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Tracks of travel: unveiling tire particle concentrations in Swiss cantonal road soils

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Abstract

Tire wear particles (TWP) originating from tire abrasion on roads are a major source of microplastics to the environment. Together with associated pollutants like polycyclic aromatic hydrocarbons and trace metals, TWP are emitted to roadside soils in the immediate vicinity of road networks. Our study aimed at quantifying TWP number and mass concentrations and investigating particle features in low-traffic roadside soils using a novel particle-based analytical approach. On the example of fifteen Swiss cantonal roadside soils, with average daily traffic volumes of 2,290 vehicles per day^{-1}, we sampled composite samples from distances of 1, 2, 5 and 10 m to the roadside. TWP were extracted via density separation and wet-chemical sample purification. TWP analysis was performed using microscope images and trainable Weka segmentation image analysis. Furthermore, associated road pollutants like polycyclic aromatic hydrocarbons, benzop[a]pyrene and trace metals were analysed using TQ GC-MS/MS and ICP-MS. We found average concentrations of 111,000 TWP per kg soil dry weight (TWP kg^{-1}) highest values reaching 615,000 TWP kg^{-1} and mean TWP masses of 52.7 ± 83.2 mg TWP kg^{-1}. TWP had a minimal Feret diameter of 62.8 ± 45.6 µm on average and showed mean circularity values of 0.7 ± 0.2, resulting in elliptic particle morphology. TWP concentrations and sizes decreased with increasing distance from the road. Positive relationships were found between TWP numbers and polycyclic aromatic hydrocarbons, benzop[a]pyrene and zinc concentrations in roadside soils. However, a moderate relationship to speed limits was identified. We were able to demonstrate that even in low-traffic areas, roadside soils act as an environmental sink for high concentrations of TWPs and associated pollutants and that spatial distribution and the spread of TWP to soils strongly dependent on the distance to the road.

Keywords Tire wear particles, Image analysis, Organic compounds, Trace metals, Traffic

Introduction

Global microplastic emissions and their distribution across all environmental systems have negative consequences for almost all ecosystems. In recent years, tire wear particles (TWP) have been found to represent a significant part of microplastics emitted globally [1–3]. TWP are generated by the abrasion of vehicles' tires on the road while the interaction of abrased particles with the road surface [4] resulting in TWP as hetero-aggregates of tire material encrusted with minerals from the road in particle sizes between < 40 nm up to 400 µm [5–7]. While the tire composition can vary widely [8],

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tires are generally composed of about 40–50% natural or synthetic (styrene-butadiene) rubber, fillers (e.g., black carbon, 30–35%), softeners (e.g., oil, 15%), additives as well as vulcanization agents containing sulfur (S) and zinc oxide (ZnO) (2–5%) [9]. Based on the trace metal contents in tires itself in addition to other traffic-related pollutants, such as metals from brake pads or polycyclic aromatic hydrocarbons (PAHs) and metal emissions from combustion gases among others [10–12], roadside soils are known to be pollution hotspots of tire additives [4, 13]. When TWP and subsequently tire additives are emitted to the environment, effects on soil properties, plants and other organisms have been detected [7, 9]. Furthermore, TWP can have effects on soil nitrogen and carbon cycling [14, 15], while toxicity arises from TWP digestion or leaching of associated chemicals [16, 17]. TWP are liable to the leaching of trace metals, in particular zinc (Zn) [18], as well as various elements like sulfur (S), organic compounds including polycyclic aromatic hydrocarbons (PAHs) or Benzop[a]pyrene (BAP) [19, 20].

While TWP are first formed and deposited on road surfaces, they are transported to other environmental compartments by road runoff or air emissions [4]. Therefore, TWP are likely to reach nearest roadside soils easily, but also soils further away due to air turbulence induced by high-speed vehicles, wind, and spray water [4, 21]. Although roadside soils are known as pollution hotspots for the classical pollutants such as trace metals and various non-polar organic compounds, data on TWP abundance in roadside soils is very limited [16, 22]. Most of the available data are model-generated predictions of the TWP amount emitted to soils. For example, Kole et al. (2017) predicted an amount of 0.23–4.50 kg $year^{-1}$ tire material emitted per capita in 13 countries, while Sieber et al. (2020) estimate a rubber emission of 0.96 ± 0.35 kg $capita^{-1}$ $year^{-1}$ for Switzerland and and Bertling et al. (2018) 1.15 kg $capita^{-1}$ $year^{-1}$ for Germany [23, 24].

Data on TWP in roadside soils from actual environmental measurements is only available from three studies, reporting TWP mass concentrations in roadside soils. Rødland et al. (2022, 2023) reported 3700–4900 mg kg^{-1} TWP (0–6 m distance from road) for annual average daily traffic volumes (DTV) of 77,000 vehicles per day as well as 2040–26,400 mg kg^{-1} TWP for DTV below 14,250 vehicles per day in Norway [25, 26]. Furthermore, Müller et al. (2022) found ranges of 155–15,900 mg kg^{-1} TWP (0.3–5 m distance from road) in Germany for DTV of 36,000 vehicles per day [27]. Despite the limited number of studies, the mass concentrations may reach levels comparable to soil organic matter in most agricultural soils [28]. For high-traffic sites, a decrease of TWP concentrations with increasing distance from the road was found [26, 27], while no such decrease was found for low traffic sites [25].

In view of the fact that roadside soils represent an environmental sink for TWP and associated pollutants, the transport behaviour of TWP must be taken into account. Various studies have shown, that transport via runoff water dominates short-distance transport (< 2 m), while splash and spray transport dominated medium-distance transport (2–10 m) and transport via air dominates longer (> 10 m) distances [25, 29]. Resulting distribution patterns are similar to those of other traffic-related pollutants, especially for trace metals such as Zn or lead (Pb) [13]. Furthermore, recent TWP studies for roadside soils, but also for other matrices such as road dust, road snow or road runoff have found a significant correlation between pollutant concentration and daily traffic volumes [30, 31] or other street and traffic-related data (e.g., traffic speed) [25]. The pollutants in roadside soils might be directly associated with the TWP but might also have other traffic-related sources (e.g., from exhaust emissions).

Various analytical methods are available for TWP quantification, generally divided in particle-based (e.g., light microscopy, micro-Fourier transform infrared spectroscopy (μFTIR), scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX) or mass-based (e.g., pyrolysis/ thermo desorption gas chromatography mass spectrometry (Py/TED-GC-MS), inductively coupled plasma mass spectrometry (ICP-MS) methods [5]. For monitoring of TWP quantities in roadside soils, spectroscopic or SEM technics are not sufficient, as they are limited in particle size or time consuming based on their single particle analysis. Previously mentioned studies providing real-world data on roadside environments, therefore used indirect mass-based Py-GC-MS technics focusing on TWP markers like Styrene Butadiene rubber (SBR) and Butadiene Rubber (BR) [25, 32] or TED-GC-MS focusing on cyclo-hexenyl benzene as a specific marker for SBR and therefore TWP [27]. In contrast to the majority of previous studies, we decided to apply a novel, particle-based method according to Foetisch et al. (2024) including a chemical extraction and purification protocol, followed by stereomicroscopy and automated image analysis. The applied method showed an accurate measurement of TWP down to a size of 35 μm and a recovery of > 85% [33]. Particle-based analytical methods have the advantage that "real" particles are measured directly, overcoming difficulties of indirect measurement of polymeric markers due to the diversity of chemical TWP components [33]. Furthermore, beside TWP concentrations, particle-based methods provide information about TWP size and shape distribution, which are important to understand their fate and effects in soils [22, 33].

As mentioned above, data on TWP abundance in soils and associated pollutants, their spatial distribution and

particle properties are vanishingly small, while particle-based TWP data for roadside soils is missing completely. Therefore, our present study aims to unveil particle-based TWP numbers and associated pollutants along low-traffic roads taking the example of cantonal roads in Switzerland. Our study focused on low-traffic roads, as these types of roads usually have no constructional barriers to adjacent soils and no wastewater collection, which allows direct emissions to the roadside soil [8, 13].

More specifically, we **(i)** compare the quality and limitations of the particle-based method with current literature data; **(ii)** investigate the particle- and estimated mass-based abundance of TWP; **(iii)** examine the spatial distribution of TWP and associated pollutants at distance gradients from roads and; **(iv)** analyse relationships between TWP and the associated trace metals, BAP, PAHs concentrations with traffic and road variables.

Materials and methods

Sampling site selection and soil sampling

For TWP and trace metal as well as BAP and PAHs analyses, fifteen cantonal roads with adjacent permanent grassland were selected for sampling. All cantonal roads are located in the canton of Solothurn (Switzerland) and exhibit different ages, road surfaces, daily traffic volumes (DTV) and speed limits. Average DTV based on daily counts and extrapolations for the selected sites were 2,290 vehicles day^{-1} in 2015, where a complete DTV data set was last available from the Office for Transport and Civil Engineering, Canton of Solothurn (Switzerland). More key characteristics of sampling sites and geospatial site locations are provided in Table S1. The chosen road sections exhibited a seamless transition to the adjacent grassland (Fig. 1A). Moreover, these sections featured straight paths without curves or gradients, and the adjoining grassland was characterised by a flat terrain with no record of ploughing. At each site, we collected a total of four samples from the top 10 cm of soil at distances of 1 m, 2 m, 5 m, and 10 m from the road. Spades, stainless-steel shovels and trowels were used to extract soil. Above-ground biomass was removed during sampling. At each of these four distances, three samples were gathered with a 5 m distance (Fig. 2B), and these were subsequently combined into a composite sample, resulting in $N=1$ sample per distance and $N=4$ samples per site (Fig. 1B), contributing to a total of 60 samples across the 15 sites. Samples were transferred into aluminium trays and sealed with a paper lid, stored at 4 °C until they were passed through a 2 mm stainless steel sieve. Samples were subsequently dried at 60 °C, filled into paper bags and stored at room temperature until extraction [5].

Tire wear particle extraction

TWP extraction was performed in the soil analytical lab of the Research Institute of Organic Agriculture. The extraction followed the protocol according Foetisch et al. (2024) including the following procedure [33]: Five grams of sieved and air-dried soil was weighed into

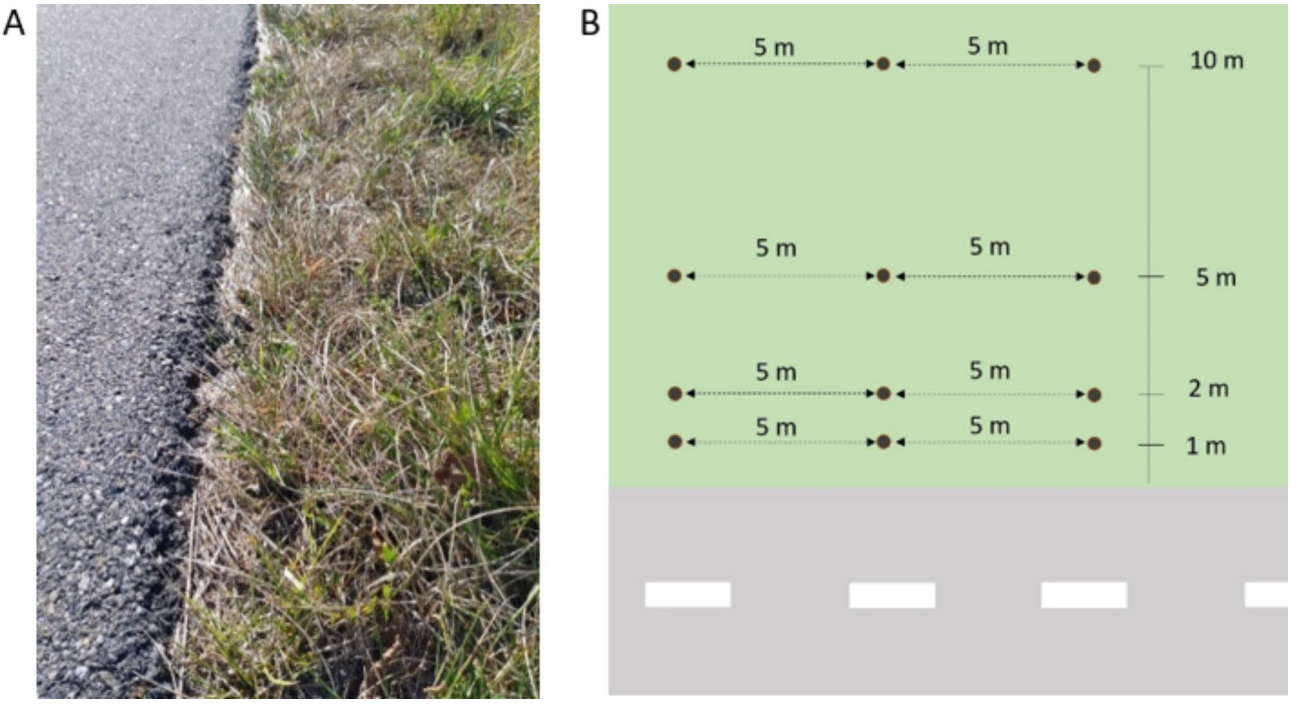

Fig. 1 (**A**) Sampling sites were free of physical barriers between the road to the permanent grassland; (**B**) Sampling scheme: Each three samplings were taken at 1 m, 2 m 5 m and 10 m distance from the roadside and merged into one composite sample per distance

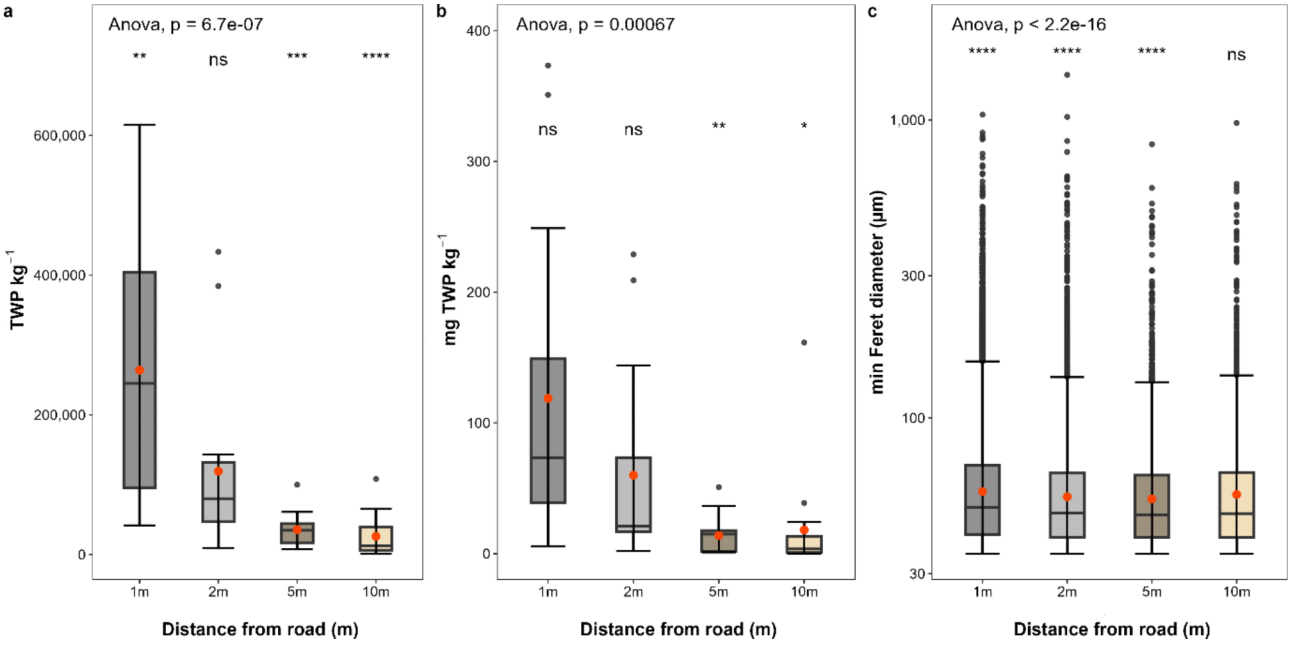

Fig. 2 Tire wear particle (TWP) abundance along cantonal roads ($n = 15$) at different distances. a: TWP concentration (TWP kg^{-1}) at different distances (m) from road; b: TWP estimated mass concentration (mg TWP kg-1) at different distances (m) from road; c: TWP min Feret diameter (µm) at different distances (m) from road (n 1 m = 19,873; n 2 m = 8976; n 5 m = 2,700; n 10 m = 1921). Significance levels: ns = not significant, * $p \leq 0.05$, ** $p \leq 0.01$, *** $p \leq 0.001$, **** $p \leq 0.001$

50 ml centrifuge tubes. For density separation, 35 ml of a sodium bromide (99%, p.a., ACS, Carl Roth GmbH, Germany) solution with a density of 1.5 g cm^{-3} was added. The samples were vortexed, sonicated for 10 min, shaken at 100 rpm for 30 min and centrifuged at 2500 x g for 30 min. Microplastic and organic particles were separated from soil particles by inserting a decanting aid [34] and collected by vacuum-filtration on a 10 µm stainless-steel filter (Rolf Körner GmbH, Niederzier, Germany) with a diameter of 48 mm. Carbonates were removed with 10 ml of 1% (v/v) hydrochloric acid, followed by rinsing with 150 ml of deionized water. To break down the organic components, the particles were incubated at -18 °C in 25 ml of NaOH-urea-thiourea solution (8% (w/v) urea (≥ 99%, puriss. p.a.), 6.5% (w/v) thiourea (≥ 99%, pure), 8%(w/v) sodium hydroxide (≥ 98%) until initiation of crystal formation. Samples were gradually brought back to room temperature while shaking at 100 rpm, subsequently passed through a 10 µm stainless-steel filter and washed in 30 ml of deionized water. The filters were placed particle-side up in a beaker kept on ice. To destroy the organic particles, 15 ml of 0.05 M Iron (II) sulfate heptahydrate (≥ 99%, p.a., ACS) solution and 15 ml of 35% stabilized hydrogen peroxide were added for an overnight Fenton reaction. The following day, filters were rinsed with deionized water before vacuum-filtration of the reaction solution and subjecting the particles to a second density fractionation in 35 ml of sodium bromide with a density of 1.5 g cm^{-3} as described above. The final filtration step was done using a 20 mm filter funnel and a 0.2 µm Whatman® Anodisc Membranfilter with a diameter of 25 mm. To enhance particle visibility, 150 µl of 2 M sulfuric acid (96%, p.a., ISO) was applied after filtration and removed with deionized water.

Tire wear particle analysis

The filters were photographed using a Leica M205C stereomicroscope equipped with a GRYPHAX® SUBRA camera (Jenoptix, Jena, Germany). The camera featured a CMOS 1/1.2" 13.3 mm sensor, with pixel dimensions of 5.86 × 5.86 µm and a resolution of approximately 2.3 megapixels. The stereomicroscope was further equipped with a Leica LED5000 HDI™ dome lighting (Leica Microsystems, Heerburg, Switzerland) to reduce glare and provide shadow-free illumination. Each filter was captured by 120 to 160 individual photos at a 40x magnification and a resolution of 0.5 pixels µm^{-1}. To facilitate seamless stitching of the individual photos into a single panorama picture, an overlap of approximately 25% was implemented. To move the filter during the photographing, we devised a white paper on a motorised metal plate capable of moving the attached filter over specified distances in both the x and y directions. The filter was covered with a coverslip and two small magnets for photographing. The individual images of the samples were stitched together into a panorama using the software "Autostich" (Brown and Lowe, 2007) with the following settings: Output size: Scale, 100%; Blending Options: Multiband, Blending

Bands 2; Advanced options: JPEG quality, 90. Due to the sparse particle content in the blank samples, which were included for contamination controls, it was not feasible to create a complete panorama but had to be stitched in two to three sub parts. Consequently, the individual parts of these filters were analysed separately, with special attention given to preventing any overlap between the blank photos to avoid counting particles multiple times.

The post-processing and analysis of the panorama images were carried out using "Adobe Lightroom" and the open-source platform "Fiji" (ImageJ) [35]. The black image background resulting from merging was transformed into white. Black or very dark particles that were clearly identified as organic or fibres were over-painted in white. Subsequently, the images were edited in Lightroom to address chromatic aberration and, where present, to remove the yellow-blue fringe around the TWP. Additionally, the contrast was maximally increased to ensure that the black particles stood out well from the background. For the analysis of the filters, an algorithm for pixel segmentation was employed in Fiji [36]. This "Weka Segmentation" plugin classified each pixel either as TWP or background. The script was trained using samples from various filters with different particle densities. Since the computer lacked sufficient memory for analysing entire images, the images were divided into tiles using a macro in Fiji. The algorithm analysed the tiles, and the results were subsequently reassembled into the complete image. The identified TWP were characterised using the "Analyze particles" function in Fiji considering a particle size ranging from zero to infinity, and circularity from zero to one.

Trace metal and PAC analysis

Trace metal, BAP and PAHs analyses were conducted at NIUTEC Labor für Industrie und Umwelt (Winterthur, Schweiz). BAP and PAHs were extracted using Soxhlet-Extraktion with Hexan, Aceton, Toluol (10:5:1) thereby following the standard method 3540 of the U.S. Environmental Protection Agency (EPA). Quantification was done using Triple Quadrupole Gas Chromatography Tandem Mass Spectrometry (TQ GC-MS/MS) following the EPA standard method 8270. Lead, cadmium, copper, zinc and mercury was extracted in hot nitric acid 2 mol/l in a ratio of 1:10 using the standard method of the Eidgenössischen landwirtschaftlichen Forschungsanstalten (FAL HNO3-Ex). Total mercury content was quantified with Cold Vapor Atomic Absorption Spectrometry (method DIN EN ISO 12846, E12). The quantification of the Quantification of the metals was done with Inductively Coupled Plasma Mass Spectrometry (ICP-MS, Method EN ISO 17294-1/2). Soil dry weight was assessed gravimetrically after drying soil samples at 105 °C to constant weight.

Quality assurance and quality control

To avoid contamination, a white cotton lab coat was used throughout the extraction process. All extraction steps were conducted under a clean hood. For blank control, an empty tube was included per extraction batch, with each batch containing 15 soil samples, resulting in a total of six blank samples. Moreover, replicated extractions were performed in triplicates for two of the 60 samples. Analysis of blank samples showed a mean TWP concentration of 9.3 ± 2.6 particles per blank sample. In comparison, within environmental soil samples (Figure S1a) an average of 559 ± 739 (range: 6–3089) TWPs were identified, resulting in a significant ($p \leq 0.001$) lower blank concentration. Therefore, a background contamination of "black particles" or TWPs from 1.6% of identified particles can be assumed. In case of replicate samples, we found a relative standard deviation of 2.94–3.14% (Figure S1b).

Laser direct infrared spectroscopy (LDIR) was use to doublecheck if the particles on the filter are TWP. For the LDIR analysis, performed at University of Applied Sciences and Arts Northern Switzerland, particles from six samples were randomly selected, concentrating on either very large particles or those that crumbled under pressure to validate their chemical composition (Figure S2). These particles were placed on a small glass plate and analysed using the 8700 LDIR Chemical Imaging System from Agilent. The analysis involved examining several points per particle using both the official Agilent Database (Ersatz Microplastic Starter 2.0) and internal database. LDIR analysis results within an overall identification of selected particles as styrene-butadiene rubber (SBR) and rubber along with natural cellulose. Hit qualities for particle identification were > 0.93 for SBR and > 0.80 for rubber.

Data handling and statistical analysis

Data was handled and analysed in R (Version 4.2.2) via RStudio Version (2022.02.3) [37]. As shown in Foetisch et al. (2024) particles down to a size of 25 µm can be reliably detected by the segmentation model while beyond this threshold, the accuracy of size estimation becomes unreliable. We therefore excluded any particle with a MinFeret < 35 µm from the data set. To obtain a particle concentration, the mass of the particles was estimated as shown in Eq. 1 [38]:

$$M_{TWP} = \frac{4}{3} \times \left(\frac{Feret}{2}\right) \times \left(\frac{MinFeret}{2}\right) \times \left(\frac{0.372 \times MinFeret}{2}\right) \pi \times \rho \quad (1)$$

Equation 1: With estimated particle mass (M_{TWP}) calculated for each particle using both, the diameter of Feret and MinFeret and the density (ρ) of the particles with ρ =1.2 g cm^{-3}. Particle density for mass estimation was oriented on the maximum density of tire rubber and tire particles without the consideration of road wear particles [39, 40].

TWP concentrations are reported as the number of TWPs per kg soil dry weight (TWP kg^{-1}) as well as estimated particle masses in mg TWP per kg soil dry weight (mg TWP kg^{-1}).

Statistical operations including data visualization, tests for normal distribution of the residuals (Shapiro-Wilk), test for mean differences, linear regression analyses, and variance analyses (ANOVA) were conducted using base R and the R-package "ggplot2" [41]. We analysed mean differences (ANOVA) between the four sampling distances using data of 15 sites (n = 15 per distance) with TWP kg^{-1} and mg TWP kg^{-1} as well as for trace metal and PAHs data. For mean difference analysis of Feret diameter (µm) we used a repeated measures ANOVA (rmANOVA) including all particles per distance. Linear regression models were set up to analyse the relationships between concentrations of PAH, BAP, Cd, Cu, Zn and Pb with TWP kg^{-1}. To assess contamination loads of trace metal and PAHs data, we used national threshold values for soil pollutants as reference [42]. Additionally, we calculated the pollution index (PI) and pollution load index (PLI) under application of geochemical background values with the following equations [43]:

$$PI = \frac{MC}{MGB} \quad (2)$$

Equation 2: with MC the median trace metal concentration (per sampling distance) and MGB the median geochemical background concentration [44].

$$PLI = \sqrt[n]{PI_{Cd} \times PI_{Cu} \times PI_{Zn} \times PI_{Pb}} \quad (3)$$

Equation 3: with n, the number of trace metals analysed and the single calculated PI values.

For Spearman correlation analyses and display of the results we used the "corrplot" [45] and "Hmisc" package [46]. We assessed the type of correlation and the correlation strengths between average number of TWP and the trace metal and PAHs concentrations per sampling site (n = 15), daily traffic volumes (DTV 2015), speed limits, the last street renovation year and data on asphalt topping grain size at each site. We interpreted statistical analysis results as significant with a p-value < 0.01.

Results

Tire wear particle abundance and properties

We found average concentrations of 111,000 TWP kg^{-1} (Sd: 147,000 TWP kg^{-1}) ranging from 1,190 to 615,000 TWP kg^{-1} in our selected roadside topsoils. The TWP concentrations decreased with increasing distance from the road (Fig. 2a) with a concentration ten times smaller at a distance of ten metres compared to one metre.

TWP were relatively small with an overall mean Feret diameter of 98.3 ± 78.2 µm ranging from 37.3 up to 4270 µm. Minimal Feret diameter was 62.8 ± 45.6 µm on average. The majority of TWP occurred with a Minimal Feret diameter between 35 and 50 µm (51.9%) or 50–100 µm (37.9%) (Fig. 3b). Larger particles account only for 8.5% (100–200 µm), 1.5% (200–500 µm) and 0.15% (> 500 µm) (Fig. 3b). Mean TWP circularity value was 0.7 ± 0.2 with the data majority between 0.5 (1st Quartile) up to 0.8 (3rd Quartile), resulting in partly rounded or elliptic particles (Fig. 3a). Average aspect ratio was 1.7, ranging between 1.5 and 2.1. With regard to distances from the road, applying rmANOVA, minimal Feret diameter showed a small but significant decrease from average 63.9 µm (1 m), over 61.1 µm (2 m) to 60.0 µm (5 m) distance (Fig. 2c). Cumulative frequency showed slight differences for particle circularity between 0.5 and 0.8 (Fig. 3a) but no significant mean differences over distance.

Estimated TWP masses averaged at 52.7 ± 83.2 mg TWP kg^{-1}, with a minimum of 0.02 mg TWP kg^{-1} and a maximum of 373 mg TWP kg^{-1}. Highest mass concentrations were found near the road (1 and 2 m distance) (Fig. 2b) and at site Seewen (1 m). Average TWP masses decrease from an average of 119 mg TWP kg^{-1} (1 m distance) over 60.0 mg TWP kg^{-1} (2 m distance) to 13.9 mg TWP kg^{-1} at 5 m distance, while increasing to 18.2 mg TWP kg^{-1} (10 m distance).

Organic compound and trace metal abundance

Both organic compounds as well as trace metals are present within studied roadside soils. We found average concentrations of 1,950 µg kg^{-1} for BAP, 18,400 µg kg^{-1} for PAH (16 EPA) and moderate Zn > Pb > Cu > Cd concentrations with highest concentrations of at comparable sites. In comparison to national threshold values for soil pollutants [42], we found that 63% of the samples exceeded the BAP limit (200 µg kg^{-1}) and 73% of the samples exceeded the PAH (16EPA) limit (1,000 µg kg^{-1}). For trace metals only 13% exceed Cd limit (0.8 mg kg^{-1}), 7% Zn limit (150 mg kg^{-1}) and 2% Cu limit (40 mg kg^{-1}). BAP and PAH showed the same concentrations at 1 and 2 m distance but decreased significantly at 5 and 10 m distance (Table 1). In contrast, the trace metal concentrations showed a uniform distribution over the distance gradient (Table 1). Pollution index (PI) according to

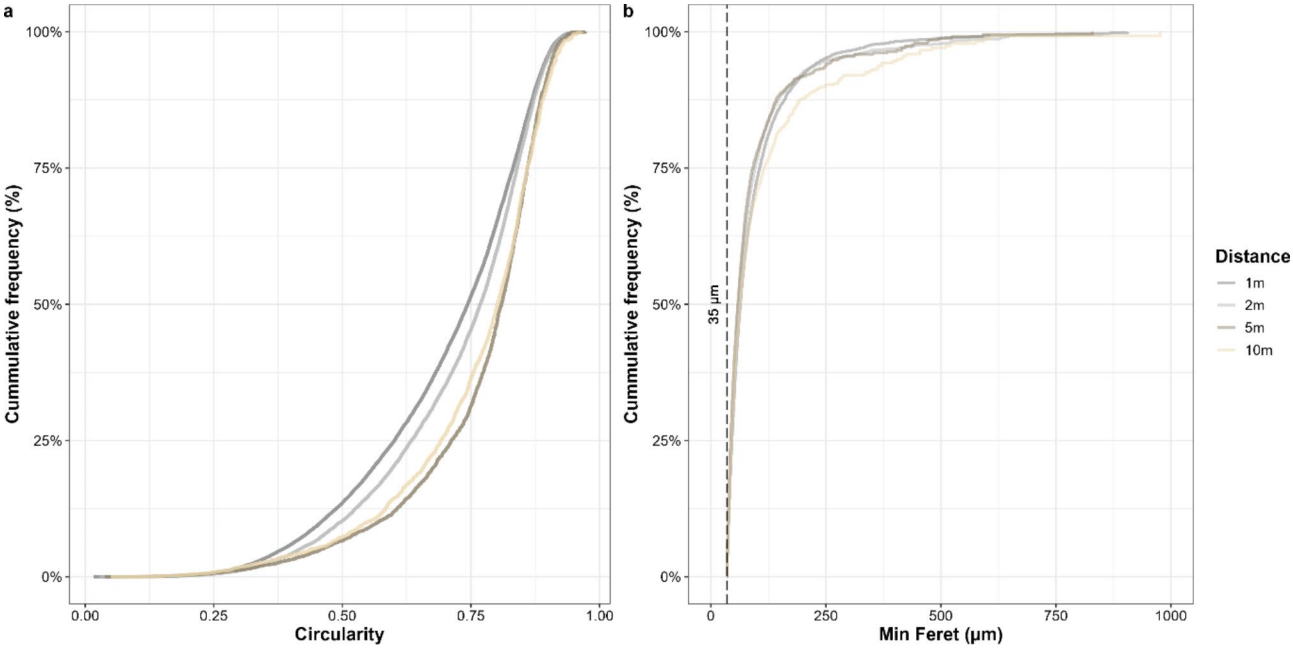

Fig. 3 Tire wear particle (TWP) features cumulative frequency at different distances. a: Cumulative frequency of TWP circularity at different distances (m) from road; b: Cumulative frequency of TWP Min Feret diameter (µm) at different distances (m) from road with lower particle size detection limit (35 µm, dashed line). Particle counts (n) 1 m = 19,873; 2 m = 8976; 5 m = 2,700; 10 m = 1921)

Table 1 Average Benzop[a]pyrene, polycyclic aromatic hydrocarbon (PAH) as sum of sixteen priority pollutant PAHs as defined by the Environmental Protection Agency (EPA) (16EPA) and trace metal concentrations along distance from roads. Significance levels: ns = not significant, * $p \leq 0.05$, ** $p \leq 0.01$, *** $p \leq 0.001$, **** $p \leq 0.001$

			Distance from road (m)			
			1	**2**	**5**	**10**
Benzo[a]pyrene		µg kg^{-1}	3667.1	2148.9	769.7	306.7
			ns	*ns*	*	***
PAH (16EPA)		µg kg^{-1}	35095.3	18864.3	7504.0	2508.8
			ns	ns	*	***
Average trace metal concen-tration	Cd	mg kg^{-1}	0.60	0.90	0.50	0.50
			ns	*ns*	*ns*	*ns*
	Cu	mg kg^{-1}	27.8	27.2	22.3	23.4
			ns	*ns*	*ns*	*ns*
	Zn	mg kg^{-1}	113.3	108.2	88.2	94.8
			ns	*ns*	*ns*	*ns*
	Pb	mg kg^{-1}	44.7	42.9	29.9	34.0
			*	*ns*	*ns*	*ns*

Posthoc test significance levels: ns: not significant; * $p \leq 0.05$; ** $p \leq 0.01$; *** $p \leq 0.001$

Kowalska et al. (2018) shows a low pollution for Cd, Cu, Zn and Pb compared to geochemical background across all distances, except a moderate pollution for Cd at 2 m distance. The pollution load index (PLI) was 2.80 (1 m), 3.32 (2 m), 1.49 (5 m) and 1.67 (10 m) and an overall deterioration of soil quality (PLI > 1).

Impact of road parameters on TWP and soil pollutant distribution

On sampling site level including the 15 different sampling sites and roads, we found that sites with a high TWP concentrations also showed the highest BAP, PAH and trace metal concentration. Both sites showed comparable road and traffic data (Table S1). In comparison between distances from road, we found a clear decline in TWP concentrations, both particle- and mass-based, corresponding to the decline of BAP and PAH concentrations with increasing distance (Fig. 3; Table 1). Linear regression models indicate that TWP concentrations are significantly affected by the distance from the road ($R = 0.27$, $p < 0.001$) with a TWP concentration decline −21,655 TWP kg^{-1} per distance interval. While TWP

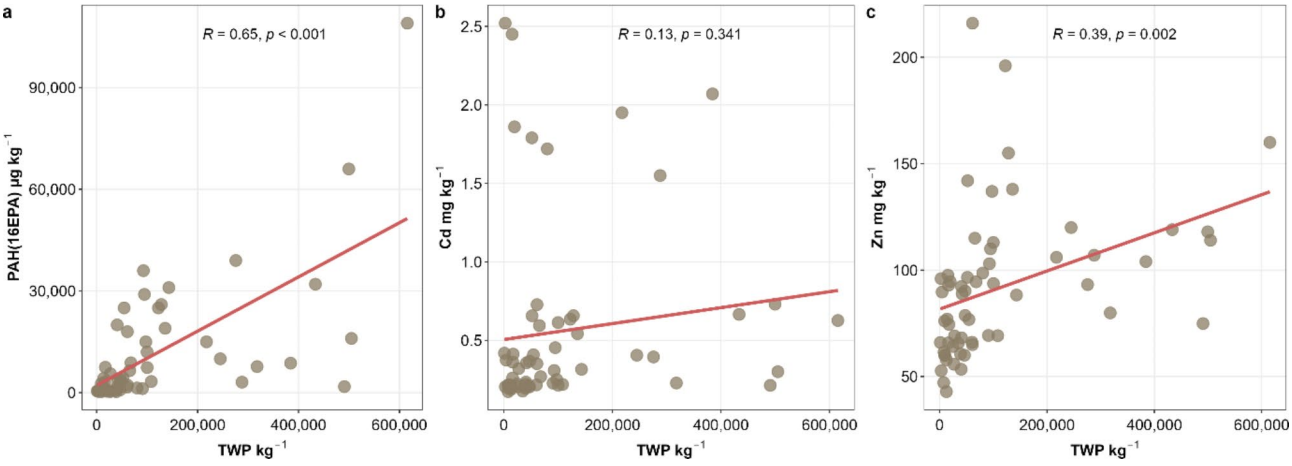

Fig. 4 Linear regression of soil pollutants and tire wear particle concentrations. **a**: Polycyclic aromatic hydrocarbon (PAH 16 EPA) concentration (µg kg^{-1}); **b**: Cadmium (Cd) concentration (mg kg^{-1}); **c**: Zinc (Zn) concentration (mg kg^{-1}); all combined with tire wear particle concentration (TWP kg^{-1}) ($n = 60$); Regression R and p values given in plots

mass concentrations were also influenced by the distance to the road, here the linear regression coefficient was weaker ($R = 0.16$, $p > 0.01$) with a decline of -9.37 mg TWP kg^{-1} per distance interval.

Considering the full ($n = 60$) dataset, we were able to show a strong positive correlation between TWP mass concentrations and both, BAP ($R = 0.70$) and PAH ($R = 0.65$) concentrations (Fig. 4a and Figure S3b). Similarly, positive linear correlations existed also between TWP mass and Cu, Zn and Pb concentrations, however, with weaker correlation strength (R 0.34–0.42) as compared to BAP and PAH. No correlation was detected between TWP mass and Cd concentrations. (Fig. 4, Figure S3).

Based on this result, Spearman correlation analysis (Figure S4) showed a strong and significant positive correlation ($R_S \geq 0.5$, $p \leq 0.01$) between PAH, BAP and trace metal concentrations. Furthermore, slight to moderate positive correlations ($R_S \geq 0.3$, $p \leq 0.01$) are reached between PAH, BAP, trace metals and TWP concentrations. In comparison to daily traffic volumes, we found moderate positive correlations ($R_S \geq 0.4$, $p \leq 0.01$) for BAP and PAH concentrations. For TWP concentrations and average Feret diameter moderate positive correlations ($R_S \geq 0.45$, $p \leq 0.01$) was found with speed limit data, while correlations with daily traffic volumes or road data, such as asphalt topping grain size or renovation year stayed weak.

Discussion

Data quality and limitations

Within the last years of ongoing microplastic research, several studies emphasise the importance of quality assurance and quality control to achieve valuable and comparable environmental data [47, 48]. Especially the importance of contamination prevention, blank control and recovery testing was highlighted [47]. Those measures are not only necessary, when studying the polymeric part of microplastics, but also for tire and other road wear-related studies. It can be assumed that TWP or other "black particles" such as rubber are clearly less present in laboratory environments, than classic polymers (e.g., PE, PP) used for lab instruments or chemical storage [48]. Therefore, we conclude that despite intensive cleaning of lab wear, particle carryover could explain the low level of black particle contamination. Performance of the applied extraction method and image analysis is discussed within the original method publication [33]. As with all methods including mass-based analytics, extraction from or interferences with organic matter in soil samples appears to remain the major challenge to method performance [5]. We controlled the reproducibility of our extraction and analysis via replicate samples [47, 49] and found it to be very good (RSD ≈ 3%). LDIR analysis performed for randomly selected particles from six samples shows, that only rubber particles, representing TWP are present on the filters.

One major limitation of the present study is the application of NaBr solution ($\rho = 1.5$ g cm^{-3}) for density separation, which was chosen as part of the method development [33]. In other studies using particle-based TWP analytical approaches, saturated sodium iodide (NaI, $\rho = 1.80–1.85$ g cm^{-3}) density solutions were used for TWP extraction from snow [30] or stormwater and sweepsand [50, 51]. With the applied density of 1.5 g cm^{-3} we clearly cover the known densities of tire rubber and tire particles (1.1–1.2 g cm^{-3}) as well as bitumen (0.9–1.1 g cm^{-3}), but not road wear particles ranging between 1.5 and 2.2 g cm^{-3} or minerals with quartz densities of 2.65 g cm^{-3} [39, 40]. Therefore, an underestimation of the road wear share of TWP accounting for approx. 50% of TWP mass has to be assumed within our

study. Consequently, we used the maximum density of tire rubber and tire particles (1.2 g cm^{-3}), without the consideration of road wear, within our mass estimation model.

Despite the benefits of particle-based TWP analysis like measuring real particle counts and the provision of particle information such as particle size distribution and morphology, which is missing for roadside soils so far [22], the chosen method is subject to limited comparability. This is due to the fact, that all available studies on TWP presence in roadside soils used mass-based (pyr-GC/MS, TED-GC/MS) methods, different extraction methods and case study sites with overall higher traffic densities [25–27]. Estimated TWP masses allow an indirect comparison, but remain a feeble estimate, because of the assumed particle density, which can vary widely, depending on road-associated particles within TWP [4]. Particle-based methods currently only allow direct comparisons with other environmental matrices, whereby there are also differences in the extraction methods.

Tire wear particle concentrations and particle features

The average TWP concentration found in roadside soils investigated in this study was significantly higher than in methodologically comparable studies using particle-based analytical approaches. The small number of studies documenting particle-based results within other environmental matrices found average TWP concentrations of e.g., 57,461 TWP kg^{-1} in urban storm retention reservoir sediments (Brazil) [31], 16,665 TWP kg^{-1} (max: 66,666 TWP kg^{-1}) in urban road dust (Iran) [52], or 1,833 TWP kg^{-1} in tributary sediments (USA) [53]. More comparable results from urban roadside snow or road sweepsand and stormwater, show TWP concentrations around 9,500 TWP L^{-1} (max: 197,000 TWP L^{-1}) in roadside snow (Sweden) [30] and 5,700 TWP L^{-1} in road stormwater, 42,320 TWP L^{-1} in road washwater or up to 2 Mio. particles per kg^{-1} in sweepsand (Sweden) [50, 51].

Our estimated TWP masses are generally lower when compared to TWP concentration estimated from mass-based methods. For example, Rødland et al. (2022) document average TWP concentrations of 11,400 mg TWP kg^{-1} for low traffic roads in Norway, while Müller et al. (2022) document average TWP concentration of 4,835 mg TWP kg^{-1} for medium traffic roads in Germany. However, TWP masses in the investigated Swiss cantonal road soils are more comparable to concentrations in freshwater sediments of e.g., 46–420 mg TWP kg^{-1} (Chesapeake River, USA) or 28–580 mg TWP kg^{-1} (Jodo River, Japan) [54], but stay well below concentrations in street sedimentation reservoirs, tunnel dust or street dust [16]. The higher particle-based concentrations, compared to other studies, could be explained on the one side, by an automated image analysis-based counting of particles, which delivers more reliable data compared to visual inspections [55]. On the other side, the first-time analysis of roadside soils using the particle-based method alone could explain the higher contents, as no direct comparison to other studies with comparable methods is possible. The particle-based data would therefore correspond with the high mass contents from other roadside soil studies [25, 27]. Vice versa, the significantly lower mass concentrations can be explained by limitations of the mass estimation. Here, the assumed density of $\rho = 1.2$ g cm^{-3}, covers tire tread densities (1.13–1.16 g cm^{-3}) and lower tire and road wear associated particles [4], but not the full range of densities reported for tire, associated road wear and resulting hetero-aggregates (1.5–2.2 g cm^{-3}) [40]. Furthermore, significant lower traffic densities in the present studies (av. 2293 vehicles day^{-1}) compared to recent roadside soil studies (e.g., 650–14,250 vehicles day^{-1}) [25]combined with the presence of drainage systems at larger streets (e.g., federal highways), excluded in the present study, could explain the lower mass concentrations in Cantonal roadside soils.

Regarding TWP characteristics, we found a TWP size distribution and morphology comparable to those of other particle-based studies. Overall, the number of TWPs appears to increase with decreasing particle size, independent of the environmental matrices investigated [16]. The same trend was frequently found for "classical" microplastics in soil [56]. Based on the circular shape and aspect ratio of the TWPs found in the current study, the particles were characterized by an elongated shape. This typical morphology of TWP is mainly caused by abrasion during driving and by the bonding with road surface material to form hetero-aggregates [6, 7].

Organic pollutants and trace metals

BAP and PAH (16EPA) concentrations analysed, exceed national threshold values for good soil quality within 63% and 73% of the samples as well as background concentrations from long-term soil monitoring (NABO) in Switzerland (average: 32 µg kg^{-1} for BAP and 338 µg kg^{-1} for PAC) and land-use specific backgrounds [57]. Comparing our data to more urban areas and roads with higher (> 25,000 DTV) traffic volumes, our PAH data still exceeds average values from high-traffic roadside soils in South Korea (1,079 µg kg^{-1}) [15] or average values from Stockholm urban soils (Sweden, 4,836 µg kg^{-1}) [58], but are below the maximum concentration reported within these studies. In contrast, trace metal concentrations including Cd, Cu and Zn exceed national threshold values only at 13%, 2% and 7% of samples. For Pb all concentrations were below the threshold values for good soil quality. Median concentrations in our Swiss cantonal roadside soils are below European wide roadside soil concentrations with medians of 0.73 mg kg^{-1} (Cd), 47.9 mg

kg^{-1} (Cu), 179.5 mg kg^{-1} (Zn) and 106.0 mg kg^{-1} (Pb) [13]. However, the results are clearly within the range of trace metal concentrations from the 27 reviewed studies across Europe by Werkenthin et al. (2014). Calculation of single pollution index (PI) per trace metal indicates a low to moderate soil pollution, based on geochemical background values. Based on PI, Cd and Pb show the highest accumulation at narrow distances to the roads (1 and 2 m). Using pollution load index (PLI) the presence of considered trace metals in roadside soils can be interpreted as a clear accumulation [43].

Regarding spatial distribution along distance gradients from roads, we found decreasing concentrations of organic compounds with increasing distances. BAP and PAH concentrations are comparable at short distances (1 m and 2 m), but showed significant declines at further distances (5 m and 10 m). This finding is comparable to decreasing PAH concentrations from tar asphalt with increasing distance (> 2 m) [59]. In contrast, trace metal concentrations stay equally distributed over the full 10 m range. This is contrary to trace metal distribution in European roadside soils, where concentrations decrease at > 5 m distances [13]. It is likely, that the spatial distribution along distance gradients displays the different emission sources and transport processes of organic compounds and trace metals. While both, organic compounds and trace metals are included within TWP, further sources like an exhaust gas, asphalt, road signs or brake pads occur in road environments [40, 60]. Additionally, most roadside soils show already higher concentrations of Cu and Zn and are influenced by soil transfer processes during road constructions within narrow distances [61]. Those narrow distances are influenced mainly by runoff- and splash water transport combined with airborne transport of pollutants, whereas for further distances only airborne transport remains [13].

Relationships

Comparing street-related pollutants, a clear positive linear relationship was found between TWP concentrations and the non-polar organic compounds BAP and PAH. Weaker linear relationships between TWP concentrations and Cu, Zn and Pb concentrations were also present. Similar patterns have been found for Spearman correlation, with moderate correlations of BAP, PAH and Zn with TWP concentration. In contrast, previous studies investigating both, TWP and trace metals in roadside soils, found no strong relationships between trace metals and Styrene Butadiene rubber (SBR) used as TWP marker [25]. However, the co-occurring and relationships of TWP, non-polar organic compounds and trace metals in roadside soils can clearly be traced back to the composition of tires itself, associated road-wear particles and other traffic emissions [12]. While BAP and PAH are known compounds in tire material which can be leached out from TWP [9], they are furthermore emitted to road environments from petrol-based vehicle emission [15, 58, 62]. In the case of trace metals, large portions of Zn are used in tires as part of the filler mixture [12] or emitted from brake pads [11], also leading to first attempts of using Zn as a TWP marker [39]. Further trace metals, like Cd, Cu and Pb among others, are also associated with TWP and used in tire material or present in brakes, catalysts, road asphalt and petrol-based emissions contribute as substances in the hetero-aggregates of the TWP, but are also emitted directly into roadside soils [9, 27].

The decreasing concentration of TWP and organic compounds with increasing distance from road is comparable to most other studies with a comparable sampling design. Exemplary, comparable results were found by Müller et al. (2022) as a decline of SBR and Zn concentrations from 0.3 to 5 m distance in roadside soils or by Vijayan et al. (2022) for roadside snowbanks between 0.5 and 2.5 m distances. Contrary, Rødland et al. (2023) found no significant difference in SBR concentrations in roadside soils along a gradient from 0 to 6 m distance. For trace metals, most studies found a concentration decrease with increasing distance [13]. The missing decrease over distance for Swiss cantonal roadside soils, could therefore be explained by the variance in emission sources and transport, as trace metals are suspected to reach roadside areas up to 20 m distance [63].

TWP sizes and morphology are decreasing slightly from 1 to 5 m distance, while morphology features (e.g., circularity) stay equally distributed. Previous studies indicate that smaller particles are more easily transported over longer distances by air or water and larger particles (> 500 µm) are directly trapped close to roads [10, 31]. Smaller TWPs could also easily be detached from the road surface [10]. However, with regard to the studied roadside soils with grassland vegetation, factors like surface roughness, vegetation, water flow direction and wind directions could influence size distribution.

Previous studies indicating daily traffic volumes or traffic data such as speed limits as major driving factors for TWP and other traffic-related pollutant emissions independent of the environmental matrix studied [15, 25, 26, 31, 51]. While most studies found a strong relationship between increasing DTV values and increasing TWP abundance, we only found a moderate positive correlation for TWP concentrations with speed limit data, indicating higher TWP emissions with increasing traffic speed as also found by Rødland et al. (2022). A possible explanation for the missing relationship at Swiss cantonal roadside soils, could be the considerably lower traffic density and resulting smaller DTV variation in comparison to all other previous studies. Moderate positive correlations were found between DTV and BAP as well as

PAH concentrations, indicating a higher petrol-based emission of organic compounds than from TWP [15, 51]. The role of other traffic and street-related data, such as road age or asphalt type along with traffic flow (e.g., braking zones at intersections) have been highlighted as further influencing factors [25, 30]. For Swiss cantonal roadside soils, such relationships were not found. Therefore, it could be concluded that TWP emissions occur independent from road age or asphalt roughness and may be more controlled by traffic speed, traffic volumes and here not covered small scale variations such as traffic behaviour (e.g., brake points).

Conclusion

The results of our study demonstrated the role of roadside soils as environmental sinks for TWPs and associated organic compounds and trace metals. Even in low-traffic areas like the Swiss cantonal road net studied, high levels of TWP and especially organic compounds occur. Using a particle-based analytical protocol for the first time, we were able to demonstrate that TWP were abundant in the selected roadside soils with comparable concentrations to other road-related environmental matrices like roadside snow or stormwater. Moreover, our protocol allowed to describe particle size and morphology. For spatial distribution and the spread of TWP to soils, distance from the road and therefore transport processes and particle sizes are key control factors. The strong relationship between TWP numbers and organic compounds underlines the linkage of TWP as heteroaggregates with associated organic compounds and other traffic-related pollutant sources. The missing influence of traffic volume on TWP abundance suggests that other driving and street factors such as speed limits or small-scale traffic variations could have higher influence on TWP abundance along low traffic roads. Future research should combine marker and mass-based TWP analysis methods with particle-based methods to generate integrated TWP data sets. Especially for research on TWP transport in soil, size and morphological data are mandatory. The clear link between TWP and other pollutants emphasizes the need for combined studies that look at complex groups of pollutants rather than individual ones. Finally, the consideration of small-scale traffic variations and trapping factors such as vegetation, will help to contribute to a better understanding of TWP transport behaviour and facilitate future mitigation measures.

Abbreviations

BAP Benzop[a]pyrene
DTV Daily traffic volumes
LDIR Laser direct infrared spectroscopy
Cd Cadmium
Cu Copper
PAH Polycyclic aromatic hydrocarbons
Pb Lead
TWP Tire wear particles
Zn Zinc

Supplementary Information

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Supplementary Material 1

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Author contributions

Conceptualization: AF, DK, MB; Methodology: AW, MB; Validation: CJW; Formal analysis: AW, CJW; Investigation: AF, DK, CJW; Resources: AF, DK; Data curation: AF, DK; Writing– Original Draft: CJW; Writing– Review & Editing: CJW; DK, AW, MB; Visualization: DK, AW, CJW; Supervision: AF, DK, EB, MB; Project administration: AF, DK, EB, MB; Funding acquisition: AF, DK, MB.

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Data availability

Data on sampling sites including traffic related data is available in Table S1 (Supplementary material). The datasets generated and analysed during the current study are available in the FigShare repository: Weber, C.J.; Kundel, D. & A. Wiget (2024): Dataset of Tracks of Travel: Unveiling Tire Particle Concentrations in Swiss Cantonal Road Soils. FigShare. DOI: 10.6084/m9.figshare.2712449.

Declarations

Ethics approval and consent to participate

'Not applicable'.

Consent for publication

'Not applicable'.

Competing interests

The authors declare no competing interests.

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