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## **Tire and road wear particles in infiltration pond sediments: Occurrence, spatial distribution, size fractionation and correlation with metals**

Tiago De Oliveira, Du Phuc Tho Dang, Maxime Chaillou, Sampriti Roy, Nadège Caubrière, Martin Guillon, Martin Guillon, David Mabilais, Sophie Ricordel, Liliane Jean-Soro, et al.

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1 **Title**

2 Tire and road wear particles in infiltration pond sediments: Occurrence, spatial distribution,  
3 size fractionation and correlation with metals

4

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16 **Highlights**

17 The TRWP footprint in infiltration pond sediments is assessed by Pyr-GC-MS.

18 Infiltration pond sediments are contaminated in TRWP up to 65 mg/g.

19 The TRWP-metals correlation suggests TRWP's role as a metal indicator.

20 Infiltration ponds prove to be efficient in retaining TRWP.

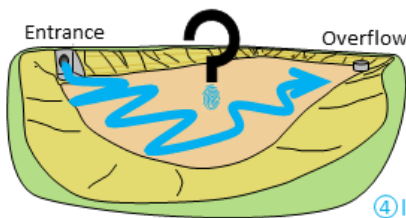
21

22 **Graphical abstract**

**Are stormwater systems efficient in retaining TRWP? Case of an infiltration pond**

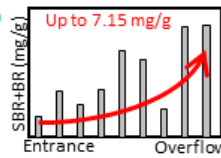
**TRWP contamination of the sediment**

- ① Spatial and ② particle size footprint?
- ③ Relation to metal contaminants?
- ④ TRWP mass balance inflow vs. retained?

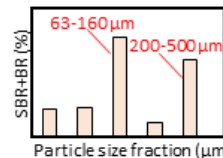


**TRWP analysis**

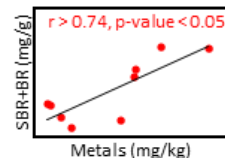
SBR + BR polymeric fraction of tire by Pyr-GC-MS



① Enrichment towards overflow



② Bimodal size distribution



③ Correlation TRWP-Metals

④ Inflow ≈ retained → The infiltration pond prevents the discharge of TRWP

23

24 **Abstract**

25 Stormwater systems, such as infiltration ponds or basins, play a critical role in managing runoff  
26 water and reducing particulate pollution loads in downstream environments through  
27 decantation. Road runoff carries several pollutants, including trace metals and tire and road  
28 wear particles (TRWP). To improve our understanding of infiltration ponds as regards TRWP  
29 and their capacity to reduce TRWP loads, we have studied the occurrence, spatial distribution  
30 and size distribution of TRWP, as well as their relationship with metals, in considering the input  
31 of metals as tire additives, in the sediments of an infiltration pond located along the Nantes  
32 urban ring road (Western France), which happens to be a high-traffic roadway site. The  
33 sediment was analyzed using pyrolysis coupled with gas chromatography-mass spectrometry  
34 to determine the polymeric content of tires, specifically in quantifying the styrene-butadiene  
35 rubber (SBR) and butadiene rubber (BR) pyrolytic markers. By applying an SBR + BR-to-  
36 TRWP conversion factor, the results showed significant TRWP contamination, up to 65 mg/g,  
37 with a spatial enrichment from the entrance to the overflow section of the pond. Size  
38 fractionation revealed a bimodal distribution, indicating two distinct types of TRWP. The first  
39 type is characterized by small diameters (63-160  $\mu\text{m}$ ), suggesting the presence of TRWP less  
40 integrated with mineral and organic particles. The second type, characterized by larger  
41 diameters (200-500  $\mu\text{m}$ ), suggests a more pronounced integration with these same mineral and  
42 organic particles. A significant positive correlation between TRWP and metals (As, Cd, Cr, Cu,  
43 Li, Mo, Ni, Sb, V, Zn) was found ( $r > 0.739$ ,  $p < 0.05$ ). This correlation implies that TRWP  
44 and/or their associated phases may act as an indicator of metal contamination in the pond  
45 sediments. Lastly, a mass balance between TRWP inputs and the amount retained in the  
46 sediments underscores the role of infiltration ponds as "sinks" for TRWP.

47 **Keywords:** Stormwater management, sediment pollution, tire and road wear particles, trace  
48 metals, pyrolysis-GC-MS.

## 49 **1. Introduction**

50 At present, the literature quite frequently states that efficient stormwater management has  
51 become a major challenge. For the past several decades, Sustainable Drainage Systems (SuDS)  
52 have been implemented in sensitive areas, particularly urban ones (Wei *et al.* 2023). Within this  
53 category of stormwater management systems, among the most widespread types of  
54 infrastructure are retention and infiltration ponds or infiltration basins. Although "infiltration  
55 pond" and "infiltration basin" refer to the same infrastructure, the term "infiltration pond" will  
56 be preferred in this manuscript. These infrastructures play a critical role in not only regulating  
57 stormwater flows, in some cases allowing aquifers to be recharged, but also reducing pollutant  
58 loads into downstream environments, mostly through decantation, photooxidation or  
59 biodegradation (Dang *et al.* 2023).

60 The runoff collected from road surfaces contains several categories of pollutants, including  
61 metals, organic micropollutants and microplastics, most of which head towards SuDS  
62 infrastructure. Numerous studies have provided valuable insights into the presence of metals in  
63 sediment from SuDS, thereby highlighting their significant accumulation and the need for more  
64 tailored management strategies aimed at the removal of contaminated sediments (Allen *et al.*  
65 2017; Clozel *et al.* 2006; El-Mufleh *et al.* 2014; Heal *et al.* 2006; Humphrey *et al.* 2023; Tedoldi  
66 *et al.* 2017).

67 More recently, studies have been increasingly focused on both macroplastics and microplastics  
68 in SuDS, in light of the evidence of their ubiquity and high load in urban runoff (García-Haba  
69 *et al.* 2023; Ledieu *et al.* 2022; Werbowski *et al.* 2021). Polyethylene, polypropylene, polyester,  
70 acrylic, polyamide and polystyrene have emerged as the most abundant polymers, typically  
71 assessed via Fourier-transform infrared spectroscopy, with concentrations ranging from a few  
72 items per kg of infiltration pond sediment to  $10^6$  items/kg (Koutnik *et al.* 2022). Reviews

73 conducted by García-Haba *et al.* (2023) and Österlund *et al.* (2023) on this topic suggest that  
74 SuDS effectively mitigate, by means of decantation, the release of microplastics to downstream  
75 environments, thus acting as sinks for these materials.

76 Studies performed on infiltration ponds with respect to metal contaminants were found to be  
77 well documented compared to the studies on macroplastic and microplastic particles. Within  
78 the microplastic category and considering that elastomers are included in the definition of  
79 microplastics, tire and road wear particles (TRWP) have been, until recently, even less  
80 extensively studied. TRWP are formed by the aggregation of tire particles (mainly constituted  
81 by a polymeric fraction of styrene-butadiene rubber (SBR) and butadiene rubber (BR)) and  
82 abraded road materials, which are recognized as a major source and pathway for microplastics  
83 into the environment (Kole *et al.* 2017). These "heteroaggregates" are being continuously  
84 generated by road traffic, with an estimated annual mass emission per capita ranging from 0.2  
85 to 5.5 kg (Baensch-Baltruschat *et al.* 2020). More specifically, the majority of the emitted  
86 TRWP (i.e. > 99%) occurs at the road surface, with a dispersion primarily ascribed to runoff  
87 into the environment (De Oliveira *et al.* 2024).

88 Consequently, road dust is highly concentrated in TRWP, hence leading to prolonged  
89 impregnation of soils and sediments in the proximity of highways and roads within urban areas.  
90 Such areas are characterized by TRWP concentrations ranging from a few to hundreds of grams  
91 of TRWP per kilogram of soil or sediment matrices (Eisentraut *et al.* 2018; Federico *et al.* 2023;  
92 Goßmann *et al.* 2021; Mengistu *et al.* 2021; Rødland *et al.* 2022; Unice *et al.* 2012). Given the  
93 occurrence of TRWP in the urban environment, their presence raises considerable concerns for  
94 several reasons, including: i) the simultaneous diffusion of a hazardous mix into diverse  
95 environmental compartments (Järnskog *et al.* 2022; Zhu *et al.* 2024); ii) the ecotoxicological  
96 effects of the many additives and other compounds constituting TRWP, encompassing both  
97 organic and inorganic additives, which have been identified as highly toxic to organisms

98 (Capolupo *et al.* 2020; Dufey *et al.* 2024; Jiang *et al.* 2023; Li *et al.* 2024; Müller *et al.* 2022;  
99 Page *et al.* 2022; Peng *et al.* 2022; Roubeau Dumont *et al.* 2023; Tian *et al.* 2021; Yang *et al.*  
100 2022); and iii) the lack of a quantitative understanding of TRWP aging processes, with the  
101 release of chemicals through leachate over time (Wagner *et al.* 2022).

102 Up until now, only a few studies have assessed the occurrence of TRWP within the sediment  
103 of the most strategic and widespread urban infrastructure; moreover, they have revealed an  
104 accumulation ranging up to hundreds of milligrams of TRWP per gram of dried sediment  
105 (Eisentraut *et al.* 2018; Gaggini *et al.* 2024; Klöckner *et al.* 2019; Ziajahromi *et al.* 2023).  
106 However, none of these studies has ever evaluated the effectiveness of stormwater systems in  
107 reducing TRWP contamination or actually proposed some keys to managing the contaminated  
108 sediments. If sediment management and its removal were to be neglected, then the intended  
109 purpose of SuDS would become obsolete, in potentially transforming the SuDS from sinks into  
110 sources of pollution (Humphrey *et al.* 2023). Hence, understanding the quality of sediments  
111 becomes imperative to optimizing the operational efficiency of SuDS (Färm, 2002).

112 To gain new insight into the fate of TRWP within infiltration ponds and infiltration ponds'  
113 ability to reduce the TRWP discharge into aquatic ecosystems, we have explored herein for the  
114 very first time: i) the contamination footprint of TRWP (including occurrence, spatial  
115 distribution and particle size fractionation) within the sediment of an infiltration pond located  
116 near the Nantes urban ring road (Western France); ii) the relationship between TRWP and  
117 metals, since metals are used as tire additives and may also originate from other vehicular and  
118 road wear sources, thus suggesting that TRWP may act as a pathway for metal pollution; and  
119 iii) the role of an infiltration pond in mitigating TRWP contamination by assessing the mass  
120 balance between TRWP inputs and the amount retained in the sediments. Our analysis has  
121 focused on the synthetic rubber content of tires (specifically SBR and BR) by using pyrolysis  
122 coupled with gas chromatography-mass spectrometry (Pyr-GC-MS). On the whole, we are

123 expecting that the data acquired will contribute to improving those management practices aimed  
124 at reducing pollutant discharge into the downstream environment.

## 125 **2. Materials and Methods**

### 126 **2.1 Sampling site**

127 The studied site is an infiltration pond (Fig. 1), located in the city of Nantes (Western France,  
128  $47^{\circ}11'05''\text{N}$ ,  $1^{\circ}36'51''\text{W}$ ), designed to receive runoff from the southern section of the Cheviré  
129 Bridge (location of the infiltration pond is available in the Appendix, Figures SI-1). This section  
130 encompasses 0.8 km of the total 1.6 km length of the bridge, covering a drainage area of 2.2 ha  
131 of impervious asphalt. The Cheviré Bridge is a component of the N844 ring road crossing the  
132 Loire River characterized by: three lanes in both directions, a 70 km/h speed limitation, and the  
133 capacity to carry heavy traffic with an annual traffic count reaching approximately 100,000  
134 vehicles per day in 2019, of which 10% were heavy vehicles (Dang *et al.* 2023). The average  
135 daily traffic during the study period, from July 2020 to March 2021, was measured at 88,500  
136 vehicles. The pond's unique drainage surface and design make it exceptionally well suited for  
137 a case study focusing on road runoff assessment.

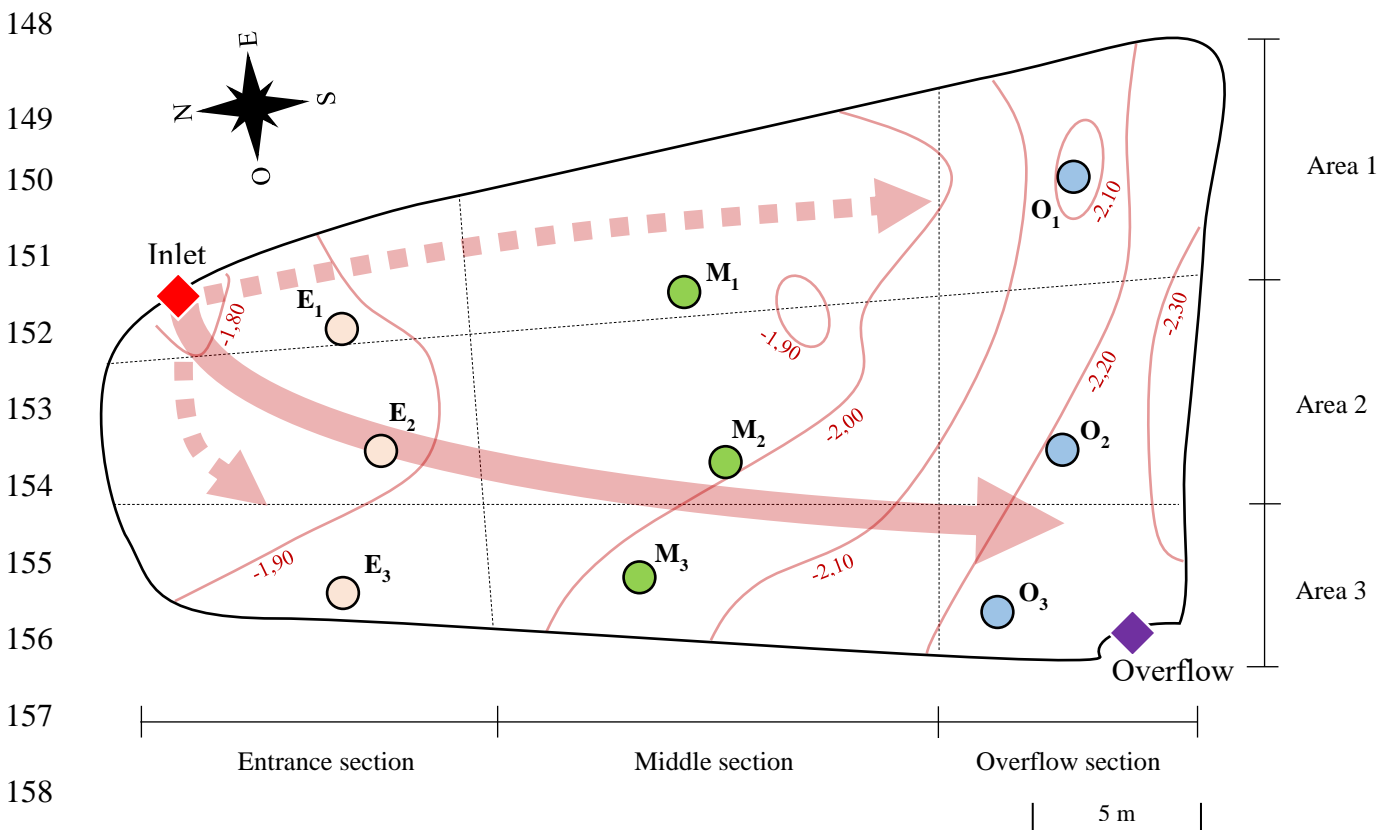


138

139 Figure 1: Photograph of the studied infiltration pond. The inlet is located on the left side of  
140 the pond. A close-up of the overflow is shown in the bottom right corner.



141 The runoff drainage system includes zinc gutters, polyvinyl chloride pipes and concrete pipes,  
 142 all of which facilitate the transport of runoff from the bridge to the infiltration pond. The pond  
 143 was commissioned in April 1991, at the same time as the bridge. Over the years, the  
 144 accumulation of sediment and the proliferation of dense vegetation prompted the first complete  
 145 cleaning of the pond on July 21, 2020. The sediment deposit covers an area of 610 m<sup>2</sup>. The  
 146 substrate beneath the pond sediments is mainly composed of silty sand, over the first 4 meters,  
 147 and clay-silt sand alluvium from 4 to 30 meters.



159 Figure 2: Location of the sampling points and topography of the infiltration pond. The  
 160 topography is represented by the light red lines. Values are expressed in meters, with the  
 161 reference level located at the soil surface. The surface of the pond was divided into three  
 162 sections (entrance, middle, overflow) and three areas (1, 2, 3). The inlet is positioned in the  
 163 top left part of the pond, while the overflow is located at the bottom right. The solid-line  
 164 arrow represents the primary runoff path from the inlet during light to moderate rain events,  
 165 and the dotted arrows indicate the secondary path during heavy rain events. The primary and  
 166 secondary flow paths were identified through in situ observations. The first sections impacted  
 167 by rain events are the overflow and middle sections, in areas 2 and 3, followed by the  
 168 entrance section and area 1.

169 The absence of an impermeable layer at the pond's bottom makes it permeable to runoff, thus  
170 enabling relatively rapid infiltration. The pond typically empties within 2 to 4 days into the  
171 substratum (El-Mufleh *et al.* 2014). However, in cases where the runoff volume surpasses the  
172 pond's capacity, a downstream overflow facilitates the direct discharge of excess water into the  
173 Loire River. The flow of runoff into the infiltration pond, and consequently its filling, is  
174 influenced by both the topography and the intensity and duration of rainfall events (Fig. 2).

## 175 **2.2 Sediment sampling**

176 The sampling step was conducted on March 25, 2021 when the pond was dry, following an 8-  
177 month period of sediment accumulation within the infiltration pond. The pond's surface was  
178 delineated into three sections: entrance, middle, and overflow, with further division into three  
179 specific areas (Fig. 2). The sediment layer thickness was measured at approximately 1 cm  
180 within the entrance section and 2 cm within the overflow section. Pond depth was found to be  
181 -1.80 m at the entrance and -2.30 m at the overflow. The sediment samples were collected at  
182 random from a surface area measuring 33 x 35 cm at a depth corresponding to the sediment  
183 thickness (i.e. 1 to 2 cm) for each defined sampling point, in utilizing a stainless-steel hand  
184 shovel. Subsequently, the sediments were air-dried and stored in polyethylene bottles.

## 185 **2.3 Metals and their physicochemical analysis**

186 The samples designated for metals, grain size distribution and organic matter content analysis  
187 underwent a pretreatment protocol in accordance with the ISO 11464 (2006) Standard. The  
188 dried samples were sieved using a 2-mm nylon sieve. For the metals analysis, the samples were  
189 subjected to grinding and dissolution processes, as specified in the ISO 14869-1 (2001)  
190 Standard. 0.5 g of the ground sediment were calcinated at 450°C. Subsequently, 10 mL of HF  
191 (47-51%) and 3.5 mL of HClO<sub>4</sub> (65%) were added. The resulting suspension was heated at  
192 160°C until complete evaporation. After cooling, the dry residue was exposed to 1 mL of HNO<sub>3</sub>

193 for 30 minutes, followed by the addition of 20 mL of ultra-pure water. The mixture was then  
194 heated again at 80°C for 1 hour. After a second cooling, the mineralized solution was transferred  
195 to a 100-mL glass volumetric flask through a Whatman No. 40 filter. The solutions were stored  
196 in polyethylene flasks in a refrigerator at 4°C before analysis.

197 The analysis was conducted using inductively coupled plasma-mass spectrometry and optical  
198 emission spectroscopy, with the 8900 Triple Quadrupole from Agilent Technologies, Inc.  
199 (California, USA) and the 720-ES from Varian, Inc. (parent company: Agilent Technologies,  
200 Inc.). The samples underwent triplicate analysis, in targeting: arsenic (As), cadmium (Cd),  
201 chromium (Cr), copper (Cu), lithium (Li), molybdenum (Mo), nickel (Ni), antimony (Sb),  
202 vanadium (V), and zinc (Zn) elements in accordance with the ISO 11885 (2007) Standard  
203 (further details regarding the limits of quantification are provided in Table SI-1). An enrichment  
204 factor (EF) was calculated according to both Equation 1 and the thresholds (i.e. minor EF 1-3,  
205 moderate EF 3-5, moderate-severe EF 5-10, severe EF 10-25, very severe EF 25-50 and  
206 extremely severe EF > 50) proposed in the Rødland *et al.* study (2023).

Equation 1: 
$$EF = \frac{M_s}{M_b}$$

207 where  $M_s$  denotes the concentration of the targeted metal element M analyzed in the sample  
208 from the infiltration pond sediment, in mg/kg, and  $M_b$  the concentration of element M  
209 corresponding to the value of the geochemical background obtained from a sample extracted  
210 from the edge of the infiltration pond, in mg/kg.

211 A sediment granulometry assessment was performed using a Mastersizer 3000 from Malvern  
212 Panalytical (Palaiseau, France); these results are presented with respect to the median diameter  
213 ( $d_{50}$ ). The organic matter (OM) content was assessed as per the EN 15935 (2021) Standard,  
214 with these results being expressed in percentage terms. In addition, exploratory scanning  
215 electron microscopy (SEM) analyses were performed on the infiltration pond sediment to

216 provide more detailed information on the heteroaggregates, comprising TRWP and metal  
217 components. The analyses were performed using an SU5000 microscope from Hitachi Energy  
218 (Zurich, Switzerland) in combination with an X-Flash detector 4010 from Bruker Nano (Berlin,  
219 Germany) for energy dispersive X-ray spectroscopy (SEM-EDX).

#### 220 **2.4 TRWP analysis**

221 Prior to conducting the Pyr-GC-MS analysis, raw samples were dried at 105°C for 24 hours, in  
222 following the recommendations found in ISO 7270-1 (2017), and then sieved at 500 µm using  
223 a stainless-steel sieve to remove the large items. For the size fractionation analysis, the samples  
224 were fractioned by means of vibratory sieve separation on dry sediment into five fractions  
225 between < 36 µm and 500 µm (i.e. < 36, 36-63, 63-160, 160-200, 200-500 µm) using the  
226 Analysette 3 SPARTAN from FRITSCH (Idar-Oberstein, Germany). The samples were stored  
227 in calcinated glass vials at room temperature. The Pyr-GC-MS analyses were then conducted  
228 using a multi-shot pyrolyzer EGA/PY-3030D, equipped with an auto-shot sampler (AS-1020E)  
229 from FrontierLab (Fukushima, Japan). The pyrolyzer, set at 600°C, was coupled with the 8890  
230 gas chromatography and the 5977B mass spectrometer from Agilent Technologies, Inc.  
231 (California, USA). More detailed information is provided in Table SI-2.

232 For the quantification of SBR and BR in the sediment samples, two specific pyrolysis products,  
233 namely 4-vinylcyclohexene (4-VCH) and 4-phenylcyclohexene (4-PCH), were selected  
234 according to our method, as presented in De Oliveira *et al.* (2024). The quantification of 4-VCH  
235 and 4-PCH was based on  $m/z = 54$  and  $104$  Da, respectively. Methodological details, including  
236 preparation of the standard SBR solution in  $\text{CHCl}_3$ , preparation and use of the d-SBR solution  
237 as an internal deuterated standard, calibration curve, establishment of the SBR/d-SBR ratio for  
238 quantification along with information on the repeatability of standard solutions, can be found  
239 in the referenced study (De Oliveira *et al.* 2024). The analysis of multiple specific blanks (i.e.

240 no cup, cup only, cup + CHCl<sub>3</sub>, cup + SiO<sub>2</sub>) showed no SBR or BR contamination. The  
241 correlation coefficient of the calibration line equaled 0.998, while the limit of quantification  
242 was determined to be 5 µg SBR/g. Approximately 3 mg of sediment were weighed in pyrolysis  
243 cups using the Mettler Toledo AB265-S/FACT balance and a stainless-steel spatula.

244 Also, 1.27 µg of the internal standard d-SBR in solution were introduced into each cup along  
245 with the sediment sample. All samples underwent triplicate analysis, with a signal-to-noise ratio  
246 of both targeted markers exceeding 10 and a variability of less than 9%. The variability of the  
247 internal standards markers was calculated at 7%. Three control samples (i.e. 1.10 µg SBR +  
248 2.50 mg SiO<sub>2</sub>) were analyzed within the sediment sample sequence. The recovery calculation  
249 yielded 94 ± 8%. Results in this study are expressed in terms of mass of SBR + BR or TRWP  
250 per mass of dry sediment. The latter parameter is obtained using a median SBR + BR-to-TRWP  
251 conversion factor of 9.12, which encompasses both the minimum and maximum of mineral  
252 encrustment and SBR content in TRWP (Kreider *et al.* 2010; Rauert *et al.* 2021; Sommer *et al.*  
253 2018; Unice *et al.* 2012).

254 Furthermore, the pond's theoretical effectiveness in retaining TRWP was evaluated by  
255 estimating both the inflow of TRWP into the pond and the total amount retained in the sediment  
256 using Equations 2 and 3, respectively.

Equation 2: 
$$TRWP_{inflow} = TWL \times W \times T \times t \times d$$

257 where  $TRWP_{inflow}$  represents the inflow of TRWP (mg) into the pond for the considered  
258 period.  $TWL$  represents the driving wheel rate of the tire weight loss specific to the ring road  
259 that is connected to the infiltration pond (mg/km).  $W$  denotes the driving wheel contribution  
260 factor to the emission that accounts for all tires on a vehicle. Also,  $T$  is the traffic density  
261 (vehicle/day),  $t$  the time frame considered for the study (days), and  $d$  the drained road distance  
262 of the southern section of Chevire's bridge connected to the infiltration pond (km).

Equation 3: 
$$TRWP_{retained} = (SBR + BR) \times C_f \times S \times Th \times \rho$$

263 where  $TRWP_{retained}$  represents the amount of TRWP (mg) retained in the sediment of the  
264 infiltration pond.  $SBR + BR$  represents the concentration of the synthetic SBR and BR  
265 polymers quantified within the sediment at each sampling point (mg/g). Moreover,  $C_f$  is the  
266 mean SBR + BR-to-TRWP conversion factor of 9.12, as obtained from the literature (Klößner  
267 *et al.* 2021; Kreider *et al.* 2010; Rauert *et al.* 2021; Sommer *et al.* 2018; Unice *et al.* 2012);  $S$   
268 the surface area of the considered section (m<sup>2</sup>);  $Th$  the thickness of the sediment layer of the  
269 considered section (m); and  $\rho$  the sediment density (g/cm<sup>3</sup>) (Table SI 3 summarizes all these  
270 data).

## 271 **2.5 Statistical analysis**

272 A statistical analysis of the data was performed using Microsoft Office Excel with the XLSTAT  
273 add-on and PAST, version 4.03. The assumption of a normal data distribution was assessed by  
274 means of the Shapiro-Wilk test. Additionally, Quantile-Quantile plots were employed to  
275 provide a qualitative visual assessment of the data. Both Pearson correlation coefficients and  
276 Spearman's rank correlation coefficients were calculated in order to assess the relationships  
277 between SBR + BR, OM and the metals variables, at a confidence level of 95%. The statistical  
278 significance p-value was set at 0.05.

## 279 **3. Results and Discussion**

### 280 **3.1 Occurrence and spatial distribution of SBR + BR**

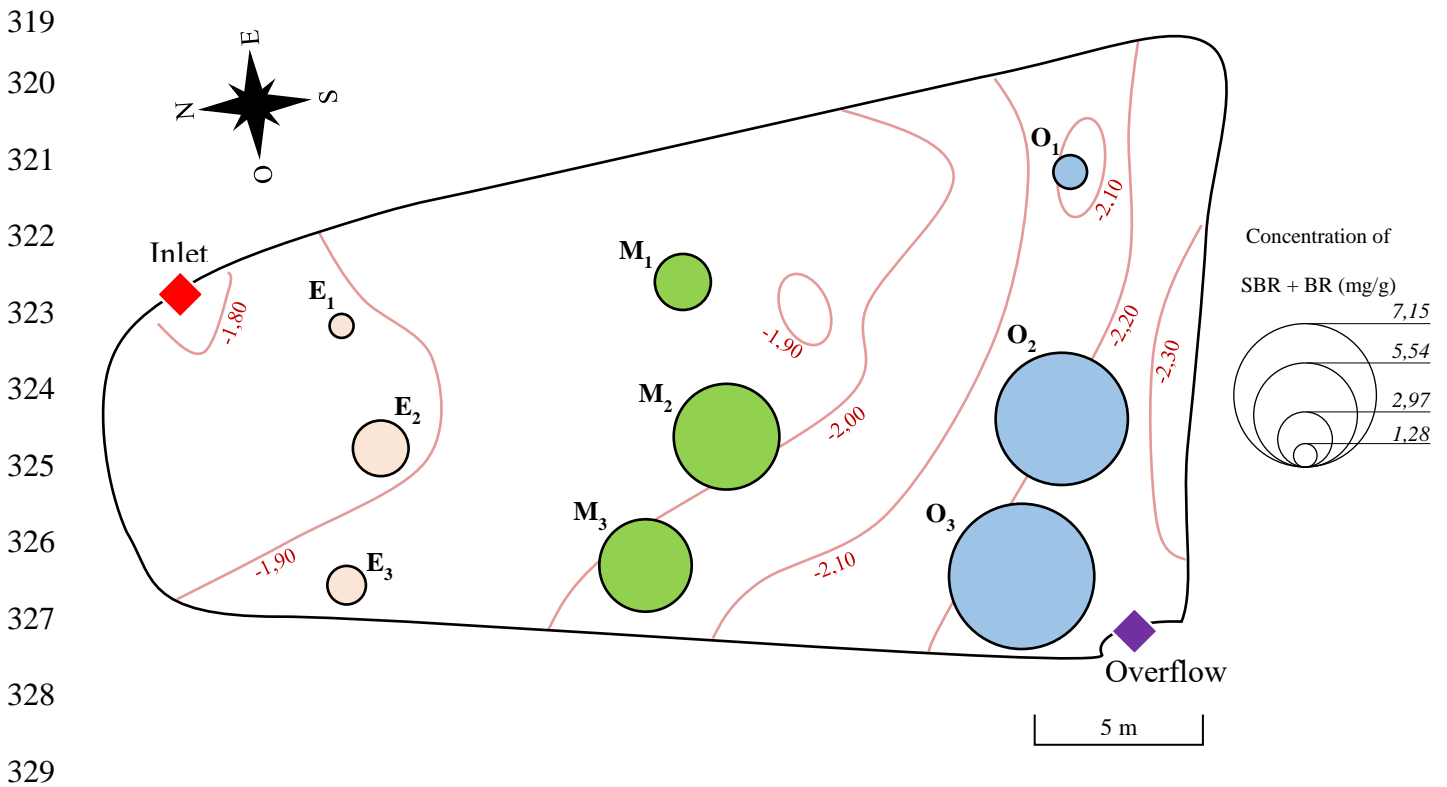
281 The amount of observed SBR + BR ranges from  $1.28 \pm 0.05$  mg/g at the entrance section  
282 (sample E<sub>1</sub>) to  $7.15 \pm 0.29$  mg/g at the overflow section (sample O<sub>3</sub>) (Fig. 3). The median  
283 concentration calculated for the pond equals  $2.99 \pm 0.12$  mg/g. These values are of the same  
284 order of magnitude as those found by Eisentraut *et al.* (2018), i.e. 9.2 mg/g of SBR from

285 sediment samples of a treatment system for highway street runoff. When converted to TRWP  
286 concentration, our data indicate that the sediment contains up to  $65.21 \pm 2.64$  mg of TRWP per  
287 gram. This measurement aligns, to a certain extent, with the findings of Klöckner *et al.* (2019),  
288 who reported  $130 \pm 15$  mg of TRWP per gram found in sediment from a highway runoff  
289 treatment facility. The discrepancy between our results and those of Klöckner *et al.* can be  
290 attributed to a higher dilution of TRWP levels within the sediment of the Cheviré pond as well  
291 as the traffic volume: the highway in their study experienced 1.9 times greater vehicle flow than  
292 the ring road investigated herein. Let's note that this increased traffic correlates with a TRWP  
293 concentration twice as high.

294 Results revealed differential concentrations across the sections, in reflecting the specific spatial  
295 distribution of SBR + BR within the pond. Median SBR + BR concentrations per section were  
296 calculated to be:  $2.06 \pm 0.07$ ,  $4.99 \pm 0.21$  and  $7.05 \pm 0.29$  mg/g for the entrance, middle and  
297 overflow sections respectively, thus representing a 3.4-fold increase from entrance to overflow.  
298 The variations in concentration are not only observed between different sections but also within  
299 the same section. More specifically, the enrichment levels of SBR + BR in areas 2 and 3 were  
300 on average 2.45 times higher than those in area 1 across all pond sections, which would suggest  
301 that samples from areas 2 and 3 were likely positioned near or at the main inflow of runoff from  
302 the inlet and thus closer to or at the main sediment accumulation zone. As a result, area 1 across  
303 all sections, by virtue of lying further from the source of road dust runoff, exhibits lower  
304 concentrations of SBR + BR.

305 To further refine the spatial distribution of SBR + BR and better understand their variability,  
306 the pond's topography was examined as a potential explanatory factor. Topography is key to  
307 shaping the sedimentary dynamics of a pond. The topographic analysis revealed a gradual  
308 increase in depth from the entrance to the overflow section, with recorded depths of -1.80 m  
309 and -2.30 m, respectively.

310 This deepening suggests a greater accumulation of materials within the overflow section. The  
 311 sediment thickness aligns with the depth gradient, starting at approximately 1 cm in the entrance  
 312 section, increasing to approx. 2 cm in the overflow section, where water covers the sediment  
 313 for several days after rain events. The pond's topography and morphology influences both the  
 314 orientation and directional flow of runoff within the pond and moreover promotes the  
 315 accumulation and settling of TRWP in areas 2 and 3, specifically in the middle and overflow  
 316 sections, along the depth gradient. This assertion is corroborated by the findings for SBR + BR.  
 317 Also, the settling velocity of particles, which depends on their granulometry, plays a role in the  
 318 specific spatial distribution of SBR + BR and will be further discussed below.



330 Figure 3: Spatial distribution of the SBR + BR (median of replicates, in mg/g), as quantified  
 331 in sediment samples (< 500  $\mu$ m) from the entrance (E<sub>1</sub>, E<sub>2</sub>, E<sub>3</sub> in orange), middle (M<sub>1</sub>, M<sub>2</sub>, M<sub>3</sub>  
 332 in green) and overflow (O<sub>1</sub>, O<sub>2</sub>, O<sub>3</sub> in blue) sections, along with the topography (light red  
 333 lines, values expressed in meters). The size of the circles is proportional to the amount of SBR  
 334 + BR quantified in the sediments (a histogram representation of these results with error bars is  
 335 provided in the Appendix, Figure SI-2).



### 336 **3.2 Size fractionation of SBR + BR**

337 The granulometry of the sediment and the size fractionation of SBR + BR content within the  
338 sediment are two parameters critical to understanding both the fate and spatial distribution of  
339 TRWP. A finer sediment was observed towards the overflow sections. The results of  $d_{50}$   
340 averaged  $104 \pm 8$ ,  $83 \pm 16$  and  $70 \pm 18$   $\mu\text{m}$  at the entrance, middle and overflow sections,  
341 respectively (Table SI-4). The granulometric dynamic can be attributed to two factors: i) the  
342 inherent faster settling velocity of coarser particles at the inlet compared to finer particles; and  
343 ii) the turbulence generated at the inlet by the inflow of runoff water, a phenomenon that varies  
344 with rainfall intensity. The high turbulence conditions promote the remobilization of finer  
345 materials, which are subsequently transported and deposited further in the middle and overflow  
346 sections along the topography, while insufficiently affecting the transport of coarser materials,  
347 which primarily settle in the entrance section (Clozel *et al.* 2006). This effect ultimately leads  
348 to an enrichment of sands and gravels in the vicinity of the inlet, whereas finer particles become  
349 more prevalent towards the overflow section (Färm, 2002; German and Svensson, 2005).

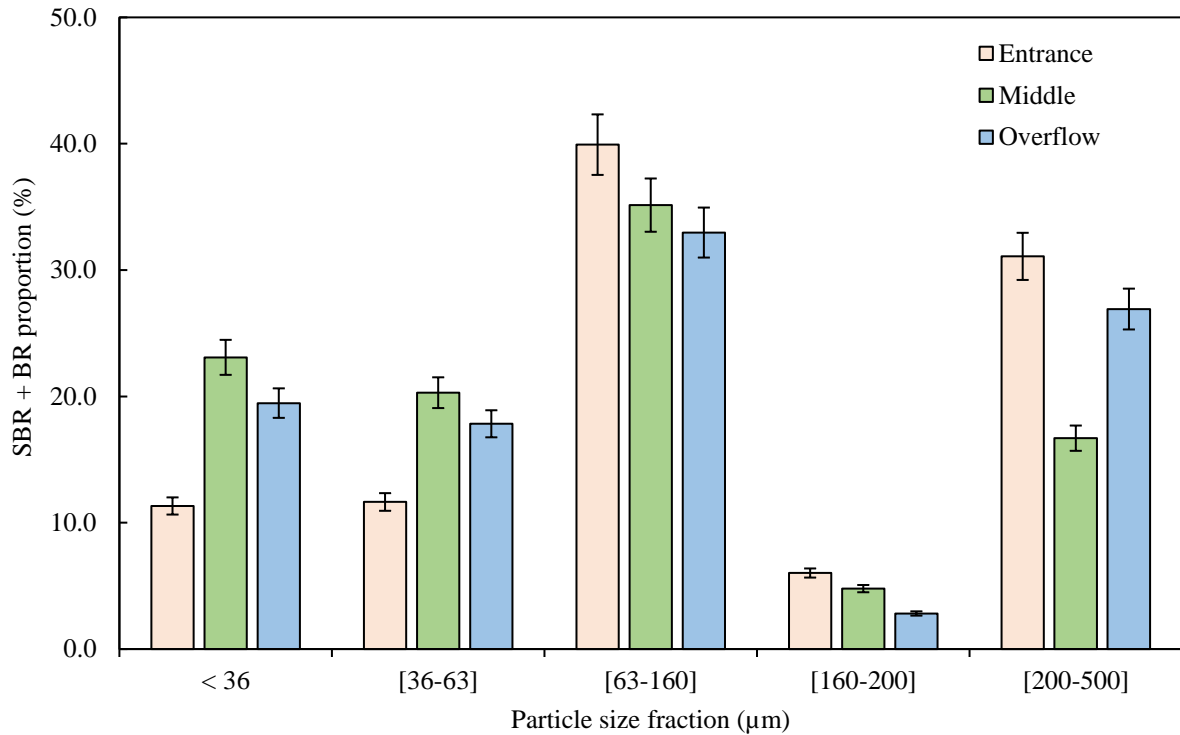
350 As regards the SBR + BR content, the grain size distribution would suggest that TRWP are  
351 characterized by a finer granulometry in the overflow section, which is corroborated by a  
352 significant negative Pearson correlation coefficient between SBR + BR and  $d_{50}$  equal to -0.910  
353 ( $p < 0.05$ ). The analysis of SBR + BR mass proportions within each sediment size fraction,  
354 spanning from  $< 36$   $\mu\text{m}$  to  $500$   $\mu\text{m}$ , extracted from the entrance, middle and overflow sections  
355 has confirmed the evolution in  $d_{50}$  (Fig. 4). The data also reveal a trend similar to that of the  
356 size fraction distribution of the sediments (Fig. SI 3). Comparing the finer sediment fraction  
357 (i.e.  $< 63$   $\mu\text{m}$ ) from the entrance section to that of the overflow section reveals an increase in  
358 the SBR + BR mass proportion.

359 More specifically, in the sediment size fraction below 36  $\mu\text{m}$ , the proportion rose from 11% to  
360 19%, and in the 36-63  $\mu\text{m}$  size fraction, the increase was from 12% to 18%, thus suggesting an  
361 enrichment of the sediment with TRWP within a relatively similar size range (i.e. < 63  $\mu\text{m}$ ).  
362 Conversely, for all sediment size fractions larger than 63  $\mu\text{m}$ , a decrease was recorded in the  
363 mass proportion of SBR + BR towards the overflow section, as a result of the granulometric  
364 dynamic discussed previously.

365 A closer examination of the size distribution reveals a similar pattern across all three sections,  
366 which can be characterized by a bimodal distribution. The first mode, and the most prevalent  
367 fraction, was centered around 63-160  $\mu\text{m}$  with a SBR + BR mass proportion of  $40 \pm 2\%$ ,  $35 \pm$   
368  $2\%$  and  $33 \pm 2\%$  for the entrance, middle and overflow sections, while the second mode was  
369 centered at 200-500  $\mu\text{m}$  with a SBR + BR mass proportion of  $31 \pm 2\%$ ,  $17 \pm 1\%$  and  $27 \pm 2\%$   
370 for the entrance, middle and overflow sections. Interestingly, the first mode aligns with the size  
371 distribution of tire wear particles generated by a laboratory-scale model that simulates non-  
372 exhaust emissions, as reported by Kreider *et al.* (2010), ranging from 5 to 250  $\mu\text{m}$  and  
373 displaying a unimodal distribution centered at 100  $\mu\text{m}$ . Note that Kovoichich *et al.* (2021)  
374 reported a similar distribution with an average TRWP size of 94  $\mu\text{m}$  in tunnel dust samples. In  
375 contrast however, they observed a coarser TRWP distribution, averaging 506  $\mu\text{m}$  in the  
376 sediment sample. The shift towards larger sizes observed in the tunnel dust samples, compared  
377 to TRWP within sediment samples, was attributed by the authors to the aging process, which  
378 includes aggregation with other environmental particles, such as minerals.

379 This process was assumed to be limited in tunnel dust but predominant in the sediment.  
380 Therefore, the bimodal distribution observed in our study may signify the presence of two  
381 distinct types of TRWP. The first and most predominant one is characterized by a smaller  
382 TRWP size and/or TRWP associated with a smaller sediment particle size, hence suggesting  
383 less aged and aggregated tire wear particles with a mineral fraction and, ultimately, with organic

384 phases within the sediment. The second type results in an association with the formation of  
385 larger heteroaggregates, featuring a higher mineral encrustation and thus falling in the coarser  
386 fraction of 200-500  $\mu\text{m}$ .



387  
388 Figure 4: Mass proportion of SBR + BR (%) for approximately 3 mg of sample extracted  
389 from the entrance (sample E<sub>1</sub>), middle (sample M<sub>2</sub>) and overflow (sample O<sub>3</sub>) sections, in five  
390 sediment particle size fractions (< 36, [36-63], [63-160], [160-200] and [200-500]  $\mu\text{m}$ ).

391

### 392 3.3 Occurrence of metals

393 The metals targeted in our study are known to be contributors to roadway runoff pollution; they  
394 stem from vehicular and road wear sources such as engines, tires, brakes, asphalt and road  
395 markings (Adachi and Tainosho 2004; Adamiec *et al.* 2016; Allen *et al.* 2017; Grigoratos and  
396 Martini 2015; Järllskog *et al.* 2021; Kwak *et al.* 2013; Thorpe and Harrison 2008; Turner and  
397 Filella 2023; Zannoni *et al.* 2016). In considering the common sources, their distribution  
398 patterns are anticipated to be analogous to those of TRWP.

399 The results of the quantification of the selected metals in the sediment matrix lie within the  $10^2$   
400 to  $10^{-1}$  mg/kg of dry sample range (Table 1). The highest concentrations across all samples were  
401 found for Zn with a median of  $779 \pm 8$  mg/kg, followed by Cu ( $178 \pm 1$  mg/kg), Cr ( $61.2 \pm 0.7$   
402 mg/kg), V ( $52.1 \pm 0.4$  mg/kg), Sb ( $30.5 \pm 0.5$  mg/kg), Li ( $25.1 \pm 0.2$  mg/kg), Ni ( $24.6 \pm 0.2$   
403 mg/kg), As ( $23.4 \pm 0.2$  mg/kg), Mo ( $3.46 \pm 0.07$  mg/kg) and Cd ( $0.53 \pm 0.01$  mg/kg). Although  
404 these values are broadly comparable to the levels of metals commonly found in similar SuDS  
405 (e.g. hundreds of mg/kg for Zn and Cu, and a few tens of mg/kg for Cr and Li) (Allen *et al.*  
406 2017; Heal *et al.* 2006; Tedoldi *et al.* 2017), significant discrepancies were nonetheless  
407 observed.

408 These variations can be attributed to specific parameters of the stormwater system that govern  
409 sediment contamination, such as the nature of the watershed surface (e.g. residential, industrial,  
410 road), the water dynamics within the watershed (e.g. precipitation and road washing by  
411 municipal services), the number of circulating vehicles, the dimensions and morphology of the  
412 infrastructure, as well as the age of the sediment as determined by the frequency of  
413 maintenance. Additionally, the technological advancements over time towards reducing the  
414 overall emission of metals have also contributed to the discrepancies observed across these  
415 studies.

416 The contamination levels of sediments in the infiltration pond were assessed by calculating the  
417 EF from a sample extracted at the edge of the pond, to serve as the geochemical background.  
418 The EFs indicate a minor enrichment for Cr, Li, Ni and V (EF from 1 to 3), a moderate to major  
419 enrichment for Cd (EF 5 to 10), a major enrichment for As, Cu, Mo and Zn (EF 10 to 25), and  
420 extreme enrichment for Sb (EF > 50) (further details are given in Table SI-5).

421

Table 1: Metal concentrations (mg/kg) found in the sediments of the infiltration pond and a geochemical background sample ( $M_b$ )

422

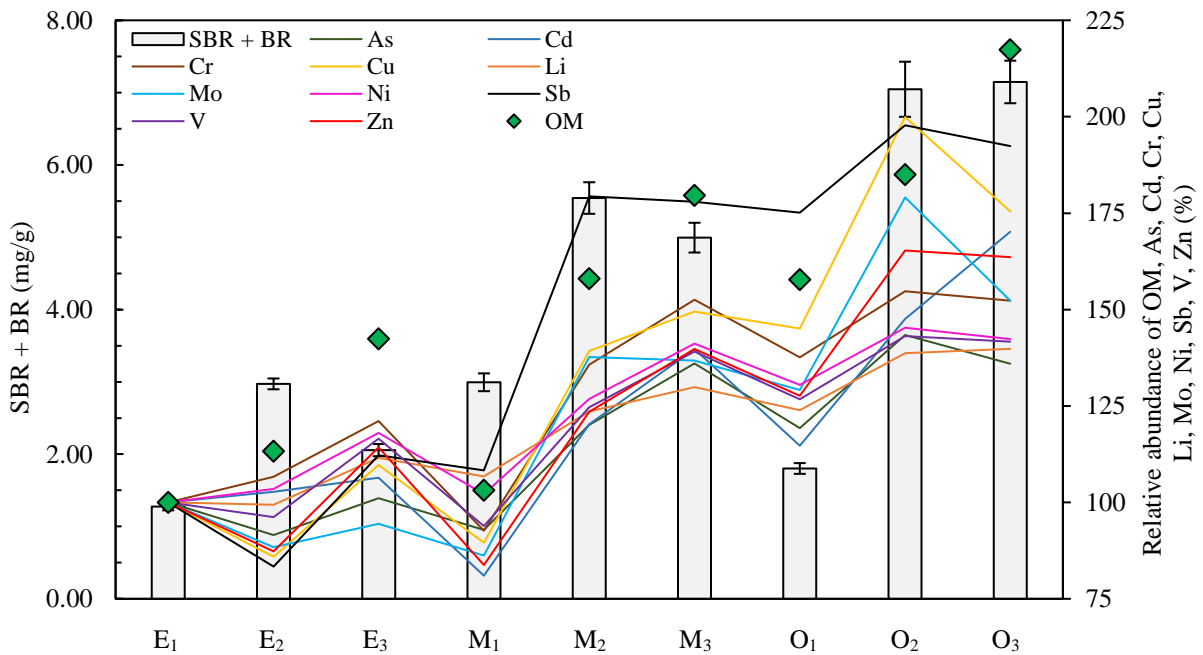
extracted from the edge of the pond

Sample	As mg/kg	Cd mg/kg	Cr mg/kg	Cu mg/kg	Li mg/kg	Mo mg/kg	Ni mg/kg	Sb mg/kg	V mg/kg	Zn mg/kg
E <sub>1</sub>	19.65 ± 0.67	0.46 ± 0.02	45.12 ± 0.10	127.88 ± 1.04	20.32 ± 0.04	2.68 ± 0.13	19.38 ± 0.28	17.41 ± 0.10	41.78 ± 0.35	630.83 ± 4.73
E <sub>2</sub>	17.99 ± 0.09	0.47 ± 0.01	48.12 ± 0.69	109.89 ± 0.17	20.19 ± 0.11	2.37 ± 0.03	20.06 ± 0.82	14.52 ± 1.30	40.17 ± 0.09	550.83 ± 2.68
E <sub>3</sub>	19.86 ± 0.74	0.49 ± 0.02	54.65 ± 0.96	140.33 ± 0.82	22.66 ± 0.11	2.53 ± 0.12	22.88 ± 0.18	19.54 ± 0.53	48.68 ± 0.51	720.98 ± 10.99
M <sub>1</sub>	18.24 ± 0.31	0.37 ± 0.01	41.80 ± 0.73	114.56 ± 3.33	21.68 ± 0.23	2.31 ± 0.06	19.81 ± 0.47	18.86 ± 0.21	39.22 ± 0.58	528.40 ± 8.03
M <sub>2</sub>	23.61 ± 0.04	0.55 ± 0.01	61.21 ± 0.48	178.12 ± 0.03	25.11 ± 0.35	3.69 ± 0.04	24.57 ± 0.08	31.24 ± 0.15	52.10 ± 0.09	779.25 ± 3.18
M <sub>3</sub>	26.73 ± 0.11	0.64 ± 0.01	68.81 ± 0.51	191.13 ± 1.86	26.39 ± 0.31	3.67 ± 0.04	27.36 ± 0.20	30.98 ± 0.88	58.11 ± 0.60	881.89 ± 10.51
O <sub>1</sub>	23.44 ± 0.23	0.53 ± 0.01	62.10 ± 1.18	185.58 ± 0.59	25.19 ± 0.11	3.46 ± 0.07	25.29 ± 0.01	30.49 ± 0.46	52.94 ± 0.13	805.34 ± 3.28
O <sub>2</sub>	28.18 ± 0.23	0.68 ± 0.01	69.81 ± 0.97	255.65 ± 4.67	28.18 ± 0.33	4.80 ± 0.31	28.16 ± 0.19	34.44 ± 0.16	59.80 ± 0.36	1042.90 ± 14.77
O <sub>3</sub>	26.73 ± 0.23	0.78 ± 0.05	68.73 ± 2.33	224.43 ± 9.19	28.41 ± 0.52	4.08 ± 0.16	27.58 ± 0.92	33.51 ± 1.47	59.20 ± 1.74	1031.77 ± 38.74
<b>Median</b>	23.44 ± 0.23	0.53 ± 0.01	61.21 ± 0.73	178.12 ± 1.04	25.11 ± 0.23	3.46 ± 0.07	24.57 ± 0.20	30.49 ± 0.46	52.10 ± 0.36	779.25 ± 8.03
$M_b$	2.01 ± 0.11	0.09 ± 0.04	22.86 ± 0.47	12.66 ± 0.29	15.78 ± 0.02	0.30 ± 0.01	13.72 ± 0.12	0.47 ± 0.01	26.43 ± 0.29	66.07 ± 0.01

423

424 **3.4 SBR + BR to OM and metals correlation**

425 The distribution of OM content, originating from vegetation decay, microorganisms and  
 426 anthropogenic sources, e.g. petroleum byproducts, in the Cheviré infiltration pond (Badin *et al.*  
 427 2009; Durand *et al.* 2005) ranged from 5.22% to 11.34% of the dry sample. Along with metals,  
 428 this OM content shows an enrichment towards the overflow section, representing an average  
 429 increase of 57% for OM and 63% for metals, respectively (Fig. 5).



430  
 431 Figure 5: Distribution of SBR + BR (median of replicate, in mg/g, < 500 μm) and the relative  
 432 abundance of OM (green diamonds), As, Cd, Cr, Cu, Li, Mo, Ni, Sb, V and Zn elements (< 2  
 433 mm) (curves) for the entrance (E<sub>1</sub>, E<sub>2</sub>, E<sub>3</sub>), middle (M<sub>1</sub>, M<sub>2</sub>, M<sub>3</sub>) and overflow (O<sub>1</sub>, O<sub>2</sub>, O<sub>3</sub>)  
 434 sections. For the relative OM and metal abundance, sample E<sub>1</sub> (i.e. the lowest SBR + BR  
 435 concentration) was set as the reference.

436 A similar trend had been previously identified in the study by Clozel *et al.* (2006). Notably, the  
 437 Pearson correlation coefficient between OM and metals ranged from 0.857 to 0.960 ( $p < 0.05$ )  
 438 (Table 2), which reinforces the significance of OM as a crucial carrier phase for metals in the  
 439 sediment matrix. The distinctive features of OM, including its large specific area, abundant  
 440 sorption sites involving hydroxyl and carboxyl functional groups, and affinity for metals  
 441 through chemisorption and physisorption, further contribute to the adsorption process and thus

442 limit both the mobility of ionic elements and bioavailability of metallic compounds in the soil  
443 or sediment matrix (He *et al.* 2019; Shi *et al.* 2018; Tedoldi *et al.* 2021). As a result, OM may  
444 function as a metal reservoir in such matrices (Sebastia *et al.* 2008).

445 Moreover, TRWP could potentially function as a carrier phase for metals, as evidenced by the  
446 significant positive correlation with SBR + BR. The Shapiro-Wilk test has confirmed the  
447 assumption of a high probability of a normal distribution for each variable, with p-values > 0.05  
448 (Table SI 6). All Pearson and Spearman rank coefficients for a confidence level of 95% fell  
449 below the 0.05 significance level, with values ranging from 0.739 to 0.824 and 0.720 to 0.945,  
450 respectively, for As, Cd, Cr, Cu, Li, Mo, Ni, Sb, V and Zn (Table 2, further details regarding  
451 Spearman's rank correlation coefficients and Quantile-Quantile plots for the qualitative  
452 assessment are provided in Table SI 7 and Figure SI 4). Rødland *et al.* (2023) first sought to  
453 correlate tire wear particles (as quantified by pyrolysis-GC-MS) with Zn, Ni, Pb, Cr, Cu and  
454 As metals using soil matrices from nearby roads.

455 Though surprising, these results showed no significant correlation between tire wear particles  
456 and the targeted metals, in a finding similar to that of Unice *et al.*'s (2013) correlations between  
457 TRWP from river sediment and brake wear-related metals (i.e. Cu, Pb and Zn). The authors  
458 attributed this lack of correlation to the presence of multiple metal sources, different particle  
459 transport mechanisms and a higher background level of metals in the matrix. The significant  
460 positive correlation identified in our study between SBR + BR and metals, specifically  
461 exogeneous metals not originating from the tire tread composition (such as As, V, Cr, Ni, Cu  
462 or Sb (Adachi and Tainosho 2004)), confirms the role of TRWP as a pathway for metal  
463 contamination in an infiltration pond sediment and/or exhibits analogous behavior, in taking  
464 the sedimentary process into account. This correlation points to two likely scenarios of TRWP  
465 and metals aggregation processes: one occurring at the tire-road interface, the other within the  
466 infiltration pond sediment.

467 The former process may take place during the formation of TRWP heteroaggregates, wherein  
468 abrasion mechanisms incorporate mineral fragments from the road and surface deposits of  
469 particulate metals originating from road wear or vehicle-related components (Zannoni *et al.*  
470 2016). As a result, TRWP becomes enriched in metals (Kreider *et al.* 2010), thus contributing  
471 to the dissemination of metal contamination into the infiltration pond through runoff. The latter  
472 aggregative process is expected to occur within the sediment of the infiltration pond, involving  
473 OM that facilitates the relationship between TRWP and particulate and/or dissolved metals.  
474 OM is known to be a key agent of aggregation, forming linkages between particles and thereby  
475 creating stable heteroaggregates (Badin *et al.* 2009). This effect is suggested by the significant  
476 correlation both between OM and metals (median Pearson correlation coefficient  $r = 0.950$ ,  $p$ -  
477 value  $< 0.05$ ) and between SBR + BR and OM ( $r = 0.801$ ,  $p$ -value  $< 0.05$ ).

478 Kovoichich *et al.* (2021) reported that as TRWP undergoes aging in a sediment matrix, it forms  
479 aggregates not only with minerals but also with other environmental particle sources. Therefore,  
480 our data specify that metals and OM could contribute to these aging and aggregation processes  
481 in the presence of TRWP. SEM-EDX observations of TRWP particles collected during  
482 emissions from our previous study using an instrumented vehicle (De Oliveira *et al.* 2024), as  
483 well as the infiltration pond sediment from O<sub>3</sub> sample (i.e. the most contaminated in TRWP),  
484 support the aggregative processes of carbonaceous, ocean-derived or terrigenous minerals and  
485 metals phases (Figures SI 5 and 6). Moreover, Unice *et al.* (2013) found significant correlations  
486 between TRWP and both total organic carbon and finer grain sizes; such findings suggest that  
487 TRWP might also be acting as a surrogate for finer materials. From an environmental standpoint  
488 and depending on their formation, these heteroaggregates may function as carriers for both  
489 metals and tire additives, thereby facilitating the diffusion of a mix of hazardous compounds  
490 throughout diverse environmental compartments, including water bodies, soils, sediments and  
491 the atmosphere (Järlskog *et al.* 2022).



492

Table 2: Pearson correlation coefficient (above the gray diagonal) with its associated p-values (below the diagonal).

493

All p-values lie below the 0.05 significance threshold for SBR + BR, OM and the metals variables

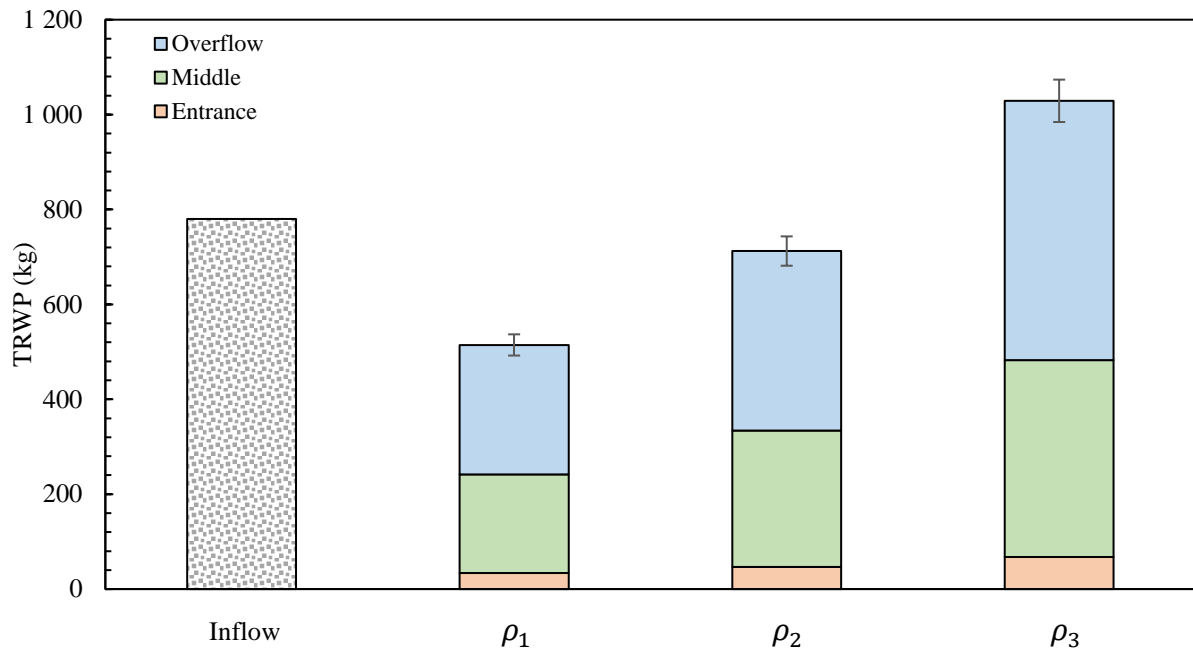
	SBR + BR	OM	As	Cd	Cr	Cu	Li	Mo	Ni	Sb	V	Zn
SBR + BR		0.801	0.795	0.824	0.741	0.792	0.824	0.818	0.762	0.755	0.739	0.778
OM	9.43E-03		0.914	0.954	0.953	0.902	0.960	0.857	0.959	0.894	0.958	0.950
As	1.04E-02	5.63E-04		0.903	0.957	0.968	0.961	0.963	0.966	0.950	0.970	0.963
Cd	6.35E-03	6.75E-05	8.50E-04		0.906	0.885	0.893	0.860	0.892	0.815	0.907	0.943
Cr	2.24E-02	6.99E-05	5.22E-05	7.69E-04		0.927	0.945	0.905	0.989	0.924	0.988	0.947
Cu	1.09E-02	8.73E-04	1.79E-05	1.54E-03	3.28E-04		0.962	0.984	0.946	0.938	0.948	0.978
Li	6.26E-03	4.17E-05	3.66E-05	1.18E-03	1.24E-04	3.36E-05		0.931	0.976	0.966	0.965	0.959
Mo	6.99E-03	3.19E-03	2.98E-05	2.92E-03	8.01E-04	1.55E-06	2.60E-04		0.913	0.937	0.915	0.943
Ni	1.70E-02	4.38E-05	2.26E-05	1.23E-03	4.79E-07	1.17E-04	7.34E-06	5.96E-04		0.943	0.992	0.956
Sb	1.86E-02	1.13E-03	9.05E-05	7.41E-03	3.73E-04	1.82E-04	2.30E-05	1.99E-04	1.37E-04		0.937	0.907
V	2.29E-02	4.69E-05	1.44E-05	7.24E-04	5.37E-07	1.02E-04	2.64E-05	5.43E-04	1.80E-07	1.97E-04		0.972
Zn	1.36E-02	8.62E-05	3.07E-05	1.36E-04	1.07E-04	5.43E-06	4.41E-05	1.39E-04	5.51E-05	7.26E-04	1.13E-05	

494

### 495 3.5 Infiltration pond as a sink for TRWP

496 We next evaluated the pond's theoretical effectiveness in retaining TRWP by estimating both  
497 the inflow of TRWP into the pond and the total amount retained in the sediment by means of  
498 Equations 2 and 3, respectively. TRWP inflow was grounded in realistic TRWP emissions,  
499 specifically using the TRWP emission value derived from the tire weight loss (TWL) parameter.  
500 Our previous study on realistic emissions, conducted with an instrumented vehicle, determined  
501 the TWL for the right front driving wheel equal to 11.0 mg of tire per km, or 16.6 mg of TRWP  
502 per km for the ring road track connecting to the Cheviré infiltration pond (De Oliveira *et al.*  
503 2024). In assuming a TRWP emissions per vehicle per km with a wheel contribution factor ( $W$ )  
504 of 2.7 (Luhana *et al.* 2004), a traffic density ( $T$ ) of 88,500 vehicles per day, a time frame ( $t$ ) of  
505 247 days and a given distance ( $d$ ) from the southern section of the bridge (i.e. 0.8 km), the  
506 theoretical inflow of TRWP into the pond could be approximated at 780 kg (Fig. 6). We then  
507 compared this value with the accumulated and retained TRWP in the sediment.

508 To estimate the amount of TRWP retained in the sediment, we considered three sediment  
509 density hypotheses ( $\rho_1, \rho_2, \rho_3$ ), corresponding to silt, TRWP and coarser sediments (with  
510 densities of 1.3, 1.8 and 2.6 g/cm<sup>3</sup>), respectively. The amounts estimated from  $\rho_1$  and  $\rho_3$  were  
511 deliberately based on extreme density scenarios: either the sediment consists entirely of silt or  
512 entirely of coarse sediment. This approach allowed us to establish rough constraints for low and  
513 high estimates. For  $\rho_2$  however, the density selected was closer to the actual nature of the  
514 sediments studied. All three calculations were based on: the surface area of each section, a  
515 gradual sediment thickness of 1 cm, 1.5 cm and 2 cm from entrance to overflow, a median SBR  
516 + BR concentration per section, and a mean SBR + BR-to-TRWP conversion factor of 9.12, as  
517 obtained from the literature (Klößner *et al.* 2021; Kreider *et al.* 2010; Rauert *et al.* 2021;  
518 Sommer *et al.* 2018; Unice *et al.* 2012).



519

520 Figure 6: Estimation of the TRWP content (kg) retained within the sediment based on three  
 521 hypotheses ( $\rho_1, \rho_2, \rho_3$ ), as outlined in Section 3.5, along with an estimation of the TRWP  
 522 amount (kg) as inflow to the infiltration pond derived from realistic emissions, as reported in  
 523 our previous study (De Oliveira *et al.* 2024) (gray dotted histogram bar). The error bars for  $\rho_1$ ,  
 524  $\rho_2$  and  $\rho_3$  represent the standard deviation of the triplicates obtained from the results  
 525 discussed in Section 3.1. The error bar for inflow has been derived from the *TWL* result,  
 526 whose measurement variation equals 0.1% and is therefore indistinguishable from the line  
 527 thickness of the histogram.

528

529 The results obtained were:  $515 \pm 22$ ,  $712 \pm 31$  and  $1,029 \pm 45$  kg of TRWP for  $\rho_1, \rho_2$  and  $\rho_3$ ,  
 530 respectively. Since,  $\rho_2$  better reflects the characteristics of the sediment, it is noteworthy that  
 531 the amount of TRWP retained in the infiltration pond is comparable to the inflow. Despite the  
 532 assumptions adopted during the calculations and the potential for vertical TRWP migration into  
 533 the substratum or a small fraction of tire wear not reaching the infiltration pond, both values  
 534 remain lie on the same order of magnitude, i.e. approximately 700 to 800 kg of TRWP.

535 These findings indicate that only a small fraction of TRWP is transferred to the atmosphere,  
536 with the majority remaining and evolving at the road surface (as confirmed by our previous  
537 study, see De Oliveira *et al.* (2024)). In addition, a large fraction of TRWP is remobilized from  
538 road surfaces by runoff, ultimately heading towards the infiltration pond. Moreover, the mass  
539 balance supports limited vertical TRWP enrichment of the substratum, thus confirming that the  
540 Cheviré infiltration pond function is an efficient infrastructure for retaining TRWP and  
541 preventing their dispersal into the downstream environment. The potential of an infiltration  
542 pond to act as a TRWP sink has also been confirmed, to a similar extent as what has been  
543 reported for other microplastics by García-Haba *et al.* (2023) and Olesen *et al.* (2019), with  
544 sedimentation processes playing a key role in microplastic retention. More specifically,  
545 Gilbreath *et al.* (2019) estimated a reduction in microplastic concentrations of between 83%  
546 and 95% in a bioretention system. Similarly, Smyth *et al.* (2021) observed reductions exceeding  
547 80% throughout a bioretention cell, while Jakubowicz *et al.* (2022) documented a minimum  
548 reduction efficiency of 77% for microplastics in a multistage constructed wetland.

### 549 **3.6 Uncertainties and future challenges**

550 Several inherent uncertainties arise when studying TRWP environmental contamination,  
551 particularly when relying on mass spectrometry methods. Among these uncertainties, three  
552 critical points are highly relevant to the scientific community and will be discussed below.

553 The first point pertains to the specificity of the pyrolytic markers. As highlighted by Rødland  
554 *et al.* (2022), the accuracy of rubber quantification is heavily dependent on the markers used to  
555 identify and quantify TRWP heteroaggregates. To address the uncertainty associated with the  
556 variations in single markers, these authors employed a method based on a combination of four  
557 different pyrolysis products, namely benzene,  $\alpha$ -methylstyrene, ethylstyrene and a butadiene  
558 trimer to assess SBR + BR content in tires, as well as the styrene-butadiene-styrene (SBS)

559 content in polymer-modified bitumen used in some road asphalt. Our study has adopted a  
560 similar approach, in targeting two specific markers to evaluate SBR + BR content. However,  
561 as noted by Rødland *et al.* (2022), SBR and SBS polymers share similar pyrolytic markers, such  
562 as the 4-VCH, with some SBS markers overlapping those of BR. To assess the potential  
563 overestimation of TRWP concentration, the authors recommend initially investigating the  
564 presence of SBS in the road surface prior to analyzing samples for SBR or BR. In the context  
565 of our study, conducting this preliminary analysis is currently infeasible due to restricted access  
566 to the bridge road, thus preventing *in situ* sampling. One potential solution would be to obtain  
567 detailed information on the bitumen composition from industries, which could then be uploaded  
568 to a collaborative open-source database, as proposed by Rødland and Lin (2023) for tire  
569 composition. Alternatively, employing a more selective analytical technique, such as tandem  
570 or high-resolution mass spectrometry, could facilitate the targeted analysis of SBR or BR while  
571 minimizing interference from SBS. Another solution would be to perform a quantification  
572 based on just 4-PCH as a marker for SBR, which would then eliminate interference from SBS  
573 since this marker is not produced by the SBS polymer.

574 The second point is the aging of TRWP. Given that these particles age in the environment, they  
575 undergo various physical and chemical transformations capable of affecting both the polymeric  
576 content of TRWP and their pyrolytic markers, hence potentially impacting the accuracy of  
577 quantification (Rødland *et al.* 2022; Wagner *et al.* 2022). This consideration calls into question  
578 a key assumption in many studies, including ours, regarding the assessment of TRWP in the  
579 environment, namely: the pyrolysis of environmental TRWP, despite their aging and the  
580 potential oxidation of their polymeric fractions over time, has a pyrolytic yield equivalent to  
581 that of tire tread or SBR and BR standards, which have not undergone any chemical or physical  
582 transformations.

583 In addition of aging process, the impact of vulcanization on the pyrolytic yield of the polymeric  
584 fraction in tires, compared to unvulcanized polymer standards, is also an important area of  
585 research worth considering. Vulcanization is known to influence rubber cracking and promote  
586 key processes such as dehydrogenation, aromatization and polymerization during pyrolysis  
587 (Sun *et al.* 2022). Further investigation is thus required to fully understand how exactly  
588 environmental aging and vulcanization influences the reliability of Pyr-GC-MS analyses in  
589 quantifying TRWP.

590 The third and final point concerns the representativeness of the SBR + BR-to-TRWP conversion  
591 factor. The main limitation herein lies in the consistency between the polymeric content of the  
592 samples and that of the tires, as well as the encrusted particle content of TRWP (Klößner *et*  
593 *al.* 2021; Rødland *et al.* 2022). Variations in tire brand, tire type (winter, summer, all-season),  
594 vehicle weight and type (thermal or electric), market location, sampling site, road conditions,  
595 type of bitumen, and traffic density can all influence both the polymeric and mineral content of  
596 TRWP. These factors inevitably introduce a level of approximation when calculating the  
597 equivalent amount of SBR + BR in TRWP. To minimize this level of approximation, we have  
598 calculated the TRWP content using a median SBR + BR-to-TRWP conversion factor of 9.12,  
599 which accounts for both the minimum and maximum levels of particle encrustment and  
600 polymeric content (Kreider *et al.* 2010; Rauert *et al.* 2021; Sommer *et al.* 2018; Unice *et al.*  
601 2012). This approach could be further refined with access to a representative mix of tire brands  
602 and types used in France or Europe, in providing a more accurate estimate of the average  
603 polymeric content. However, without a specific *in situ* assessment of TRWP encrustment and/or  
604 density, relying on a median value of encrustment from the literature remains the most practical  
605 solution. It is obvious that future studies assessing tire particles in the environment should  
606 systematically include both the quantified content of the polymeric fractions, based on the  
607 selected polymer, and the corresponding conversion factors for estimating TWP or TRWP

608 equivalence. This approach is aimed at progressively standardizing methodologies and ensuring  
609 consistency across studies, thereby empowering researchers to accurately compare data and  
610 facilitate future meta-analyses, in ultimately leading to a better understanding of the  
611 environmental implications of these tire particles.

612 Although these uncertainties are present in our study, we still anticipate that the regular  
613 introduction of freshly generated TRWP into the infiltration pond may mitigate the effects of  
614 aging on the overall TRWP concentrations over the short 8-month period of sediment  
615 accumulation. Moreover, the estimated amount of TRWP inflow, based on the realistic local  
616 weight loss of tires, and the TRWP amount retained in the sediment remain relatively close, i.e.  
617 with a difference of  $< 9\%$ . This finding supports the notion that the SBS pyrolytic interferences  
618 with SBR or BR, as well as the potential effects of aging on polymeric quantification, are likely  
619 to be limited. If present, these factors might account for a maximum variation of 9% in our  
620 study. As for the reliability of this conversion factor, it does not affect the overall trend observed  
621 within the infiltration pond but may call into question the TRWP values when compared to  
622 other studies using different conversion factors.

623 To shed new light on the formation of TRWP heteroaggregates and given that our data suggest  
624 metals and organic matter contribute to the aggregation process in the presence of TRWP, a  
625 more in-depth characterization of the surface properties and reactivity becomes imperative.  
626 This step is crucial due to the current lack of such information in the scientific literature and  
627 when considering the possible adsorption of dissolved metals. Moreover, increasing the number  
628 of sampling points to achieve a better distribution across the pond and comparing SBR + BR  
629 with the OM and metals variables within the same size fraction and granulometric range is  
630 expected to significantly refine the correlation, improve the statistical representativeness of the  
631 data and help determine whether the observed associations between TRWP with OM and metals  
632 are causal. These future steps are particularly important given that we currently possess a

633 limited number of observation (i.e. nine samples), as compared to the number of variables (i.e.  
634 twelve variables including SBR + BR, OM and metals), and have used different sieving  
635 thresholds of 500  $\mu\text{m}$  for TRWP and 2 mm for OM and metals analysis (see Sections 2.3 and  
636 2.4). Additionally, further analysis using SEM and EDX techniques would be necessary to  
637 characterize the intricate relationship of heteroaggregates containing OM, metals and TRWP  
638 within the sediment. From these findings, utilization of the SBR + BR polymeric fraction from  
639 tires could serve as a reliable indicator of metal contamination in stormwater systems.

640 On a broader level and from a management perspective, data are currently lacking on TRWP  
641 contamination thresholds in environmental matrices, notably in stormwater systems sediment.  
642 Establishing these thresholds is crucial to determining the stormwater system maintenance  
643 schedule and the fate of the sediment post-removal as regards TRWP contamination.  
644 Collaborative efforts by the scientific community and public policymakers should be directed  
645 at bridging this gap.

646 One innovative approach to addressing this gap entails considering SBR + BR not only as a  
647 proxy for TRWP but also as an indicator of metal contamination in the stormwater system  
648 environment. Comprehensive analyses across multiple stormwater system sites are essential, in  
649 order to examine both SBR + BR levels and metal pollution while accounting for variations in  
650 watershed surface use and traffic density. Subsequently, we could propose an innovative and  
651 novel correlation factor between SBR + BR and metals, to facilitate the assessment of TRWP  
652 or metal levels, whereby one can be used as a proxy for the other depending on the analytical  
653 methods available (e.g. Pyr-GC-MS, ICP-MS or portable X-ray fluorescence). In addition, by  
654 using such a correlation, preliminary TRWP thresholds could be established for sediment  
655 matrices based on existing metal contaminant thresholds, thereby serving to both determine the  
656 hazardous sediment level and schedule stormwater system maintenance that targets TRWP by  
657 means of Pyr-GC-MS.



### 658 **3.7 Management recommendations**

659 The implementation of optimized sediment removal practices is invaluable for mitigating the  
660 potential of a SuDS to become a source of pollution when cleaning actions are neglected  
661 (Humphrey *et al.* 2023). Numerous studies have revealed that SuDS can achieve high retention  
662 rates, exceeding 79% and sometimes reaching up to 100%, for total suspended solids as well as  
663 for contaminants such as polychlorinated biphenyls, polycyclic aromatic hydrocarbons, total  
664 nitrogen, total phosphorus, mainly due to their affinity for fine sediment particles and ability to  
665 settle (DeBusk and Wynn, 2011; Gilbreath *et al.* 2019; Jakubowicz *et al.* 2022; Maurer *et al.*  
666 2021). In light of these studies and our findings demonstrating that infiltration ponds are also  
667 capable of retaining TRWP, high retention rates would imply a progressive accumulation of  
668 contaminants in the sediment layer. Attention therefore must still be drawn to potential  
669 stormwater systems limitations as regards hazardous TRWP constituents.

670 The inherent nature of the Chevire infiltration pond, as characterized by the absence of an  
671 impermeable layer at the bottom, combines with intricate physicochemical and biological  
672 processes within the pond, leading over time to the photooxidation and microbial degradation  
673 of TRWP heteroaggregates and, hence, the leaching of hazardous constituents (Baensch-  
674 Baltruschat *et al.* 2020; Ding *et al.* 2023; Klun *et al.* 2023; Wagner *et al.* 2022). Metals, in  
675 particular, can leach into and accumulate in sediments, with some elements being easily  
676 remobilized due to their intrinsic mobility and affinity for the dissolved phase. These processes  
677 are influenced by physicochemical parameters of the medium, such as pH, ionic strength,  
678 competing ions, sorption sites and their affinity for dissolved organic matter. Under specific  
679 conditions, it is not uncommon for certain metals, such as copper or nickel, to exhibit poor or  
680 even negative retention rates (Humphrey *et al.* 2023; Jakubowicz *et al.* 2022).

681 This outcome raises concerns over the spread of such environmental micropollutants (Maurer  
682 *et al.* 2021). The infiltration of micropollutants, including those from TRWP leachates, deep  
683 into the alluvial substratum of the Loire River beneath the pond and, consequently, into  
684 groundwater contaminating water bodies (Tedoldi *et al.* 2017) can indeed be anticipated. It  
685 therefore becomes necessary to analyze core samples taken from the infiltration pond  
686 substratum in order to assess: i) the occurrence and concentration of the targeted  
687 micropollutants over substratum depth, and ii) overall relevance of the infiltration pond relative  
688 to a broader hazardous spectrum (Maurer *et al.* 2021).

689 The acquisition of data regarding contamination levels of TRWP and their spatial distribution  
690 in a stormwater system is necessary to establish an effective water management strategy.  
691 Chemical analysis targeting TRWP, coupled with a topographic and size fractionation  
692 assessment, has revealed the pollutant footprint of the entire Cheviré infiltration pond. The  
693 information gathered can then serve as a foundation for developing a cost-effective remediation  
694 strategy by means of prioritizing specific sections of the infiltration pond, namely the most  
695 contaminated ones. The recommended strategy involves cleaning efforts in areas 2 and 3,  
696 located in the middle and overflow sections, before addressing sediment removal in the entrance  
697 section and area 1. This strategic sequence would aim to efficiently target and address the areas  
698 of maximum TRWP accumulation. Given the intricate relationship between TRWP and metals,  
699 such a cleaning effort intended for TRWP removal will also effectively remove metals by  
700 focusing on the same areas of accumulation, thereby optimizing the overall remediation  
701 process.

702 Although infiltration ponds offer the advantage of capturing and retaining a significant amount  
703 of TRWP, as well as a fraction of the associated metal pollutants, their implementation in areas  
704 with high anthropogenic pressure and high traffic density, e.g. urban centers, remains highly  
705 challenging. Several major constraints are to be considered. First, the number, size and

706 associated costs of these installations will depend on the drained surface area and expected  
707 volume of particulate runoff, yet densely populated areas offer limited to no space for  
708 constructing such infrastructure. Second, infiltrated water could potentially damage nearby  
709 preexisting infrastructure, such as underground electrical networks. Third, soil quality in urban  
710 areas is often compacted, thus making it unsuitable for effective infiltration. Fourth and last is  
711 the presence of a groundwater table contamination risk (U.S. EPA, 2021). Under these  
712 conditions, infiltration ponds are preferentially constructed in suburban areas. One potential  
713 solution could be to adapt existing urban drainage infrastructure by increasing the use of gully  
714 pots with sediment traps. This approach has proven to effectively trap a variety of organic and  
715 particulate contaminants, such as microplastics and TRWP (Mengistu *et al.* 2021; Öborn *et al.*  
716 2024; Rødland *et al.* 2023; Wei *et al.* 2023). However, gully pots cover a much smaller drainage  
717 area and have limited capacity, as compared to infiltration ponds. Therefore, it would seem  
718 crucial to increase the number of gully pots and ensure their regular maintenance, including  
719 frequent cleaning of the sediment trap. Like with infiltration ponds, without proper  
720 maintenance, gully pots are at risk of sediment remobilization during heavy rainfall events  
721 and/or lixiviation of TRWP, which could further propagate pollution through the pipe network  
722 and, ultimately, into water bodies if discharges are directly connected.

723 **4. Conclusion**

724 This study has significantly contributed to enhancing knowledge on the fate of TRWP within  
725 an infiltration pond and moreover emphasizes the pivotal role of such infrastructure in  
726 managing TRWP pollution. Through a sampling strategy covering the surface of the Cheviré  
727 infiltration pond and leveraging a multi-characterization technique, namely the pyrolysis-GC-  
728 MS method, to target the polymeric signature of tires, we have been able to address the points  
729 raised in the introduction and have derived the following key findings:

- 730 i. Extensive SBR + BR contamination of the infiltration pond sediment has been found  
731 to lie in the mg/g range, but at various degrees depending on the location of the  
732 sample, with enrichment towards the overflow. Two sections (i.e. middle and  
733 overflow) have been identified as major zones of SBR + BR accumulation, which  
734 aligns with the topographic configuration of the pond.
- 735 ii. The size fractionation of SBR + BR within the sediment reveals a bimodal  
736 distribution, thus indicating two distinct types of TRWP. The one characterized by  
737 a smaller size (63-160  $\mu\text{m}$ ) suggests TRWP less aged and aggregated with other  
738 environmental particles. The second type, characterized by a larger particle size  
739 (200-500  $\mu\text{m}$ ), suggests higher environmental particle encrustation and thus more  
740 aged TRWP.
- 741 iii. A strong correlation has been detected between SBR + BR and metals, such as As,  
742 Cd, Cr, Cu, Li, Mo, Ni, Sb, V and Zn. This relationship can be attributed to either  
743 the preexistence of metals in the tire composition or the incorporation of metals not  
744 originating from the tire. This type of incorporation occurs during the formation of  
745 TRWP heteroaggregates at the road surface and/or within the sediment, with organic  
746 matter facilitating the process.

747       iv.     The approximate mass balance estimation between TRWP inflow from realistic tire  
748             loss and the amount retained within the sediment supports a limited mass transfer of  
749             TRWP emissions to the atmosphere and has moreover confirmed the role of an  
750             infiltration pond as a major sink for TRWP from runoff.

751     This study paves the way for an effective management strategy of stormwater system sediments  
752     contaminated with TRWP. Given the wide range of contaminants in stormwater system  
753     sediment, including metals, we would recommend focusing cleaning efforts on those areas with  
754     the highest TRWP accumulation. In conclusion, let's reiterate the importance of conducting  
755     routine chemical analyses on sediment from stormwater systems. This practice is indeed  
756     essential to enhancing management strategies aimed at ensuring that such infrastructure, and  
757     more specifically infiltration ponds intended as sinks, does not evolve into sources of pollution  
758     for the downstream environment.

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766

767 **Declaration of interests**

768 The authors hereby declare that they have no known competing financial interests or personal  
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770

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