



# Bioaccessibility and size distribution of metals in road dust and roadside soils along a peri-urban transect



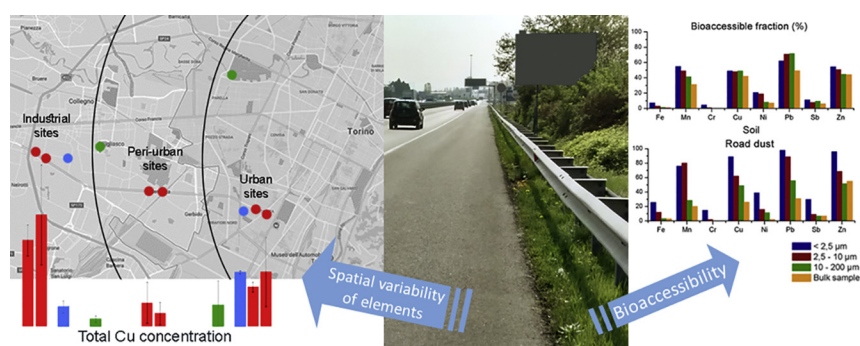
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## HIGHLIGHTS

- Metal concentrations are higher than legislative limits for Cu, Cr, Ni, Pb and Zn.
- Cu, Sb and Pb have the highest concentration in 2.5–10  $\mu\text{m}$  fraction, Zn in  $<2.5$   $\mu\text{m}$ .
- Pb and Zn are the most bioaccessible metals, followed by  $\text{Cu} \approx \text{Mn} > \text{Ni} \approx \text{Sb} > \text{Fe} \approx \text{Ni} \approx \text{Cr}$ .
- In road dust, above 90% of Zn, Pb and Cu is bioaccessible in the  $<2.5$   $\mu\text{m}$  fraction.
- Traffic is the major source of Cu, Sb, Zn bioaccessible fraction of Fe and Mn.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Road dust (RD), together with surface soils, is recognized as one of the main sinks of pollutants in urban environments. Over the last years, many studies have focused on total and bioaccessible concentrations while few have assessed the bioaccessibility of size-fractionated elements in RD. Therefore, the distribution and bioaccessibility of Fe, Mn, Cd, Cr, Cu, Ni, Pb, Sb and Zn in size fractions of RD and roadside soils ( $<2.5$   $\mu\text{m}$ , 2.5–10  $\mu\text{m}$  and 10–200  $\mu\text{m}$ ) have been studied using *aqua regia* extraction and the Simple Bioaccessibility Extraction Test.

Concentrations of metals in soils are higher than legislative limits for Cu, Cr, Ni, Pb and Zn. Fine fractions appear enriched in Fe, Mn, Cu, Pb, Sb and Zn, and 2.5–10  $\mu\text{m}$  particles are the most enriched. In RD, Cu, Pb, Sb and Zn derive primarily from non-exhaust sources, while Zn is found in greater concentrations in the  $<2.5$   $\mu\text{m}$  fraction, where it most likely has an industrial origin.

Elemental distribution across soils is dependent on land use, with Zn, Ni, Cu and Pb being present in higher concentrations at traffic sites. In addition, Fe, Ni and Cr feature greater bioaccessibility in the two finer fractions, while anthropic metals (Cu, Pb, Sb and Zn) do not.

In RD, only Zn has significantly higher bioaccessibility at traffic sites compared to background, and the finest particles are always the most bioaccessible;  $>90\%$  of Pb, Zn and Cu is bioaccessible in the  $<2.5$   $\mu\text{m}$  fraction, while for Mn, Ni, Sb, Fe and Cr, values vary from 76% to 5%. In the 2.5–10  $\mu\text{m}$  fraction, the values were 89% for Pb, 67% for Zn and 60% for Cu. These results make the evaluation of the bioaccessibility of size-fractionated particles appear to be a necessity for correct estimation of risk in urban areas.

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## 1. Introduction

In Europe, urban areas host three-quarters of the population. However, urbanization is still ongoing and, nowadays, peri-urban spaces

are increasing at a much faster rate than traditional core cities (European Commission, 2011). One of the consequences of the expansion of urban areas on the environment is soil and air pollution. This is currently one of the primary causes of concern for human health (Lim et al., 2012).

Road dust (RD) is at the interface between the soil and atmosphere, being a complex environmental medium originating from both natural and anthropogenic processes. Major sources include particles from soil materials, vehicle exhaust and non-exhaust emissions, atmospheric deposition and industrial activities (Thorpe and Harrison, 2008; Pant and Harrison, 2013). Together with surface soils, RD is a source and a sink of various pollutants in urban environments (Biasioli et al., 2006; Christoforidis and Stamatis, 2009). Metals are one of the most problematic as they are potentially toxic, persistent in the environment and often present at high concentrations.

RD and soil particles can be easily re-suspended by vehicles or wind, resulting in an important source of atmospheric particulate matter (PM) (Amato et al., 2009; Pant and Harrison, 2013). In recent years, research has focused on RD characterization and source apportionment of mineral PM (Aimar et al., 2012; Fujiwara et al., 2011; Kumar et al., 2013; Putaud et al., 2010). Concerns over RD contributions are growing based on the lack of legislation pertaining to all non-exhaust sources (Amato et al., 2014; Kousoulidou et al., 2008; Querol et al., 2004). In fact, with the continuous reduction of exhaust emissions, non-exhaust particles will increase their contribution to the total vehicle-generated particulate matter and may become dominant over the coming years in terms of both emissions and contributions to air quality (Denier van der Gon et al., 2013).

In city environments, metals in soil and RD can exert their toxicity through entering the human body via inhalation, dermal contact and ingestion, with ingestion being the most likely pathway for RD and soil fine particles (for example,  $<50\ \mu\text{m}$ ) (Siciliano et al., 2009; Ruby and Lowney, 2012). Shi et al. (2011) calculated that, for children, ingestion contributed 97.5% and 81.7% to total exposure doses of non-carcinogenic and carcinogenic elements, and that the contribution rates for adult were 91.7% and 52.9%, respectively. Kong et al. (2012) computed that for  $<2.5\ \mu\text{m}$  and  $<10\ \mu\text{m}$  particles of RD, non-carcinogenic risks based on exposure to metals were two orders of magnitude higher for ingestion than for inhalation.

While a number of studies on RD have concentrated on the elemental composition of samples (Amato et al., 2011; Gunawardana et al., 2012; McKenzie et al., 2008; Varrica et al., 2003), few have considered that the total or pseudo-total content does not necessarily represent the most dangerous chemical fraction of the metal. Analogously, whole soil content would not serve as a reliable assessment of the transfer potential of particles, as the coarser fractions are rarely removed (Bi et al., 2015; Paustenbach, 2000; Turner and Ip, 2007). Therefore, an evaluation of metal bioaccessibility is necessary to better evaluate human health risks to metals in order to obtain a more realistic ingestion exposure (Hu et al., 2011; Yu et al., 2014).

As there is a general need to better define the hazards and the health relevance of RD and non-exhaust PM sources (Denier van der Gon et al., 2013; Guney and Zagury, 2016), the aim of this study was to assess the total and bioaccessible concentrations of metals in size-resolved fractions of RD and roadside soils in Turin (Italy).

## 2. Experimental

### 2.1. Study area

The metropolitan area of Turin (Italy) ( $45^{\circ}04' \text{ N}$ ;  $7^{\circ}41' \text{ E}$ ) has a population of 1.7 million inhabitants and is the fourth most populated metropolitan area in Italy. It is characterized by a very high volume of vehicular traffic and has a long industrial history, mainly because of the automotive and metallurgical industries. The city was built and extended at the bottom of an alluvial plain in the Po valley, and is shielded

by hills to the east and by the Alps to the north and west. This setting causes PM levels to often exceed EU limits not only in Turin, but also in many other urban areas within the Po valley (Eeftens et al., 2012; Padoan et al., 2016), and these atmospheric emissions can result in a major source of diffuse metal soil contamination (Biasioli and Ajmone-Marsan, 2007).

10 sampling sites were selected along a peri-urban transect (7.5 km), starting near the ring road and reaching the city center along an approximately straight line (Fig. 1). Six sites were on a main road (Corso Allamano) and can be separated into three groups: samples influenced by industrial activities (A1, A2), sites located in a residential area (S1, S2) and sites surrounded by agricultural fields (A3, A4). In addition, each group included a sampling spot just before a traffic light or a roundabout (braking sites: A1, A4, S1) and one at least 200 m after (acceleration sites: A2, A3, S2). All sites have a similar traffic intensity, varying between 13,000 (S1, S2) and 9500 (A3, A4) vehicles  $\text{day}^{-1}$  (data provided by 5T s.r.l.). The industrial area and the residential area counted with one additional sampling site each, located on a secondary road, with a mean traffic of 1000 and 3000 vehicles  $\text{day}^{-1}$ , respectively, to appreciate the impact of low traffic volumes. Two urban background sites were additionally sampled - a paved road within the largest park of the city (PE) and within the university campus (AG), as remotely as possible from direct traffic sources but still in the urban area. Soil samples were collected as close as possible to the RD sampling point.

At each site, samples were gathered during three different seasons: April 2015, August 2015 and February 2016, after, at least, seven dry days. In order to minimize time variability between sampling sites, during each season, all samples were collected on the same day.

### 2.2. Sampling methodology

At each site, three subsamples of RD were collected,  $1\ \text{m}^2$  each, centered within the right-most active lane using a polyethylene brush (Acosta et al., 2011; Li et al., 2013; Sutherland et al., 2012; Varrica et al., 2003). This method could result in a loss of fine particles, so special care was taken to avoid re-suspension during the sampling by sweeping directly into a plastic bag. The collected sample quantities varied from 20 to 200 g.

Soil samples were taken from a 0–5 cm depth at all locations and within 2 m from the edge of the road. At each site, three sub-samples were collected at a distance of 1 m between each other, and the sub-samples were homogenized into one composite sample for further analysis. The brush and shovel were washed with a mild acidic solution and deionized water before and between samplings. All samples were air dried in the laboratory at room temperature and passed through a plastic 2-mm sieve prior to laboratory analyses.

### 2.3. Analytical procedures

All samples were analyzed for pH (1:2.5, soil:water), total carbon (TC) and nitrogen (TN) (CE Instruments, NA2100 Elemental Analyzer, ISO 10694) and carbonates (volumetric method, ISO 10693). The particle-size distribution (PSD) was measured via the hydrometer method (Colombo and Miano, 2015; Gee and Bauder, 1986).

RD and soil samples were partitioned into four size fractions ( $<2.5$ , 2.5–10, 10–200 and 200–2000  $\mu\text{m}$ ). Samples were dispersed with Na-hexametaphosphate and resuspended, with the  $<2.5\ \mu\text{m}$  fraction separated by centrifugation (ALC-4227R) while the remaining fractions were separated by repeated sedimentation and decanting (Ajmone-Marsan et al., 2008); the process was repeated until the supernatant was clear. Following separation, the fractions were dried prior to analysis.

An *aqua regia* (HCl/HNO<sub>3</sub>, 3:1 v/v) extraction was performed with microwave digestion of 0.5 g of sample (Milestone Ethos D, ISO 11466). Extractable (pseudo-total) content of Fe, Mn, Cd, Cr, Cu, Ni, Pb and Zn were determined in bulk samples (ground to pass through a

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