmosphere and in the road dust, washed away by the rain, and can end up in the HSW runoff. Hence, SWs can be a significant source of MPs and APFs, representing a relevant input of these chemical compounds in receiving water bodies with great concern as they exhibit a high propensity to enter all trophic levels (Ding et al., 2022; Sridharan et al., 2022). Most additives and plasticizers have not yet been analyzed and identified in environmental matrices; in a few recent studies, only a small number of specific additives with other sampling procedures, pretreatment, and analytical techniques in road dust (Kitahara and Nakata, 2020) or an urban tributary in Australia for tire particles (Rauert et al., 2022) were analyzed. Hence, comparing the abundance and distribution of APFs observed in this research with other studies is, in fact, critical.

Several different additives and plasticizers were identified in SW samples. DMA, a PVC plasticizer, was present in all the samples, and its highest concentration was observed in SW2 (43351 APFs/L). Other PVC plasticizers were identified (e.g., NNE identified in the water and sediments of the Venice Lagoon (Corami et al., 2021)). Since PVC, identified only in SW4, can also be employed for road signs, the presence of PVC-specific plasticizers, additives, and stabilizers corroborated the occurrence of PVC in SW runoff.

Some APFs can be related to tire wear since they are employed in tire manufacturing: antioxidant additives for rubber compounds (e.g., 3-DTPA-DE/TEG), vulcanizing agents, pre-vulcanization retarders like 5-MBTR, antiozonants to prevent rubber degradation (SPPDA), accelerators for temperature cures, and plasticizers, which can be employed for rubbers and elastomers. Other plasticizers and additives with broader applications can be traced back to the automotive industry and tires.

Nonplastic synthetic and natural fibers, i.e., rayon and cellulose, were quantified and identified. They may come from atmospheric depositions or garbage debris on the curb of the highway, e.g., cigarette butts abandoned at curbsides. Besides, rayon may be a proxy of tire wear particles since it is used as an additive in tires to maintain structural integrity and resist mechanical wear (Grammelis et al., 2021).

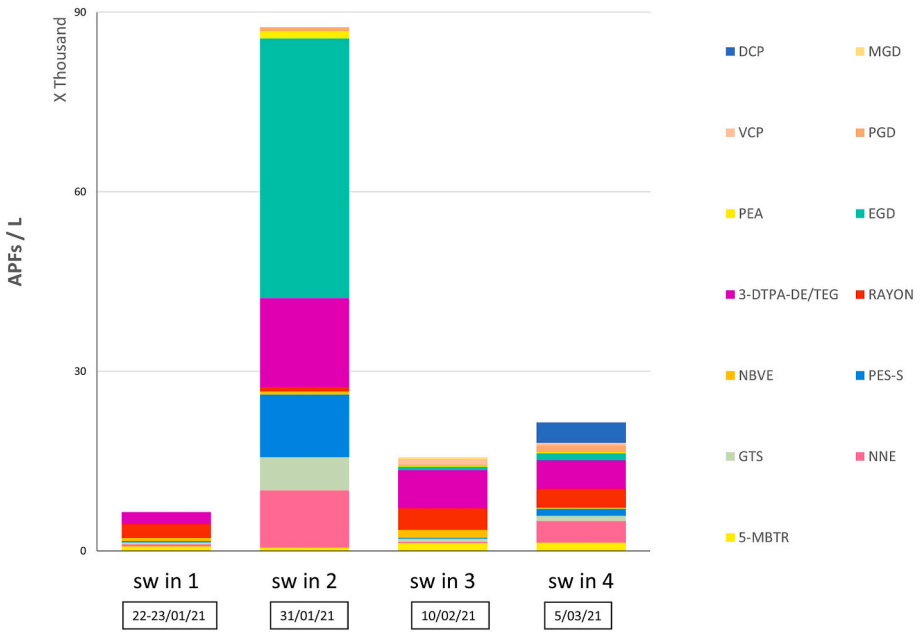


Fig. 3. Abundance (APFs/L) and typology distribution of APFs in highway stormwater runoff. Abundances and errors (fiducial interval according to Poisson Distribution) are reported.

4. Conclusions

The optimized pretreatment procedure (oleo-extraction and purification) employed in this study allowed the concurrent quantitative analysis and characterization of SMPs and APFs in HSW samples with accuracy and replicability. The pretreatment method avoids further denaturation/degradation of SMPs and APFs, which can be identified with an optimal match percentage. SMPs and APFs were present in all the HSW samples studied, albeit with substantial differences in concentrations, weight, size, and polymeric composition. The majority of SMPs and APFs can be traced back to vehicular traffic and automotive wear and tear. Most notably, APFs can be excellent proxies for checking the presence of tire wear particles, which are often not unambiguously identified, given the complexity of tires. These preliminary data have highlighted that HSW runoff can be a significant source of all the microlitter components. Besides, assessing aerosol and precipitation, along with other meteorological variables and vehicular traffic, is crucial in drawing a more accurate picture of the pathways and transport of SMPs and APFs in the SW runoff. Therefore, characterization and quantification of SMPs and APFS will undoubtedly be relevant to regulatory aspects, evaluation of potential solutions with appropriate mitigation technologies, toxicity studies, and risk assessment.

Credit author statement

Beatrice Rosso: Methodology, Validation, Investigation, Data curation, Visualization, Writing – original draft; Writing - review & editing. Fabiana Corami: Conceptualization; Methodology; Validation; Formal Analysis, Visualization, Writing - original draft, Writing - review & editing; Supervision. Luca Vezzaro: Resources; Writing - review & editing. Stefano Biondi: Resources; Barbara Bravo, Resources; Carlo Barbante: Supervision, Writing - review & editing; Andrea Gambaro: Supervision, Resources, Writing - review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

data published will be available on request

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Appendix A. Supplementary data

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