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High levels of tire wear particles in soils along low traffic roads

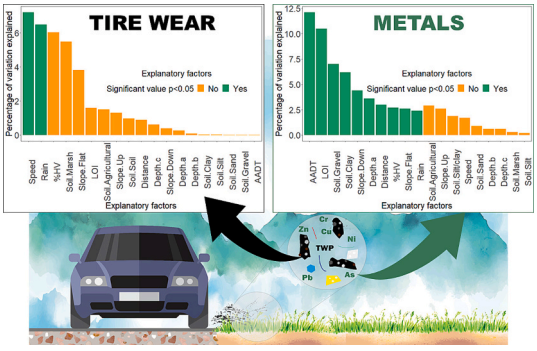
Elisabeth S. Rødland [a,*], Lene S. Heier [b], Ole Christian Lind [c], Sondre Meland [a]

[a] Norwegian Institute for Water Research, Økernveien 94, 0579 Oslo, Norway
[b] Norwegian Public Roads Administration, Construction, Postbox 1010, N-2605 Lillehammer, Norway
[c] Norwegian University of Life Sciences, Faculty of Environmental Sciences and Natural Resource Management, P.O. Box 5003, NO-1432 Ås, Norway

HIGHLIGHTS

- High levels of tire wear particles (0.2–2.6 % d.w.) in soils along low traffic roads
- Positive correlation between tire wear particles, traffic speed and precipitation
- Positive correlation between metals, traffic density and organic matter
- No correlation between tire wear particles and metals
- No vertical or horizontal gradient found for tire wear particles or metals

GRAPHICAL ABSTRACT



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ABSTRACT

Traffic pollution has been linked to high levels of metals and organic contaminants in road-side soils, largely due to abrasion of tires, brake pads and the road surface. Although several studies have demonstrated correlations between different pollutants and various traffic variables, they mainly focused on roads with medium to high traffic density (>30,000 vehicles per day). In this study we have focused on investigating tire wear particles and road-related metals (zinc, copper, lead, chromium, nickel, and the metalloid arsenic) in the soils of low traffic roads in rural areas (650–14,250 vehicles per day). Different explanatory factors were investigated, such as traffic density, speed, % heavy vehicles, organic matter content, annual precipitation, soil types and roadside slope profiles. The results show high levels of tire wear particles, from 2000 to 26,400 mg/kg (0.2–2.6 % tire wear in d.w. soil), which is up to five times higher compared to previously reported values in roadside soils of high traffic density areas. A weak but significant correlation was found between tire wear particles, traffic speed and the annual precipitation. No significant relationship was found between tire wear particles metals. The concentrations of metals were comparable to previous studies of high traffic areas of Norway, as well as both urban and rural soils in other countries. For the metals, all factors together explained 45 % of the variation observed, with traffic density (11 %) and organic matter content (10 %) as the most important single variables. The analysis of tire wear particles in soils using Pyrolysis Gas chromatography Mass Spectrometry is challenging, and the results presented demonstrate the need for pretreatment to remove organic matter from the samples before analysis.

* Crresponding author.
E-mail address: elisabeth.rodland@niva.no (E.S. Rødland).

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1. Introduction

Traffic pollution contributes to elevated levels of metals in roadside soils (Sager, 2020; Turer et al., 2001; Werkenthin et al., 2014; Zehetner et al., 2009). High levels of metals have also been reported in road dust (Hwang et al., 2016), road runoff (Huber et al., 2016), tunnel wash water (Meland and Rødland, 2018) and roadside snow (Müller et al., 2022b).

The biggest sources of metals and other road-related pollutants are abrasion of tires, brake pads and the road surface (Wagner et al., 2018). Tires, as the main source of the three, have been reported to contain up to 1.1 % Zn for passenger vehicles and 2.4 % zinc (Zn) for heavy vehicles (Smolders and Degryse, 2002), whereas the total mass of Zn found in tire wear particles (TWP) from the road is lower (0.3–0.4 %) (Kreider et al., 2010). Both sulphur (S) (vulcanization agent) and zinc oxide (ZnO) (catalyst) are added to tires during the production process, along with silica (SiO_2) (filler) and sometimes calcium carbonite ($CaCO_3$) (filler) (Rausch et al., 2022). For TWP, both from laboratory studies and field studies, a large range of elements are found, such as iron (Fe), calcium (Ca), titanium (Ti), barium (Ba), antimony (Sb), aluminum (Al), silisium (Si) (Gustafsson et al., 2008; Kupiainen et al., 2005; Kwak et al., 2013; Panko et al., 2009), however, these are mainly related to the interaction between tire tread and the road surface both during driving and after the TWP has been released to the road surface (Rausch et al., 2022).

Tire and road wear particles have in recent years also been recognized as one of the largest sources of microplastic particles to the environment (Boucher et al., 2020; Knight et al., 2020; Sundt et al., 2021). So far, only two studies have reported mass concentrations of microplastic particles from tire wear in roadside soils; 3700–4900 mg/kg in Norway (E6 highway at Skullerud, Oslo) with annual average daily traffic (AADT) at 77000 v/d (Rødland et al., 2022c) and155–15,900 mg/kg in Germany ((Bundesstraβe B27 near Tübingen, AADT 36000v/d (Müller et al., 2022a), both studies also indicating a decrease in TWP concentrations with distance from the road. However, the knowledge of TWP in soils and the potential impact on the environment is limited, and no current studies have investigated the levels of TWP in soils along roads with low traffic.

Studies have shown that runoff water is the main transportation route of road pollutants within 2 m distance from the road, whereas splash and spray dominates the distances between 2 and 10 m from the road (Sager, 2020). At distances >10 m, the pollutants are mainly transported by air (Sager, 2020). Traffic derived pollution in roadside soils has been assumed to be positively correlated with the annual average daily traffic (AADT) (Francek, 1992; Werkenthin et al., 2014), where especially metals such as Zn and Pb showed increasing levels with increasing AADT (Kadi, 2009; Wang and Qin, 2007). Studies have shown that levels of road-related metals decrease with depth and distance from the road along with a gradient of accumulation in the soil, suggesting that it takes time before such relationships can be detected (Carrero et al., 2013; Dierkes and Geiger, 1999). Similar patterns for various pollutants have been found for road dust, roadside snow, tunnel wash water and road runoff treatment ponds (Grung et al., 2022; Gunawardena et al., 2015; Li et al., 2014; Meland and Rødland, 2018; Moghadas et al., 2015; Viklander, 1999), although recent studies on microplastic particles in roadside snow looking at both high and low traffic density found no correlation with AADT (Vijayan et al., 2022). Our group recently reported that traffic speed, rather than AADT correlated with accumulation of tire wear particles in roadside snow (Rødland et al., 2022b).

Retention of road pollution in soils is influenced by soil pH and soil type (Sager, 2020), with high clay content being positively correlated with retention of both organic and inorganic pollutants (Koutsopoulou et al., 2010; Kowalska et al., 1994). The level of organic matter in soils have also been linked to retention of metals (Turer et al., 2001; Turer and Maynard, 2003). Also, the physical characteristics of the road verges such as depth and profiles have been reported to impact the retention, in which road verges with upwards facing profiles potentially acts as traps for the runoff and for retaining road pollutants.

The overall levels of road-related contaminants, including metals, polycyclic aromatic hydrocarbons (PAHs), polychlorinated bisphenyls (PCBs), oil and road salt (NaCl) for 16 different low-traffic roads in the county of Trøndelag, Norway were reported in 2018 (Rabben, 2018). This study found no impact on the level of pollution from the traffic density, the type of soil or the vegetation on the roadsides, although the authors indicated that marshland could have a higher retention of contaminants compared to other soil types. The report also indicated that distance from the road could have an impact on the levels of contaminants. Overall, the concentration of contaminants reported where considered low compared to classifications of polluted soils (NEA, 2010) with levels of PAHs being classified as low (<2 mg/kg) in all samples and not detected in 22 % of samples, whereas PCBs were not detected in any of the soil samples. The levels of metals were also classified as low for all metals, with a few exceptions for arsenic (As, 33 mg/kg), chromium (Cr, 106 mg/kg) and zinc (Zn, 272 mg/kg), all at 0 m distance from the road.

As previously mentioned, the levels of TWP in soils have not yet been investigated along low traffic roads, nor has it been thoroughly discussed in relation to metals and different traffic and soil variables. In the present study, we re-analyzed soil samples from the above-mentioned study in the county of Trøndelag (Norway) for TWP and utilized the dataset of metal analysis to investigate the relationship between TWP, metals, traffic variables and soil variables using multivariate statistical tools. We hypothesize that 1) the levels of TWP are lower along low-traffic rural roads compared to high-traffic urban roads from previous studies and 2) the TWP and metal levels in roadside soils are strongly correlated and 3) each contaminant (TWPs and metals), the traffic variables and the soil variables are strongly correlated.

2. Methods and materials

2.1. Sampling

Road-side soils were collected in 2013 and 2018 from 16 different sites in Trøndelag county, Norway, along low traffic (AADT 650–14,250) roads (Fig. 1, Table 1). Images of all sampling sites are presented in the Supplementary Information (Figs. S1-S16). All the roads included in the study are rural roads with vegetation on both sides. Some roads had pedestrian paths on one side of the road, this side was not sampled. The age of the roads could not be obtained for this study, but all roads were constructed before 2010 according to images provides by Google maps. The annual precipitation for each site (based on the closest weather station (Seklima., 2023) varied between 810 mm per year at the Støren site and up to 1485 mm precipitation at the Vennastrand sites (Table 1, Table S1). The annual precipitation was calculated as an average of minimum three years between 2014 and 2022, depending on the available data. All samples were collected as mixed samples, comprised of five samples taken 20-40 m apart on a 100-200 m road stretch and mixed together in Rilsan bags. Samples were collected using a small shovel. The upper layer of the soil (a: 0.0–0.1 m) was collected at all sites except Vikhammerdalen and Bjørndalen (Table S1). The lower layer of soil (b: 0.1–0.2 m, c: 0.1–0.3 m) wascollected at all sites except Bratsberg, Levanger and Mosløkkja. At most sites, samples were collected at 0 m, 3 m and 5 m from the road. At some sites this was not possible and for these, samples at 6 m and 8 m distance were included. The sites exhibit large variations in terms of soil properties and in the present work, the sites were grouped according to 8 soil types; agricultural production soil (agriculture), topsoil (soil) marshland (marsh), clay, silt, mixture of silt/clay, gravel, sand). There are also variations among the sites and the collected samples in terms of the profile of the roadside (flat, up, down), the organic content in each soil sample, annual average daily traffic (AADT), traffic speed limits and percentage of heavy vehicles (Table 1).

ABS

2.2. Sample preparation

Soil samples were oven dried (40 °C) inside Rilsan bags to remove all moisture. The samples were sieved with 500 μm sieves using an automated sediment shaker and placed in small glass jars (pre-treated in muffle furnace). Samples were then mixed using a small metal spoon before multiple aliquots from several places within the jar were taken out and placed in the pyrolysis cup and weighed. The amount of sample analyzed was 11.5 ± 2.7 mg (d.w.).

To investigate the need for removal of organic matter, 3 samples with various organic matter content determined by loss of ignition (LOI) and various distance from the road (0 m, 5 m, 8 m) were chosen and analyzed with no treatment and with treatment for comparison. Sample aliquots of 30 mg (dried and sieved as described above) were weighed into Falcon tubes (VWR), 1 mL of hydrogen peroxide (H_2O_2) were added to each tube and they were kept in an incubator for 24H at 40 °C. The samples were then filtered onto pre-weighed glass fiber filters (GF/A, 15 mm, 1.6 μm pore size), dried at room temperature and weighed before inserted into pyrolysis cups for analysis.

2.3. Analysis of road metals

Sample aliquots were subjected to metal analysis at a commercial laboratory (ALS Laboratory) following ISO 11885, EPA 200.7, EPA 6010, SM 3120 standard analytical methods for metal analysis using acid digestion and atomic absorption spectrometry (AAS). The metals reported were zinc (Zn), copper (Cu), lead (Pb), arsenic (As), chromium (Cr) and nickel (Ni) and the concentrations levels used for this study has been previously published in Rabben (2018).

Enrichment factors (EFs) are calculated as the level above or below the norm level (background level) of the pollutant, e.g. metal/metalloid, according to the methods described by Varol (2011):

$$EF = \frac{[M\ sample]}{[M\ norm\ value]}$$

Where the concentration level of metals/metalloids (M) in the sample is divided against the concentration level of M reported as norm value. The norm levels of metals in soils from the Trøndelag region has previously been reported in Andersson et al. (2011). Input of anthropogenic sources would cause an enrichment of the metals in soil, where the EFs would be >1. EFs from 1 to 3 demonstrates minor enrichment, EF 3–5 moderate enrichment, EF 5–10 moderate-severe enrichment, EF 10–25 severe enrichment, EF 25–50 very severe enrichment and EF > 50 extremely severe enrichment. If the EFs are <1, there are no enrichment of this metal in the soil and thus no indication of anthropogenic sources being present.

2.4. Analysis of tire wear particles

2.4.1. Pyrolysis gas chromatography mass spectrometry

Samples were analyzed with a Multi-Shot Pyrolyzer (EGA/PY-3030D) equipped with an Auto-Shot Sampler (AS-1020E) (Frontier lab Ltd., Fukushima, Japan) coupled to a gas chromatography mass spectrometer (GC/MS) (5977B MSD with 8860 GC, Agilent Technologies Inc., CA, USA), following the method of Rødland et al. (2022c). Samples were pyrolyzed in single-shot mode at 700 °C for 12 s. Injections were made using a 50:1 split and with a pyrolyzer interface temperature at 300 °C. The selected markers for Styrene Butadiene rubber (SBR) and Butadiene Rubber (BR) consisted of *m/z* 78 Da for benzene, m/z 118 Da for α-methylstyrene, m/z 117 Da for ethylstyrene and m/z 91 Da for

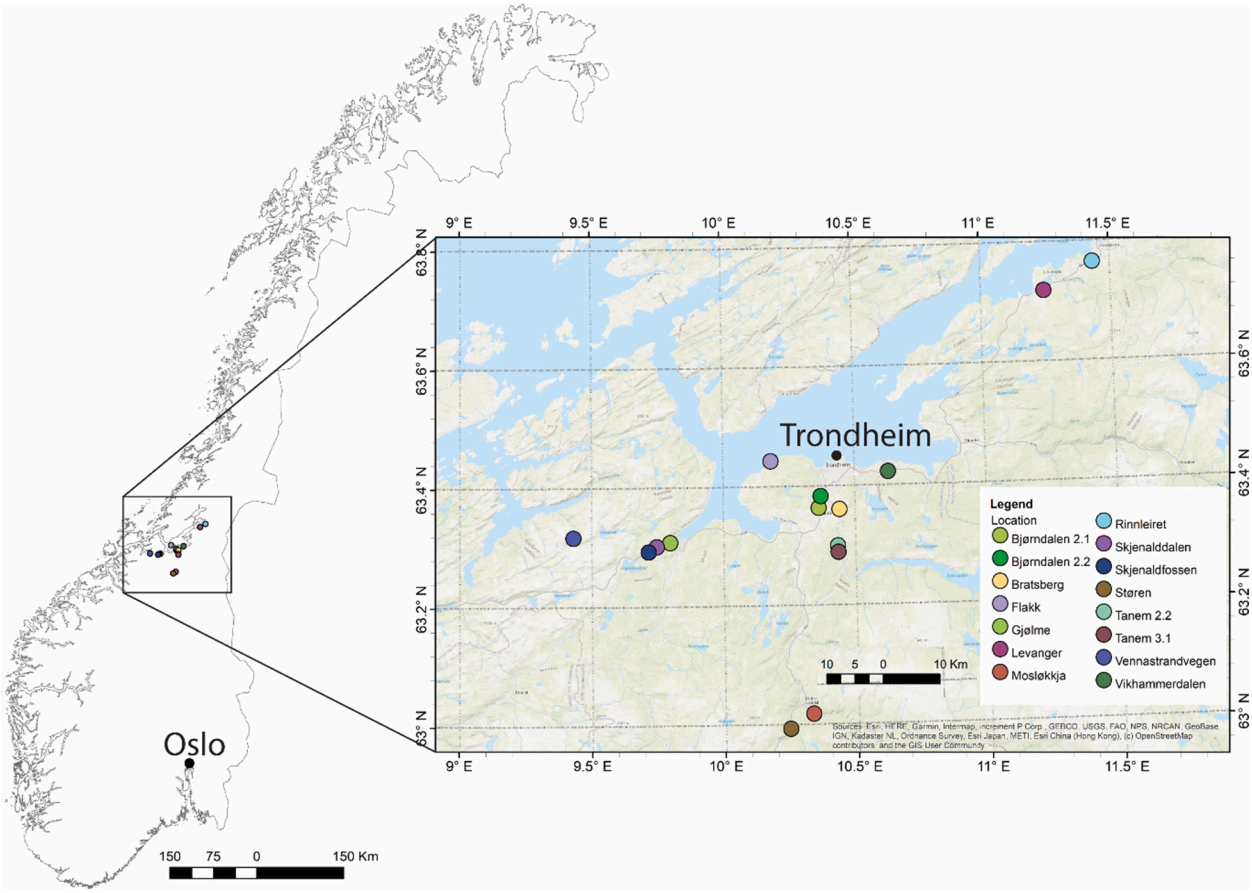

Fig. 1. Map of Norway (to the left) and a map of the sample sites of this study, all in the county of Trøndelag. Locations for Vennastrandvegen 1.1 and 1.2 is shown as one site on the map due to short distance between the sites for the resolution of the map. See more details in Supplementary Fig. S14 and S15.

butadiene trimer. The total mass of SBR and BR is calculated based on the combined peak heights of the four markers normalized against an internal standard (deuterated Polybutadiene, d6- PB).

2.4.2. Calibration, limit of detection and limit of quantification

The calibration curve was established by adding 1 μg, 2 μg, 5 μg, 25 μg, 50 μg and 100 μg of SBR (SBR1500 standard, Polymer Source) into separate pyrolysis cups and spiked with 25 μg d6-PB as internal standard. The normalized sum peak of all marker compounds is plotted against the mass of SBR at each calibration level to form the calibration curve ($R = 0.995$) (Fig. SI-1). The signal to noise ratio (S/N) is determined by the Agilent Masshunter software for each of the selected

Table 1
Overview of the traffic parameters (Annual Average Daily Traffic (AADT), percentage heavy vehicles (%HV) and driving speed), soil parameters (soil type, road-side profile/slope, organic matter as percentage loss of ignition (LOI)), distance from the road (m), soil layer depth (a: 0–0.1 m, b: 0.1–0.2 m, c: 0.1–0.3 m) and annual precipitation (mm) for all sites.

Site	Distance	Depth	Soil	Slope	LOI	AADT	%HV	Speed	Annual precipitation
Bjorndalen_2_1	0	a	Gravel	Up	4.12	5800	10	60	827
Bjorndalen_2_1	3	a	Clay	Up	11.2	5800	10	60	827
Bjorndalen_2_1	3	c	Gravel	Up	8.55	7400	12	60	827
Bjorndalen_2_1	5	a	Clay	Up	8.73	5800	10	60	827
Bjorndalen_2_2	0	b	Gravel	Up	7.06	5800	10	60	827
Bjorndalen_2_2	5	c	Clay	Up	5.89	7400	12	60	827
Bratsberg_2_2	0	a	Gravel	Down	2.53	3500	12	60	1074
Bratsberg_2_2	3	a	Clay	Down	9.62	3500	12	60	1074
Bratsberg_2_2	5	a	Clay	Down	2.53	3500	12	60	1074
Flakk_2_1	0	b	Sand	Down	6.31	1600	6	60	1079
Flakk_2_1	3	c	Soil	Down	9.97	1600	6	60	1079
Flakk_2_1	5	a	Soil	Down	11.1	1600	6	60	1079
Flakk_2_1	5	c	Soil	Down	13.2	1600	6	60	1079
Gjolme_2_1	0	a	Sand	Up	3.32	4200	21	80	834
Gjolme_2_1	0	b	Sand	Up	7.38	4200	21	80	834
Gjolme_2_1	3	a	Clay	Up	8.14	4200	21	80	834
Gjolme_2_1	5	a	Clay	Up	6.51	4200	21	80	834
Gjolme_2_1	5	c	Clay	Up	8.71	4200	21	80	834
Levanger_1_2	0	a	Gravel	Down	5.25	12,500	16	70	873
Levanger_1_2	3	a	Agricultural	Down	8.31	12,500	16	70	873
Levanger_1_2	8	a	Agricultural	Down	NA	12,500	16	70	873
Moslokkja_1_2	0	a	Gravel	Down	5.73	2400	12	70	874
Moslokkja_1_2	3	a	Silt	Down	4.81	2400	12	70	874
Rinnleiret_3_1	0	a	Sand	Flat	4.12	14,250	12	70	1074
Rinnleiret_3_1	0	b	Sand	Flat	4.12	14,250	12	70	1074
Rinnleiret_3_1	3	a	Soil	Flat	24.6	14,250	12	70	1074
Rinnleiret_3_1	3	c	Soil	Flat	24.6	14,250	12	70	1074
Rinnleiret_3_1	6	a	Marsh	Flat	16.7	14,250	12	70	1074
Rinnleiret_3_1	6	c	Marsh	Flat	16.7	14,250	12	70	1074
Skjenalddalen_1_1	0	a	Gravel	Up	3.18	4200	21	80	834
Skjenalddalen_1_1	0	b	Sand	Up	5.49	4200	21	80	834
Skjenalddalen_1_1	3	a	Soil	Up	13.2	4200	21	80	834
Skjenalddalen_1_1	3	c	Sand	Up	8.31	4200	21	80	834
Skjenalddalen_1_1	5	a	Soil	Up	14.4	4200	21	80	834
Skjenalddalen_1_1	5	c	Sand	Up	8.14	4200	21	80	834
Skjenalddalen_3_1	0	a	Sand	Up	2.26	4200	21	80	834
Skjenalddalen_3_1	0	b	Sand	Up	3.65	4200	21	80	834
Skjenalddalen_3_1	3	a	Soil	Up	23.1	4200	21	80	834
Skjenalddalen_3_1	3	c	Soil	Up	21	4200	21	80	834
Skjenalddalen_3_1	5	a	Marsh	Up	66.4	4200	21	80	834
Storen_1_1	0	a	Gravel	Down	2.65	6160	23	80	810
Storen_1_1	3	a	Soil	Down	8.35	6160	23	80	810
Storen_1_1	3	c	Gravel	Down	6.8	6160	23	80	810
Storen_1_1	3	c	Sand	Down	6.8	6160	23	80	810
Storen_1_1	5	a	Soil	Down	10.3	6160	23	80	810
Tanem_2_2	0	a	Gravel	Flat	2.97	2300	13	80	1074
Tanem_2_2	3	a	Clay	Flat	16.3	2300	13	80	1074
Tanem_2_2	5	a	Clay	Flat	NA	2300	13	80	1074
Tanem_2_2	5	c	Clay	Flat	5.78	2300	13	80	1074
Tanem_3_1	0	a	Sand	Flat	4.78	2300	13	60	1074
Tanem_3_1	0	b	Sand	Flat	3.75	2300	13	60	1074
Tanem_3_1	3	a	Silt	Flat	25.9	2300	13	60	1074
Tanem_3_1	3	c	Silt/clay	Flat	29.3	2300	13	60	1074
Tanem_3_1	5	a	Marsh	Flat	61.4	2300	13	60	1074
Tanem_3_1	5	c	Silt/clay	Flat	88.1	2300	13	60	1074
Tanem_3_1	6	a	Silt/clay	Flat	61.4	2300	13	60	1074
Vennastrand_1_1	0	b	Gravel	Up	4.65	650	10	80	1485
Vennastrand_1_1	3	c	Gravel	Up	28.4	650	10	80	1485
Vennastrand_1_1	5	c	Gravel	Up	29.7	650	10	80	1485
Vennastrand_1_2	0	a	Gravel	Up	5.6	650	10	80	1485
Vennastrand_1_2	5	a	Soil	Up	13.4	650	10	80	1485
Vikhammer_2_2	0	b	Sand	Down	7.28	3400	12	60	919
Vikhammer_2_2	3	c	Silt	Down	7.26	3400	12	60	919
Vikhammer_2_2	5	c	Clay	Down	6.88	3400	12	60	919

markers and then summarized to represent the sum of markers. The limit of detection (LOD) of each marker compound was determined by summarizing the S/N value for 4 levels of SBR (1, 2, 5 and 10 μg SBR and 2 replicates per level). The S/N level was plotted against the concentration level of SBR to determine the S/N vs concentration relationship following the method by Donovan (2016), using power of regression. The S/N for each marker (from SBR 1-100 μg/cup) were 14.0–560 for benzene, 0.71–57.4 for methylstyrene, 0.62–7.59 for ethylstyrene and 0.77–11.2 for the butadiene trimer. The calculated LOD (3 x S/N) using the sum of markers were 0.2 μg SBR and LOQ (10 x S/N) were 0.6 μg SBR, however, the calibration range used for this study was 1-100 μg of SBR.

The peak heights of all markers were compared to the peak heights of the markers from SBR1500 reference material and from reference tires previously published (Rødland et al., 2022c). In soils there is a potential for competing ions in the organic matter of the soil to impact the analysis, especially the benzene (mz 78) and ethylstyrene (mz 117). The butadiene trimer (mz 91) has shown to be less impacted from competing ions. If the marker combinations do not exhibit the normal ratio pattern expected to find in tires, only the butadiene trimer was used for quantification. If both the marker combinations and the butadiene trimer showed values that were out of range (under or over the calibration curve range), the sample was discarded.

2.4.3. Tire wear calculation

Calculation of tire wear particle concentration based on the measured SBR concentration following the methods described in Rødland et al. (2022c). In brief, the SBR concentration values are first converted with a styrene conversion factor of 0.94 to adjust for the different styrene content used in the SBR1500 calibration samples (23 %) compared to the average styrene content reported for tires (15 % (Unice et al., 2012; Unice et al., 2013). The adjusted SBR values are then used to calculate the TWP concentration based on previously measured levels of SBR in 31 relevant car and truck tires for Norway (Rødland et al., 2022c) by applying the *Eq. 1* and predicting the TWP concentration in each sample using Monte Carlo simulation. This method is further described in (Rødland et al., 2022c).

$$M_T = \frac{M_S * Sc}{(S_{PV} * R_{PV}) + (S_{HV} * R_{HV})} \quad (1)$$

where.

M_T is the mass of tire in a sample (mg);
M_S is the mass of SBR + BR in a sample (μg);
Sc is the conversion factor for styrene content in standards vs tires;
S_{PV} is the mass of SBR + BR in personal vehicle tires (μg/mg);
R_{PV} is the ratio of personal vehicles at the sampling location;
$S_{HV}$is the mass of SBR + BR in heavy vehicle tires (μg/mg);
R_{HV} is the ratio of heavy vehicles at the sampling location;

2.5. Statistical analyses

The tire particle concentrations were calculated and predicted by Monte Carlo Simulation (Crystal Ball Add-In, Microsoft Excel), as described in (Rødland et al., 2022c). The distribution of both datasets (personal vehicle (PV) and heavy vehicle (HV) tires) were tested in the Crystal ball and normal distribution were chosen as best fit for both datasets. The model applied 100,000 simulations and the prediction statistics obtained for were mean, median, standard deviation, minimum, maximum, and the 10th, 25th, 75th and 90th percentiles for each sample. 2023.03.0 + 386The statistical analysis of the data was conducted in RStudio 1.3.109, R version 4.2.3 (2023), using the ggplot2-package (Lai et al., 2016) (gplot2_3.3.3), the car-package (Fox, 2019) and the dplyr-package (Wickham et al., 2018) for creating boxplot graphs, linear regression and for performing Analysis of Variance (ANOVA). The correlation between SBR/TWP and the other road pollutants were explored in RStudio using a a correlogram (Soetewey, 2020).

ANOVA were used to evaluate if the difference between TWP concentrations grouped according to soil type, soil profile, sites, depth, distance and precipitation were significant. For the factor soil type, the evaluation was done by Redundancy analysis (RDA) and not ANOVA due to low number of samples per group. For some of the tests, the TWP concentrations were log-transformed before performing the ANOVA (TWP grouped by sites; soil profile; depth). For TWP grouped by distance, original data were applied for the ANOVA. The assumption of normal distribution of residuals was tested using an Andersen-Darling normality test. The assumption of equal variance was tested using Levene's Test of Homogeneity of Variance. Whenever this assumption was not met, Welch's one-way ANOVA was used. The statistically significant level was set to $p = 0.05$.

Redundancy analysis (RDA) was performed to assess the relationship between the response variables (SBR, Zn, Ni, Cd, Cr, Pb) and explanatory variables (traffic speed, annual average daily traffic (AADT), % heavy vehicles and distance from the road). The RDA was conducted by using Canoco 5.12 (Braak and Šmilauer, 2018). The dataset was first explored using a constrained RDA with all variables selected. This enables a full exploration of the total variation explained by all variables. Following this, RDA with forward selection were tested, where the explanatory variables contributing the most to the variation can be selected until there is no more variation to explain. In the forward selection mode, both the simple effects (the effect of each independent variable) and the constrained effects (the effect of the variable considering the other variables) were tested. The significance level in the RDA is derived by Monte Carlo permutation tests (9999 permutations performed). For all tests, $p < 0.05$ is set as the level of significance.

3. Results

3.1. Analysis of tire wear particles

To test the need for pretreatment to remove organic matter before PYR-GC/MS, triplicate samples from three sites were analyzed with and without the use of hydrogen peroxide (H_2O_2). There was no clear indication that using H_2O_2 had a significant impact on the detection of SBR + BR rubber in the soil samples. Before the treatment, the organic matter content (LOI, %) of the Vikhammer 0 m, Flakk 5 m and Venna 5 m samples were 4 %, 15 % and 30 %, respectively. Accounting for the variation in the replicates (standard deviation), there was no change in the average concentration of SBR in the samples tested (Table 2).

Another way of investigating how the pretreatment impacts the analysis is to look at the ratio of relative peak height (%) of the four pyrolysis markers, *benzene, methylstyrene, ethylstyrene and butadiene*. If any of them are impacted by competing products from the soil matrix, we would expect a change in the relative contribution of the marker. Again, there seems to be very little change comparing before and after treatment of the samples, which indicates that the analysis of SBR + BR rubber using these pyrolysis markers are not impacted by the presence of organic material in the soil.

The remaining samples were therefore analyzed without the use of H_2O_2. A total of 268 samples were analyzed (including triplicates), in which 13 samples were discarded due to being out of the calibration range. Out of the 255 samples, 49 samples (SI Table S2) were quantified using only the butadiene trimer due to potential impacts on the other markers. This impact was observed by a change in the relative % marker contribution from benzene (m/z 78) compared to expectations from the SBR calibration samples (Fig. 2). For these 49 samples, the calculated concentrations of SBR were reduced by 41–91 % when applying only the butadiene trimer as a marker. The average SBR concentration for each sample was calculated, and 64 samples in total were used for the statistical analysis.

The SBR concentrations for all samples ranged from 640 mg/kg to

Table 2
Concentration of SBR rubber (μg/mg) in three different soil samples (Flakk 5 m, Vikhammer 0 m and Venna 5 m) with no treatment and with hydrogen peroxide (H_2O_2) treatment indicated no effect of treatment. The relative contribution from four chosen markers for SBR rubber (*m/z* 78 benzene, m/z 118 methylstyrene, m/z 118 ethylstyrene and m/z 91 butadiene trimer) before and after treatment indicated no distinct change in marker signals.

No treatment	SBR + BR (μg/mg)		Relative contribution of markers (%)			
	Mean	Std	m/z 78	m/z 118	m/z 117	m/z 91
FLAKK 5 M	2.53	0.27	91	3	4	1
VIKHAMMER 0 M	2.77	0.49	87	9	4	1
VENNA 5 M	4.37	0.94	93	2	3	2
	SBR + BR (μg/mg)		Relative contribution of markers (%)			
H_20_2 treatment	Mean	Std	m/z 78	m/z 118	m/z 117	m/z 91
FLAKK 5 M	2.20	0.20	88	7	4	1
VIKHAMMER 0 M	2.09	0.13	84	12	3	1
VENNA 5 M	3.32	0.80	93	2	3	2

8200 mg/kg (3600 ± 2000 mg/kg, $n = 64$) (SI Table S3). For each sample, the SBR concentration was used to calculate the TWP concentration with Monte Carlo prediction modelling. The predicted mean concentrations of TWP between different sites ranged from 2040 to 26,400 mg/kg TWP (mean of all sites ± st.d., 11,400 ± 6420 mg/kg) (Table 3). The predicted median, standard deviation, minimum, maximum, 10th. 25th, 75th and 90th percentile for all samples can be found in SI Table S4. The variation found for different sites (SI Fig. S18) were statistically significant (ANOVA, $p < 0.05$), although the differences were too low to determine which sites were different to each other. There were no statistically significant difference between samples taken at the top soil layer (a: 0–0.1 m, 11,000 ± 6100 mg/kg) or the lower soil layer for samples at 0 m (b: 0.1–0.2 m, 11,150 ± 7690 mg/kg) and at >0 m (c: 0.1–0.3 m, 12,300 ± 6700 mg/kg).

Comparing the TWP concentrations for all samples showed that there were no significant difference between samples grouped by distance (ANOVA, $p = 0.73$, Fig. 3). The median value (19,900 mg/kg, $n = 4$) for samples collected >6 m distance from the road was almost a factor of two higher compared to 0 m (9200 mg/kg, $n = 21$), 1 m (10 300 mg/kg, n = 21) and 5 m (9200 mg/kg, $n = 18$) (Fig. 3). Note, however, the relatively low number of samples collected from >6 m compared to the other distances.

Comparison between samples collected from the surface layer

Table 3
Summary table over the predicted mean tire wear particle (TWP) concentration (mg/kg) for all sites (for all distances and depths).

Site	Minimum mg/kg	Median mg/kg	Mean mg/kg	S.D. mg/kg	Maximum mg/kg
Bjørndalen 2.1	9200	16,100	16,100	7200	23,100
Bjørndalen 2.2	8200	8500	8500	496.0	8900
Bratsberg 2.2	12,000	17,000	15,000	2900	17,000
Flakk 2.1	2600	8000	8200	4800	14,200
Gjølme 1.2	4100	6000	5700	1200	7360
Levanger 1.2	2000	5500	5200	3000	7980
Mosløkkja 3.1	3700	7900	7900	5900	12,000
Rinnleiret 3.1	6000	13,000	13,800	7800	25,700
Skjenalddalen 1.1	4600	5900	7200	4100	15,400
Skjenalddalen 3.1	9200	10,900	12,300	3700	18,300
Støren 1.1	9600	10,300	10,400	819.0	11,800
Tanem 2.2	3000	11,100	10,200	5290	15,500
Tanem 3.1	5100	18,600	15,400	8630	26,400
Vennastrand1.1	4200	4400	10,700	11,100	23,500
Vennastrand1.2	18,300	21,100	21,100	3960	23,900
Vikhammer 2.2	2900	14,300	16,300	4770	21,700
All samples	2000	9820	11,440	6420	26,400

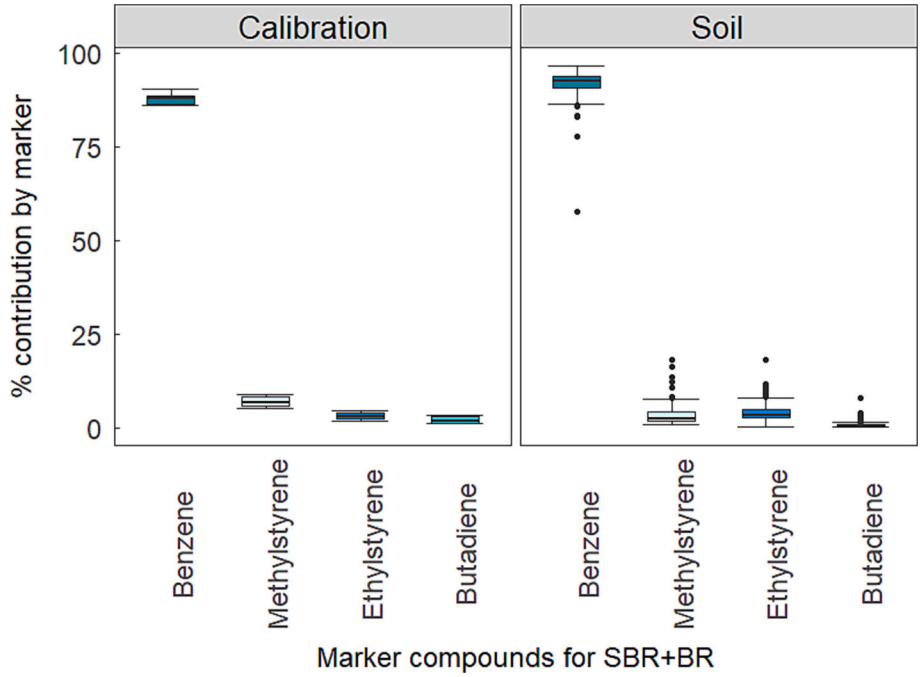

Fig. 2. The relative percentage (%) of marker contribution based on the normalized peak height by each marker (benzene, methylstyrene, ethylstyrene and butadiene trimer) against the sum of all markers. Peak height signal normalized against the peak height signal from internal standard (deuterated polybutadiene).

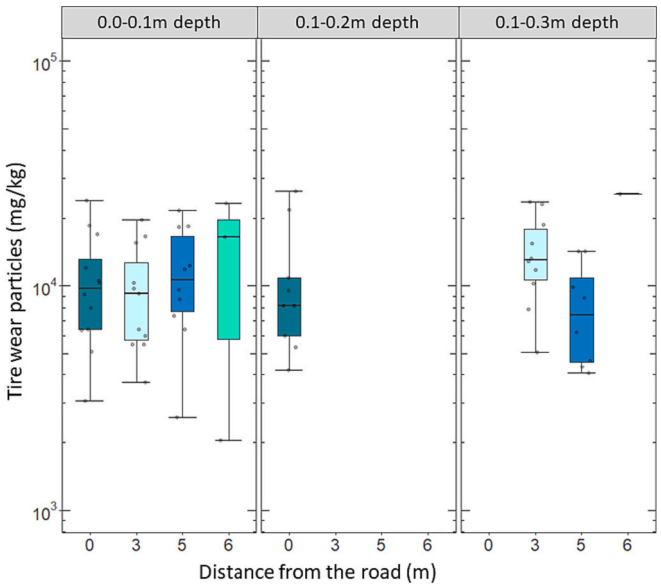

Fig. 3. Concentration of tire wear particles in road-side soil by distance from the road (0 m, 3 m, 5 m, >6 m) and by depth (0.0–0.1 m, 0.1–0.2 m and 0.1–0.3 m).

(0.0–0.1 m) and deeper layer (0.1–0.2 m) showed no significant difference (ANOVA, $p = 0.571$). There was also no statistically significant difference between sites grouped by soil profile (ANOVA, $p = 0.408$, Fig. S3) A weak but significant relationship was found between TWP and the annual precipitation, where precipitation could explain 5.4 % of the variation in TWP concentration across different sites (R2-adj =0.054, $p = 0.035$). The sites could be classified by the different soil types (agricultural soils, clay, silt, sand, silt/clay mixtures, gravel, marsh land and topsoil), determined by the grain size of the soils (NPRA, 2005). There was no significant difference found for the soil types (RDA $R^2 = 0.00$, $p = 0.484$), although there is a trend showing that the samples collected from marsh soils had higher concentrations of TWP (17,300 ± 6960 mg/kg) compared to overall average values for all samples (Fig. 4).

The organic matter (OM) in the different soil samples were measured by loss of ignition (LOI, %) in all samples except two (Tanem 2.2 5 m-a and Levanger 1.2 6 m-a). Four classes of LOI were assigned, 2–5 % ($n = 13$), 5–10 % ($n = 22$), 10–20 % ($n = 15$), >20 %($n = 12$). For TWP levels grouped by the LOI (Fig. 5), the results indicate a slight tendency towards higher levels in the groups with higher LOI, however, the differences between the groups were not significant (ANOVA, $p = 0.655$).

3.2. Road-related metals

The average concentration of the selected metals varied across the different sites. The highest concentrations were found for Zn (66.8 ± 42.0 mg/kg, Table 3), followed by Cr (39.9 ± 18.8 mg/kg), Cu (39.9 ± 47.8 mg/kg), Pb (24.0 ± 34.2 mg/kg), Ni (23.6 ± 14.8 mg/kg), and As (7.97 ± 14.4 mg/kg).

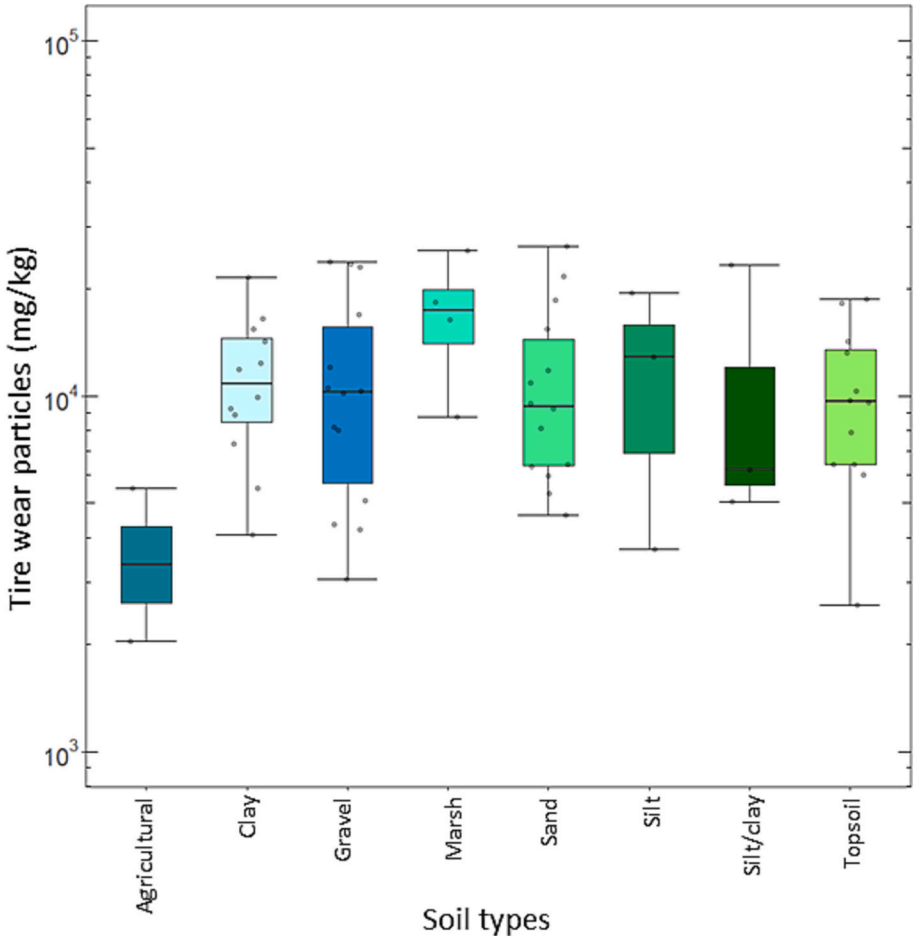

Fig. 4. Concentration of tire wear particles (mg/kg, logarithmic scale) grouped by the different soil types (agricultural soils, clay, silt, sand, silt/clay, gravel, marsh land and soil).

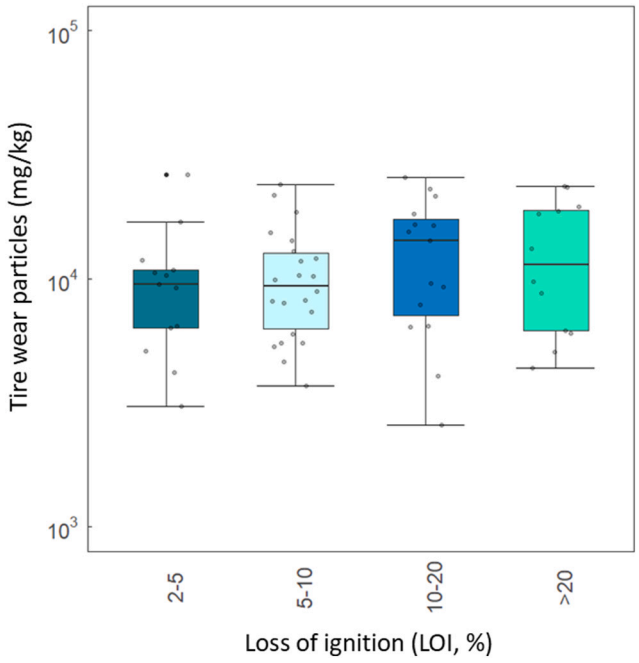

Fig. 5. Concentration of tire wear particles (mg/kg, logarithmic scale) grouped by the amount of organic matter, as loss of ignition (LOI %).

The EFs were calculated for all metals and showed that especially some areas did have levels of metals and metalloids exceeding the norm value (the expected background level) of Trøndelag (Table 4). Especially two sites, Rinnleiret 3.1 and Skjenalddalen 3.1, exhibit elevated levels (EFs >1 indicating anthropogenic sources) for both As (EFs 1.3–4.9) and Pb (EFs 1.2–2.3). The highest levels of As were found at Tanem 3.1 (0 m), where EFs of 11.4 were calculated, indicating severe enrichment of As at this site. For Zn, only one site exceeded the norm values (Levanger 1.2 0 m), and for Ni and Cr there were no sites with EFs >1.

Soil accumulation of metals as a function of distance from the road was found to be statistically significant for Cu (ANOVA, $p = 0.0109$) between 0 m and 5 m ($p = 0.0371$) and 0 m and 6 m distance ($p = 0.0347$) (Fig. 6). No statistically significant relationship was found between distance and Zn (ANOVA, $p = 0.174$), Ni (ANOVA, $p = 0.314$), Pb (Welch ANOVA $p = 0.249$), Cr ($p = 0.276$) or As ($p = 0.113$). The soil depth had no statistically significant impact on any of the metals (Fig. 7) (ANOVA, Zn $p = 0.158$, Cu $p = 0.174$, Ni $p = 0.295$), Pb $p = 0.457$, Cr $p = 0.246$), however, the levels for As indicates that there is a difference between levels in 0.0–0.1 m depth and 0.1–0.2 m depth, with highest median values (6.0 mg/kg) in the 0.1–0.2 m depth (Welch ANOVA As $p = 0.0319$). Grouped by organic matter content (LOI %) (Fig. 8), significant differences between the LOI levels where found for Cu (ANOVA $p = 0.0000084$), Ni (ANOVA, $p = 0.000052$) and Cr (ANOVA, $p = 0.000017$). No significant differences based on the LOI groups were found for Zn (ANOVA, $p = 0.991$), Pb (ANOVA, $p = 0.156$) or As (Welch's ANOVA, $p = 0.250$) (Fig. 8). Significant correlation between metals are indicated in Fig. 9.

Table 4
Descriptive statistics for metals zinc (Zn), nickel (Ni), lead (Pb), copper (Cu), chromium (Cr) and arsenic (As). Norm values for soils in the Trøndelag region are used to evaluate the background level of each metals and metalloid. All values are in mg/kg.

	Zn	Ni	Pb	Cr	Cu	As
No. samples (n)	64	64	64	64	64	64
Norm values	200	75	60	100	100	8
Min.	19.1	<5.0	1.50	6.42	6.8	<0.50
Median	53.5	22.8	9.70	36.6	29.2	2.80
Mean	66.8	23.6	24	39.9	39.0	7.97
S.D.	42	14.8	34.2	18.8	47.8	14.4
Max.	272	69.3	140	106	371	91.1

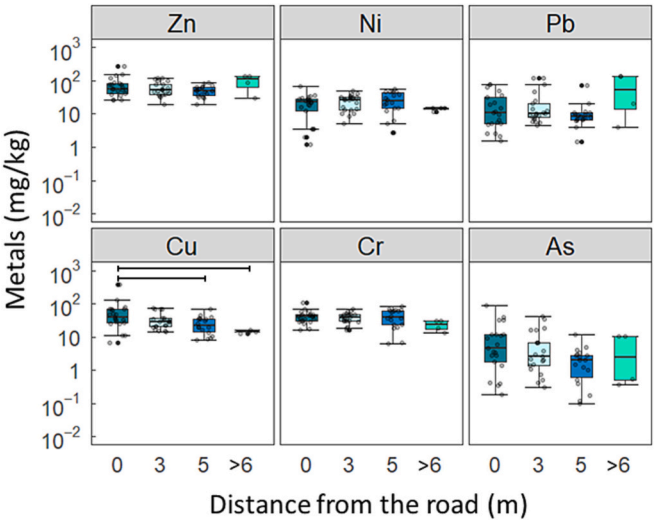

Fig. 6. Boxplot of the levels of metals (zinc (Zn), nickel (Ni), lead (Pb), copper (Cu), chromium (Cr) and arsenic (As)) grouped by the distance from the road (m). All values are in mg/kg (logarithmic scale). Significant difference was found for As between 0 m and 5 m, and between 0 m and 6 m distance, indicated by lines in the figure.

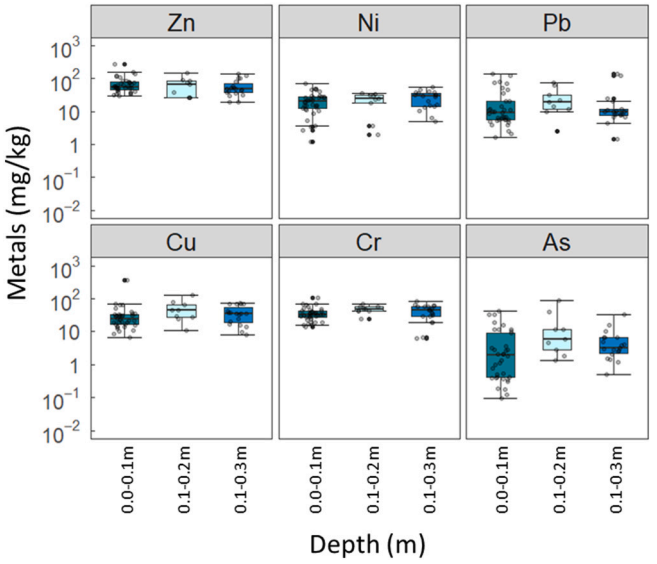

Fig. 7. Boxplot of the levels of metals (zinc (Zn), nickel (Ni), lead (Pb), copper (Cu), chromium (Cr) and arsenic (As)) grouped by the soil depth (m). All values are in mg/kg (logarithmic scale).

3.3. Tire wear particles compared to metals

The relationship between SBR and metals can be tested by linear correlations. Using a correlogram, the correlations between all pairs of variables chosen can be tested simultaneously (Fig. 9). From this analysis we see that there are no strong relationships between SBR, and the other road pollutants tested. There is no statistically significant linear relationship between SBR and Cr, Cu or Ni. Concentrations of SBR were positively correlated with As ($R = 0.37$) and Pb ($R = 0.31$), but no correlation was found for SBR with other metals included in the study. Noteworthy, Cr and Ni ($R = 0.79$) and Cu and Zn ($R = 0.58$) were

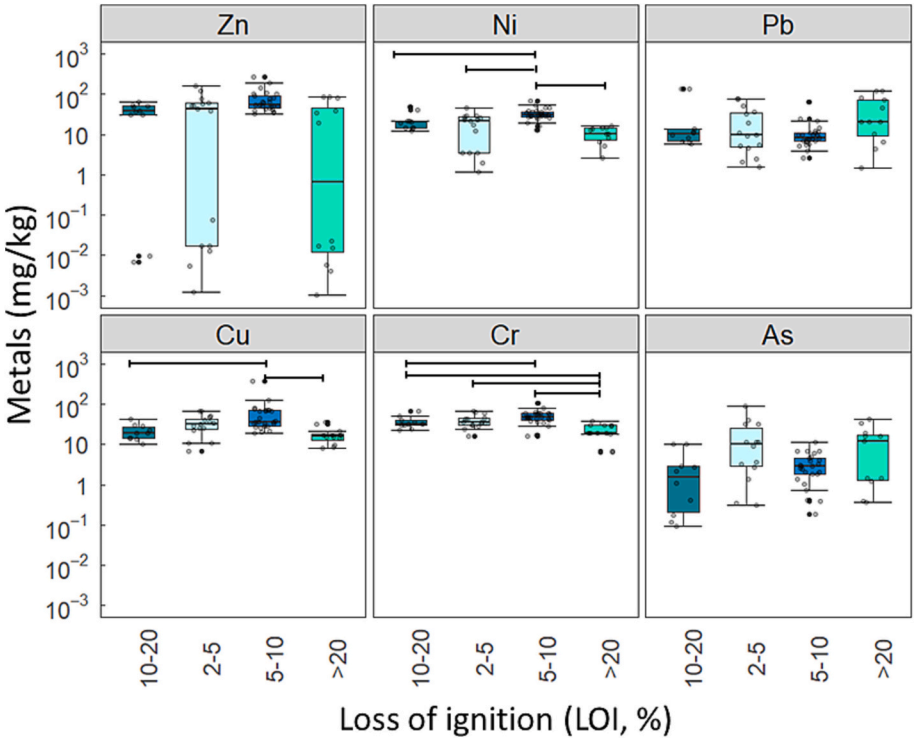

Fig. 8. Boxplot of the levels of metals (zinc (Zn), nickel (Ni), lead (Pb), copper (Cu), chromium (Cr) and arsenic (As)) grouped by organic matter content (loss of ignition, LOI %). All values are in mg/kg (logarithmic scale). Significant difference was found for Ni, Cu and Cr, indicated by lines in the figure.

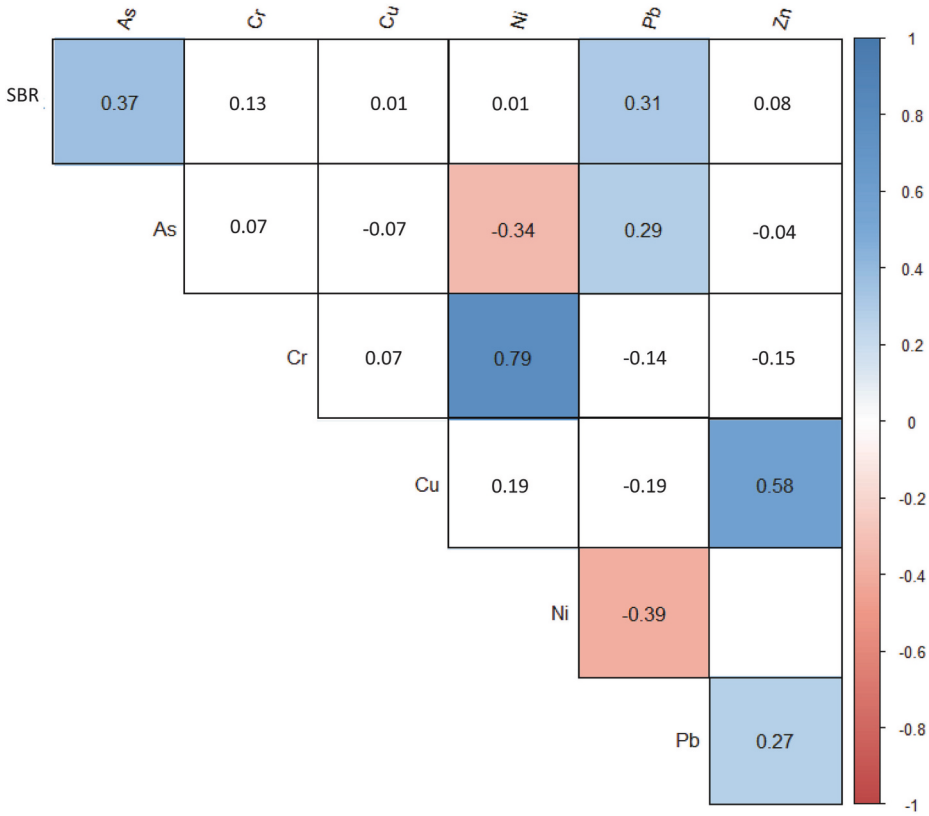

Fig. 9. Correlogram between SBR and the metals CR, Cu, Ni, PB and Zn, and the metalloid As log transformed. The correlogram shows correlation coefficients for all pairs of variables. The positive correlations are depicted in blue and the negative in red. The stronger the colors or higher values towards 1 or − 1 shows the strength of the correlation. For the pairs where the correlation is not significantly different from 0, no linear relationship can be detected and therefore there are no significant correlation detected. These are shown as white boxes in the correlogram, with no values provided. The relationships between tire wear particles and metals were also tested against different explanatory factors, using redundancy analysis (RDA). A large part of the variation (41.4 %, $p = 0.0001$) of tire rubber and the metals (As, Cr, Ni, log transformed: Cu, Pb, Zn) were explained by distance, depth, soil types, annual precipitation, slope profile, LOI (log transformed), AADT (log transformed) and % HV as explanatory factors (summary effects RDA, Fig. 11, Tables S5-S8). Using simple effects RDA, it is found that the AADT (10.4 %), slope profile (flat, 9.1 %) and LOI (8.4 %) explains most of the variation. In addition, soil types (clay, gravel, marsh, sand, 24.5 %), slope profiles (flat, down, 14.4 %), distance (3.9 %) and annual precipitation (3.9 %) were significant explanatory variables. For the conditional effect RDA, the explanatory variables with significant effect on the variation were AADT (10.4 %), LOI (9.2 %), soil types (clay, gravel, 11.3 %), traffic speed (3.9 %) slope profile (down, 3.3 %), annual precipitation (2.7 %), soil depths (0–0.1 m, 2.8 %) and %HV (2.7 %).

strongly correlated, whereas positive correlations were also found between Pb and As (R = 0.29) and Pb and Zn (R = 0.27). Negative correlations were found for As and Ni (R = -0.34) and Ni and Pb (R = −0.39).

The correlation between TWP and the explanatory variables alone were also tested by performing an RDA, which essentially is a linear correlation between TWP and each of the explanatory variables (see Fig. 10). However, no significant relationship was found between TWP and the explanatory variables using conditional effect RDA (R2-adj = 0.022, p = 0.446), although simple effect RDA indicates a significant correlation between traffic speed (7.2 %, p = 0.0358) and precipitation (6.5 %, p = 0.0473) Removing TWP from the response variables and performing RDA with the metals alone increased the percentage explained to 48.6 % (P = 0.0001). The most important explanatory variables (simple effects RDA) were still AADT (12.1 %) and LOI (9.5 %), whereas soil types (marsh, gravel, sand and clay) and slope profiles summarized (down and flat) explained 27.7 % and 16.1 % of the variation, respectively. Results for all variables are summarized in Table S5-S8.

4. Discussion

4.1. Analytical challenges

One of the objectives of this study was to analyze TWP levels in environmental samples with as little pretreatment as possible in order to increase the number of samples that could be analyzed. Thermal analysis without pretreatment have previously been done for German (93.3–104.8 %) (Müller et al., 2022a) and Norwegian (88.1 ± 14.7 % recovery) (Rødland et al., 2022c) roadside soils, demonstrating good recovery results. However, the traffic density in both of these studies were medium (AADT 36000 v/d, Müller et al. (2022a)) to high (AADT 71250 v/d, Rødland et al. (2022c). As the sites from the present study were from low traffic rural areas, investigating the effect of pretreatment in order to enhance the signals for TWP was needed. The pretreatment tests demonstrated that there was very little effect of pretreatment (H_2O_2) on the concentration of SBR in the samples, as well as on the markers used in the quantification of SBR and BR rubber. Based on these results the remaining samples were analyzed without pretreatment. However, when investigating the % contribution of each single marker for the remaining samples analyzed, it was observed that the % marker

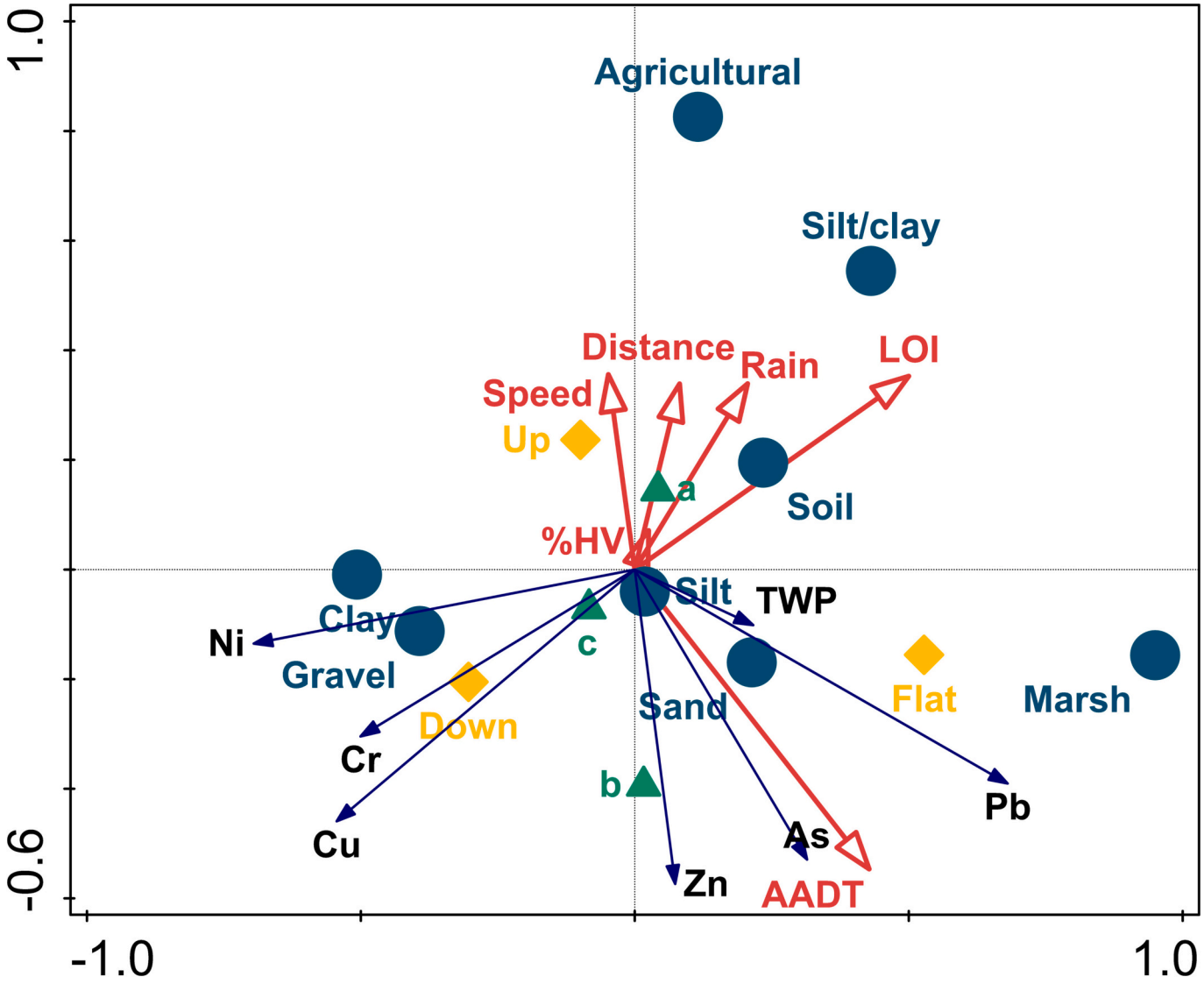

Fig. 10. Plot of the redundancy analysis (RDA) with response variables (TWP, As, Cr, Ni, Cu (log-transformed), Pb (log transformed) and Zn (log-transformed) against the explanatory variables distance, depth, soil types, slope profile, LOI, AADT, % HV and annual precipitation (rain). Explanatory variables explained 41.4 % of the variation in the response variables (p = 0.0001).

contribution from *benzene, methylstyrene* and *ethylstyrene* differed from what was expected based on the calibration samples (SBR1500, Fig. S17) and previous analysis of 31 different tires (Rødland et al., 2022c). As the marker *butadiene trimer* did not display the same signal variation, for these samples only *butadiene trimer* was used for quantification in order to minimize the potential overestimation of TWP concentration due to competing signals. However, the use of the *butadiene trimer* as a single marker is also sensitive to potential background noise by other components present in the samples due to low peak height signal as demonstrated by its low S/N, which is contributing to uncertainty for those samples analyzed with only the *butadiene trimer*.

The presence of organic matter in soils has previously been suggested to cause issues in quantification of microplastic particles (Bläsing and Amelung, 2018). Although the removal of organic matter using H_2O_2 had limited effect on the quantification of SBR in this study, the results also showed that some samples did have potential issues with signal interference in some samples. This suggests that additional methods for quantification of TWPs in complex matrices should be explored. More et al. (2023) recently investigated the difference in PYR-GC/MS results for TWP in environmental sediment samples with and without pretreatment (H_2O_2, potassium hydroxide (KOH), thermal desorption), concluding that the TWP levels found were lower when applying pretreatment methods. This indicates the presence of competing signals elevating the marker signals of PYR-GC/MS when analyzing samples without pretreatment. KOH treatment was also suggested as a required step for environmental samples in order to increase the mass of the sample analyzed with PYR-GC/MS (from 5 to 10 mg/cup) (More et al., 2023), however, the average sample mass analyzed in our study were 11.5 mg d.w. without treatment. Another reason to use pretreatments would be to prolong the need for time-consuming cleaning of the PYR-GC/MS instruments, however, these pretreatment methods are also time consuming, so the choice of pretreatments and analysis methods for a specific sample matrix need to consider both the potential cost and quality of the data possible to obtain from each method.

4.2. Tire wear concentrations

Surprisingly, the TWP concentrations in the Trøndelag roadside soils were more than a factor of 5 higher than the maximum concentration previously reported in Norwegian soils (Skullerud highway) (Rødland et al., 2022c) and 1.6 times higher compared to the maximum value found in German soils (Bundesstraβe B27) (Müller et al., 2022a). The levels of TWPs in Trøndelag were also higher than previously reported TWP values in other solid matrices; such as freshwater sediments 20–2000 mg/kg in France (Seine), 46–420 mg/kg in the US (Chesapeake), 28–580 mg/kg in Japan (Yodo) (Unice et al., 2013); road dust (70–12,400 mg/kg) (Eisentraut et al., 2018; Hopke et al., 1980; Klöckner et al., 2020; Kumata et al., 2000; Kumata et al., 2002; Rogge et al., 1993; Zakaria et al., 2002), tunnel dust (120–25,600 mg/kg) (Klöckner et al., 2021; Kumata et al., 2000; Rødland et al., 2022a; Wik and Dave, 2009) and sediments of gully-pots (80–15,000 mg/kg) (Mengistu et al., 2021; Rødland et al., 2022a) and sedimentation basins (13,000 ± 1500 mg/kg) (Klöckner et al., 2019). The traffic density in these comparable TWP studies were between 10,000–250,000 vehicles/day, which is >17 times higher than the highest traffic density for the sites used in this current study. In the study of Unice et al. (2013), a trend of increasing TWP concentrations with traffic density was found for samples <3 m distance from the road, although this was not statistically different. This supports the results presented in this current study, where no statistically significant difference between samples were found with either traffic density or distance from the road. Previous studies of TWP have reported a significant relationship with traffic speed, where increasing traffic speed (from 40 to 80 km/h) was associated with increasing TWP concentration in road-side snow (Rødland et al., 2022b). Although a significant correlation was found between TWP and traffic speed (60–80 km/h) using simple effects RDA, traffic speed only explained 7.8 % of the observed variation, compared to 22 % found for road-side snow. One possible explanation for the weaker relationship between TWP and traffic speed found in this study is the low variation in traffic speed, from 60 to 80 km/h, which would have lower impact on the TWP results compared to studies with larger variations in traffic speed. There are also evidence showing that there is a large difference between the traffic speed limit and the actual driving speed, where the difference is particularly well expressed when speed limit is >60 km/h (Haglund and Åberg, 2000). Thus, any variation in TWP release through different traffic speeds are masked by the lack of information on the true driving speed. Another aspect that could potentially be of more importance than the driving speed is the driving behavior. All roads included in this study had similar road patterns, with no stop signs, traffic lights, roundabouts or similar, thus stimulating a similar driving behavior across all sites. Driving behavior has previously been demonstrated to have a significant impact on the levels of TWP in roadside snow (Rødland et al., 2022b).

Another potential explanation for the observed high levels of TWP found in our study compared to previous road-side soil studies is that highways usually have drainage systems to remove water from the driving lane quickly, hence removing some of the TWPs potentially being transported to the nearby soils along roads that do not have these systems. Another factor could be the roadside design. For highways and urban areas, the roadsides are often constructed with soil materials to either retain pollutants, such as grass-filled swales or bioretention filters (Blecken, 2016; Bäckström et al., 2004; Flanagan et al., 2018; Hatt et al., 2009), or quickly drain road runoff into the ground and transport it away from the road system. The site at Skullerud is an example of the latter, where the road runoff is transported through the drainage system in the road side terrain into a sedimentation pond for treatment (Rødland et al., 2022c), thus a possible explanation of why the TWP concentration in the soils were lower here. In Trøndelag, none of the sites have sophisticated drainage systems (except gully-pots at some sites) or constructed swales. Thus, runoff and splash and spray should be considered the main transportation of road pollutants from the road to the road-sides at these sites, in accordance with previous studies on urban soils (Sager, 2020). This is also supported by the significant linear correlations found between TWP/SBR and annual precipitation at the different sites, at 5.4 % (ANOVA) and 6.5 % (simple effects RDA). Although precipitation explained a relatively low percentage of the variation, there is an indication that higher levels of precipitation together with the slope profiles of the roadsides can explain why we found no significant difference between the distance from the road. The high levels of TWPs found in these low traffic rural roads indicate that the TWPs accumulate in these soils over time, thus understanding the transport of TWPs also in low traffic areas is important for future studies. The relationship between soil types and TWP concentrations in this study did not find any significant differences, however, it indicated that there are potentially higher levels associated with marshland soils. These soil-TWP relationships should be further investigated to understand how soil properties may impact the retention of TWPs along roads. Retention of TWP in roadside soils may serve as a measure to prevent spreading of TWP into waters.

4.3. Metals and metaloids

The average concentration of metals in the road side soil found at the Trøndelag sites were generally low, but comparable to levels previously reported in road-side soils from the highway at Skullerud, Norway (As 2.53–5.6 mg/kg, Pb 6.7–15 mg/kg, Cu 32–51 mg/kg, Cr 23–32 mg/kg, Ni 17–22 mg/kg and Zn 130–180 mg/kg (Johnsen and Aaneby, 2019). The levels of metals at Trøndelag were also comparable to roadside soil studies in both urban areas (Zn 92.3 ± 37.7 mg/kg) and more rural roads in Hangzhou city, China (Ni, 24.0 ± 6.30, Cu 39.9 ± 19.6 (Wang and Zhang, 2018) and along highways in Europe within 5 m distance from the road (Cd: 0.1–10.5 mg/kg, Cr: 0.42–240.3 mg/kg, Cu: 7–413

mg/kg, Ni: 2.7–228 mg/kg, Pb: 5.1–1769 mg/kg, Zn:25–1580 mg/kg) (Werkenthin et al., 2014). Some of the sites at Trøndelag did exceed the maximum values reported in other studies, and this is likely related to elevated background levels of As (2.3 % above national norm value), Pb (1.4 %), Cr (57 %) and Ni (35 %) found in the soils of Trøndelag (Andersson et al., 2011).

The relationship between metals in roadside soil and the distance from the road has been described in several studies, where most metals demonstrate a significant decrease in concentration with distance from the road (Aslam et al., 2013; Clarke et al., 2015; Zhang et al., 2015). Werkenthin et al. (2014) reviewed the metal concentrations in road-side soils across 27 studies and 64 sites in Europe and found that the metal concentrations in the topsoil in the first 5 m from the road was significantly influenced by traffic density. Strong correlations were found for Cd and Cr, but only moderate correlations with Cu and Zn. However, there are also studies where no such correlation between metals and traffic was found (Pérez et al., 2008). The lack of correlation with traffic density is suggested to be related to the variations in other traffic variables such as braking and acceleration, traffic pattern, road construction and road maintenance (Werkenthin et al., 2014). Others have found that the natural variations in the soil, especially the organic matter content (LOI) are strongly related to the concentrations of metals (Ni and Cr) and thus reduced the correlation between metals and traffic variables (Carlosena et al., 1998).

Previous studies have suggested that the impact of road pollution in soils does not reach further than 20 m distance from the road (Swaileh et al., 2004). No such relationship with distance were observed in the roadside samples of Trøndelag, where metal concentrations grouped by distance were only significant for Cu. The relationship between the metals and the different explanatory factors were further investigated using RDA. Contrary to the results for TWP, the RDA analysis revealed that the variations found for the road metals could be explained by the different explanatory variables, in which the AADT and LOI were the most important variables, followed by soil types and the slope profiles. No significant relationship was found between the metals and traffic speed, however, as previously discussed for TWP concentrations, this might be due to the lack of variation in traffic speed at the sites investigated. While many confounding factors precluded identifying strong relationships with traffic variables in previous studies of urban area roadside soils, both pH and soil organic matter were found to be correlated with concentrations of Cu, Pb, Cd, Cr and Zn (Wang and Zhang, 2018), in which higher organic matter content favored retention of metals. The RDA analysis in our study also shows that the organic matter (LOI) is important in explaining the variation of road metals in the soils (explaining 10 %). However, it is negatively correlated with Ni, Cr and Cu and only positively correlated with Pb and As.

4.4. Relationship between TWP and metals/metaloids

As demonstrated, this study found no significant correlation between TWP and the metals/metaloids in the soil. This is in contrast to previous studies, where presence of Zn in road-side soils have been linked to the presence of tire particles (Councell et al., 2004; Goonetilleke et al., 2017), as Zn has been demonstrated to leach out from tire particles in soil (Smolders and Degryse, 2002). However, there was no significant correlation between the levels of TWP and Zn in the Trøndelag soils (R^2 = -0.01, p = 0.546, linear regression). Previous studies have tested the use of Zn as a marker for tire wear particles (Klöckner et al., 2019; Müller et al., 2022a), although recognizing the need to remove other non-tire related Zn sources from the sample in order to use Zn as a marker (Wagner et al., 2018). Other potential Zn sources along roads are brake dust, road paint (Wagner et al., 2018) and the Zn-coated guardrails (Blok, 2005), which were present at several of the sample sites in Trøndelag. The presence of other Zn sources and potential impact of higher background levels of As, Pb, Cr and Ni in the soil might explain why this study failed to find any significant relationship between TWP and the metals/metaloids. As such, this is potentially a local issue due to the geochemical features of the Trøndelag area. However, it does demonstrate the need to be aware of such influences if Zn or other metals are used to estimate the presence of TWP in environmental samples. Another aspect that is not fully understood is how the fate of tire-related metals in soil is affected by impact on leaching and sorption from factors such as UV, temperature, water, biota, bacteria and other potential factors. A better understanding of the fate of TWP and related metals in different soils would increase our understanding of differences between studies and better explain the lack of correlation between TWP and metals in this present study.

4.5. Limitations of the present work

A potential weakness with this study is the sampling method. In this study, the upper 10 and 20 cm layer of the soil was sampled for analysis, however it was sampled with a shovel. In contrast, using a soil corer is potentially more efficient in soil sampling to avoid mixing of the soil layers and to determine the exact depth of the soil sampled. Thus, the sampling method used herein could, at least partially, explain lack of correlation between contaminant concentrations and soil depth. For future studies, it would be important to consider the sampling set-up in more detail in order to investigate the true impact of soil depths for the different soil types and how TWPs and road metals are transported through the soil column.

5. Conclusions

The results from this study demonstrated that roadside soils from low traffic areas could contain high levels of TWPs, enabling us to reject our initial hypothesis that the levels of TWPs in low traffic areas would be lower compared to high traffic areas. Traffic density did not explain the variation of TWPs in this study. This could potentially be linked to the accumulation of TWPs in the soil over time, the lack of sophisticated drainage systems along these roads which increases the runoff flow into the roadsides, as well as the presence of natural soils retaining the TWPs compared to the constructed soil systems along highways and urban areas. Traffic speed did, on the other hand, explain some of the observed variation of TWP, although a much lower correlation than previously found for other road samples such as road-side snow. This could potentially be explained by the low variation in traffic speed between sites, as well as the lack of data on real driving speed versus speed limit on rural roads. Other variables such as the distance from the road, soil type, soil depth and soil profile did not have any significant impact on the TWP concentrations in this study, suggesting that the TWP distribution at these low traffic rural sites are more evenly distributed within the first 6 m of the roadside compared to urban and highway roads from previous studies. The study did not find any correlation between the TWPs and metals either. Thus, both the second and the third hypotheses for TWPs could be rejected. For the road-related metals, traffic density and the level of organic matter in the soil were found to have the highest impact on the metal variation, however, also soil types, soil profile, traffic speed, depth and the percentage of heavy vehicles did have significant impact on the variation of metals between different sites, thus supporting the third hypothesis with respect to metals.

This study has demonstrated that analyzing TWPs in soil with PYR-GC/MS without pretreatment is possible, however, it is important to investigate potential impact on the selected markers for TWPs. Different types of pretreatment should be considered in future studies of solid samples such as soils and sediments, especially if the levels of TWPs are expected to be low.

CRediT authorship contribution statement

Elisabeth S. Rødland: Conceptualization, Methodology, Investigation, Formal analysis, Writing – original draft, Writing – review &

editing, Visualization. **Lene S. Heier:** Conceptualization, Writing – review & editing. **Ole Christian Lind:** Conceptualization, Writing – review & editing. **Sondre Meland:** Conceptualization, Writing – review & editing, Project administration.

Declaration of competing interest

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

Data availability

Data will be made available on request.

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All graphics are made using free vectors from Freepik and Macrovector.

Appendix A. Supplementary data

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