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Preliminary assessment of road dust from Portuguese motorways: chemical profile, health risks, and ecotoxicological screening

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Abstract

Among non-exhaust emissions, road dust resuspension represents a rather important contribution to particulate matter in urban areas. This study aimed to achieve a chemical characterisation of road dust particulate matter (PM_{10}) on two motorway sections, one rural and one urban, and to explore the related health and ecotoxicological risks. Measured PM_{10} dust loadings reached very low levels (0.66–1.49 mg m^{-2}) compared to equivalent studies in other road environments in Portugal and other countries. Emission factors ranged from 33 to 62 mg veh^{-1} km^{-1}. The carbonaceous content represented 14% of the total PM_{10} mass, whereas the highest contribution to the mass was given by mineral matter. Elements such as Si, Al, Ca, Fe and K accounted for almost three quarters of the total element mass for all samples, whilst Cu and Zn, mostly associated with brake and tyre wear, were the most enriched elements in relation to the soil composition. Nonetheless, Ti and Zr presented the highest non-carcinogenic risks for human health. Despite the low amounts of particulate matter in the aqueous solution, the ecotoxicological screening with the *Aliivibrio fisheri* bioluminescence inhibition bioassay allowed to classify the samples as toxic.

Keywords Road dust · Motorways · Heavy metals · Health risks · Ecotoxicity

Introduction

Air pollution from multiple sources is a matter of concern worldwide, held responsible for 7 million deaths globally per year (WHO 2016). The most targeted pollutants are PM_{10} and $PM_{2.5}$, due to their demonstrated effects on human health. Airborne particles are indeed responsible for a variety of health dysfunctions and pathologies, mostly related to heart, cardiovascular and respiratory diseases, as well as neurodegenerative and perinatal disorders (Chen et al. 2017; Sharma et al. 2020; Yin et al. 2020). According to a data compilation by the World Health Organisation, only 16% of the assessed population is exposed to PM_{10} or $PM_{2.5}$ annual mean levels below the air quality guidelines (AQG) (WHO 2016). Besides, the European directives on air quality are being revised and current limits for single pollutants will be foreseeably reduced to align with WHO 2021 guidelines. Among a broad variety of sources, road traffic constitutes the biggest contributor to ambient particulate matter, accounting for 25% globally.

The external costs of air pollution on life quality are especially relevant in urban centres, where population concentrates relentlessly and where multiple sources of airborne particulate matter lead to an increased human exposure. Transport is often targeted as one of the top relevant sources of pollutants (Pant and Harrison 2013), whereas urban sprawl and different urbanisation dynamics create new traffic patterns with often unknown outcomes. Nonetheless, road environments are not only areas where

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source generation but also a channel of distribution of pollutants in surrounding environments. Road dust contamination with toxic components, such as heavy metals and organic compounds (e.g., polycyclic aromatic hydrocarbons, PAHs), may trespass to nearby soils via different physical and chemical mechanisms, it may also be washed out by surface runoff, or may be conveyed to other anthropised environments (Pagotto et al. 2001; Polukarova et al. 2020; Vlasov et al. 2022; Vlasov et al. 2021).

Different and sometimes opposite factors have a direct effect on traffic intensity of road infrastructures. On the one side, a generalised economic wealth leads to higher circulation of people and goods on road infrastructures, on the other side, the uprise of cleaner technologies applied to vehicles exhaust systems, including catalytic converters, as well as electric engines, provoked a sharp decrease of emissions from the transport sector (Amato et al. 2014). Consequently, exhaust emissions keep diminishing whereas the non-tailpipe component, also known as non-exhaust emissions (NEE), are expected to outdo them, at least in the OECD countries (Rexeis and Hausberger 2009; Woodburn et al. 2022). The current booming of electrical and hybrid vehicles and the new legal requirements that progressively push towards the phasing-out of combustion vehicles will certainly increase the interest on the other vehicle-related pollution mechanisms. The new EURO-7 emission regulation will be the first to include standards for brakes, as well as tyres, as emission sources for microplastics.

NEE embrace a rather various set of sources that are related to the vehicle itself and to their interactions with the surrounding environment. It includes tyre wear and road abrasion, intrinsically related to the car-pavement interface, brake wear, strictly depending on both driving conditions and brake pad and brake disc compositions, and road dust resuspension. The relevance of NEE from motor vehicles and, more specifically, of vehicle-resuspended dust, should apply to all geographical contexts, but it becomes even more influential in regions were warm and dry weather enhance the capacity of particulate matter to bounce and circulate, in a balance of deposition and resuspension mechanisms (Amato et al. 2012a). In the last decade, evidence have been indicating a growing interest in various components of NEE, such as road dust, targeting geographical distribution, chemical profiling, geographical distribution sources and potential health hazards (Casotti Rienda and Alves 2021; Denby et al. 2018). On top of it, various studies showed that road dust (RD) itself, also described as road dust resuspension, accounts for up to 59% share of NEE (European Environmental Agency 2019; Pant and Harrison 2013).

Road dust mostly consists of a major mineral fraction from soil-related suspended materials (Gunawardana et al. 2012), with a big contribution from quartz minerals, whereas the remaining part is constituted by clay-forming minerals, organic matter from biogenic origins, a vast array of components from vehicle-related emissions (tyre and brake wear, exhaust emissions, abrasion and degradation of car frame and engine components) and a further contribution from road materials (road paint, anticorrosive coating of guardrails, etc.). The release of pollutants from road materials due to vehicle-induced turbulence depends also on the characteristics of the road surface. The behaviour of concrete asphalt (made of bitumen and filler – sand or stone dust) are indeed different during the contact with vehicle tyre, since driving on a concrete surface entails higher emissions than an asphalt surface (Duong and Lee 2011).

A broad variety of studies have shown interest in road dust processes, physical and chemical aspects, geographical distribution and potential health effects (Casotti Rienda and Alves 2021). Most of them focused on urban areas and, more specifically, active lanes of streets, avenues, ring roads and kerbsides. The attention to peri-urban road environments is lower and to interurban high-speed road infrastructures, even less. A dozen of studies focusing on motorways were scrutinised to evaluate the current state of the art. Synonyms of motorway and related words were used as keywords: highway, orbital motorway, ring road, expressway, freeway. Other aspects considered during literature consultation were sampling methodology (deployed device), particle size, geographical context, and scope of the study.

Sampling road dust on motorway lanes had already been practiced with the same device used for this study. One study refers to Birmingham (UK) (Pant et al. 2015), whereas the second describes a rather trafficked ring road with heavy traffic in the urban area of Barcelona (Amato et al. 2012a). Examples of various studies regarding trafficked sections of urban motorways will be presented hereafter. Quantitative results, however, should be cautiously compared, since they could noticeably be influenced by surrounding sources, either traffic or other anthropogenic activities. Other studies on orbital motorways available in the literature were based on various sampling methodologies, therefore targeting various size fractions. Al-Shidi et al. (2020) collected road dust in a motorway of Muscat (Oman) with a vacuum cleaner, whereas Aryal et al. (2017) opted for a PVC brush and dustpan to collect dust in the orbital motorway of Sydney (Australia). A rather different approach was instead chosen by Adamiec et al. (2016), who collected dust of various sizes in the A-4 Katowice-Chorzów Batory motorway (Poland), as far as possible from residential areas, as well as in a nearby mountain road, allegedly unpolluted, to perform a comparison with background. Similarly, Faiz et al. (2009) employed a vacuum dust collector in the Islamabad Expressway, one of the busiest roads of the capital city of Pakistan. The same researchers also collected soil samples with a depth of 10 cm by the scraper plate method in undisturbed green land areas. A couple of more studies targeted related topics, such as the accumulation of metals in roadside soils in a highway environment in the USA (Turer et al. 2001), solid and metal element distributions in urban highway stormwater (Sansalone and Buchberger 1997) and metal element concentrations immediately outside the kerb of a motorway of Istambul, Türkiye, in comparison to other sites 0.5-1 km m away, including soils (Sezgin et al. 2004).

This study intends to quantify the PM_{10} dust loadings and the related emission factors from various motorway sections in northern Portugal. The chemical analysis focused on elemental composition, including the use of enrichment factors to establish the contamination patterns of specific elements. Moreover, the health risks associated with the chemical composition of road dust were estimated with long standing methodologies by the United States Environmental Protection Agency, whereas the deployment of a bioassay with *Aliivibrio fisheri* bioluminescent bacteria drew the attention on the potential ecological risks of the mixed composition of small loading of dust on the environment.

Materials and methods

Sampling

The toll road chosen for sampling was the A-29 motorway that connects Porto Metropolitan Area with Aveiro, stretching on 53 km, mostly on a 4-lane configuration, 2 lanes per each direction. The first three-quarters of the motorway are in a rather rural or suburban context, frequently sided by moderately anthropised lands, devoted to agriculture (smallholdings), grasslands and forest, whereas the surrounding cities are heavily industrialised and host the biggest manufacturing and chemical poles of the region. The last kilometres, instead, fall within the suburban area of Vila Nova de Gaia, southern side of the Metropolitan Area of Porto and third most populated city of Portugal (304 thousand inhabitants). The road is used by both light-duty and heavy-duty vehicles. Samples were collected in both areas, namely in the rural stretch within the municipality of Ovar and in the suburban stretch of the motorway, with the municipality of Vila Nova de Gaia, whereas different types of pavements were chosen: older and newer asphalt.

As per previous studies (Adamiec et al. 2016), sampling was undertaken by the end of summer period, to avoid the impact of non-traffic related sources such as biomass burning for domestic heating, which is very common in colder months, and that could be found in deposited dust. Other studies have demonstrated clear seasonal variations of dust loadings as well as chemical composition between winter and summer months (Gulia et al. 2019; Han et al. 2007; Wang et al. 2022), mostly related to the washout effect of rain and to higher humidity at the level of the pavement surface (Amato et al. 2012b).

Sampling was carried out with a field resuspension chamber developed to capture road dust with PM_{10} particle size. The equipment was first used by Amato et al. (2009) but it has been extensively tested in various road environments, from urban centres, ring roads, to peri-urban locations and parking lots (Alves et al. 2020; Amato et al. 2011; Casotti Rienda et al. 2023a). More specifically, the device consists of a vacuum pump that suctions air at a flow rate of 25 l min^{-1}, with the help of a rotary vane pump. The air flow continues via an acrylic resuspension chamber to allow coarse particles to deposit. The remaining particles above 10 µm are retained by a stainless-steel elutriation filter. Size-selective collection of particulate matter happens on either 47 mm quartz fibre filters (Pallflex®) or 47 mm Teflon filters, depending on the use to which they will be dedicated in laboratory proceedings. In general, each filter corresponds to 1 m^2 of pavement, but the sampled area may vary according to the total amount of dust present on the road surface. A total of 8 samples was obtained.

Before sampling, quartz fibre filters were calcinated at 500°C for 6 h to guarantee the removal of any potential organic contaminants. Both quartz and Teflon filters were left under controlled temperature and humidity conditions (20°C, 50% relative humidity) allowing them to reach the necessary stability for weighing on a Radwag MYA 5/4Y/F1 microbalance (readability 1 µg). The process followed the European Standard EN 12341 (Ambient air - Standard gravimetric measurement method for the determination of the PM_{10} or $PM_{2.5}$ mass concentration of suspended particulate matter). After sampling and weighing for gravimetric determination, all filters were kept in a freezer at -20°C until further processing.

Analytical methodologies

The quantification of organic (OC) and elemental carbon (EC) was performed on quartz filters by a thermal optical transmission technique. The instrument was developed at the University of Aveiro and it works by mimicking the temperature programme of the EUSAAR-2 protocol. Two 11 mm-diameter punches were analysed for each sample to quantify the carbon released. Nonetheless, the low amount of material on filter did not allow a clear distinction between OC and EC, whereas OC_{min} and TC were thoroughly estimated. Samples undergo a controlled heating process, with a step-by-step temperature program that allows the progressive volatilisation of carbon. The first half of the analysis runs in a non-oxidising nitrogen atmosphere, whilst the second one happens in a 4% oxygen-rich O_2/N_2 air flow, allowing the oxidation of EC in a catalytic oven at 600°C. A laser beam and a photodetector are deployed to measure light transmittance, and the lightening of the filter will then be used to distinguish the EC formed during pyrolysis of OC with the EC present in the sample. Thereafter a nondispersive infrared (NDIR) analyser (LI-COR Environmental, model LI-7000) measures the carbon converted into CO_2. Further details about the system functioning, as well as data treatment and interpretation can be found in Pio et al. (2011).

The remaining part of the quartz filters was utilised for a bioluminescence inhibition bioassay to assess the ecotoxicity of the samples in accordance with the ISO standard 21338:2010 (Water quality – Kinetic determination of the inhibitory effects of sediment, other solids and coloured samples on the light emission of *V. fischeri*/kinetic luminescent bacteria test). The protocol involves direct contact of the bacteria *Aliivibrio fischeri* (previously known as *Vibrio fischeri*) with an aqueous extract of the desired samples. With this propose, punches were ground in an agate mortar and then washed out to a pre-cleaned vial with 2 ml high-purity Milli-Q water. At the same time, a set of lyophilised bacteria was defrosted and rehydrated with reconstitution solution, then left to stabilised for 35 min at 12°C. For the direct contact test, 150 µl of the liquid extract were injected in a 96-well plate, then diluted two-fold eleven times, as well as one blank. Afterwards, the plate was placed in the instrument luminometer (Luminoscan), where 150 µl of the bacterial suspension contained in a 2% NaCl solution were added to each and every well, to guarantee the optimal growing living conditions for the microorganisms during the time of the reading (35 min). The direct contact test involved a first reading of bioluminescence 30 seconds after the bacterial suspension was added and a second one after 30 min of exposure. The software used for registering data was the Ascent Software provided by Aboatox Co. (Finland). Readings and subsequent calculations provided values of EC_{50} and EC_{20} (concentration that provoked 50% and 20% of bioluminescence inhibition, respectively), whereas the toxicity of the samples was expressed in toxic units (TU), calculated as $100/EC_{50}$. Further details on the methodology and its application to road dust studies can be found in Kováts et al. (2012) and Casotti Rienda et al. (2023a).

Teflon filters were dedicated to the analysis of the elemental composition through a particle induced X-ray emission technique (PIXE) performed in the laboratories of INFN (Florence, Italy). Both crustal elements and tracers of anthropogenic activities, such as Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, As, Se, Br, Rb, Sr, Y, Zr, Mo, Ba and Pb, were analysed (Lucarelli et al. 2018). Concentrations were then expressed as their respective oxidised form based on stoichiometric proportions.

Health risks associated with road dust

Metals present in road dust can enter in contact with the human body via three pathways: ingestion, inhalation from nose and mouth and dermal contact. Their effects can be either carcinogenic or non-carcinogenic. Literature available regarding road dust agrees on the adoption of the calculation method developed by the US Environmental Protection Agency (USEPA 2010), whereas reference doses are usually taken by similar studies on soils or drinking water (Faisal et al. 2021). The concept of dose itself entails the amount of substance available for interaction with metabolic processes once it has entered the outer boundary of the organism. The daily dose through ingestion (ADD_{ing}), dermal contact (ADD_{dermal}) and inhalation (ADD_{inh}) can be calculated as follows (Adimalla 2020; and references therein):

$$ADD_{ing} = \frac{(C \times IngR \times EF \times ED)}{(BW \times AT_{nc})} \times 10^{-6} \tag{3}$$

$$ADD_{drm} = \frac{(C \times SA \times SAF \times DA \times EF \times ED)}{(BW \times AT_{nc})} \times 10^{-6} \tag{4}$$

$$ADD_{inh} = \frac{(C \times InhR \times EF \times ED)}{(PEF \times BW \times AT_{nc})} \tag{5}$$

ADD may also be found in the literature as CDI, referring to the chronical daily intake. The description of the parameters for calculations are described in Table S1. The cumulative dose resulting from ingestion, inhalation and dermal contact of non-carcinogen metals were also calculated.

The chronic hazard quotient (HQ) was estimated for non-cancer risks, for each element and route, for children and adults, with values of RfD (the ratio of average daily intake to the reference dose) retrieved from the literature (Adimalla 2020; Ferreira-Baptista and De Miguel 2005):

$$HQ_{route} = \frac{ADD_{route}}{RfD_{route}} \tag{6}$$

$$HI = \sum HQ = HQ_{ing} + HQ_{drm} + HQ_{inh} \tag{7}$$

A health risk quotient below 1 is assumed to entail acceptably low risk (safe), otherwise it is considered to have potential health risks associated with overexposure. Besides, the carcinogenic risk (CR_n) represents the chances of an individual of developing any type of cancer during an average lifetime as a result of exposure to carcinogenic hazards. It was calculated as follows (Adimalla 2020) and references herein).

$$CR_n = ADD \times SF \tag{8}$$

where CR_n is the carcinogenic risk for a specific metal (Cr, Ni and Pb) and *SF* is the slope factor, which is an upper bound, approximating a 95% confidence limit, on the increased cancer risk from a lifetime exposure to a chemical. Due to the lack of slope factors for a large number of elements, it was not possible to extend the analysis to the whole set. The sum of CR values for each n single metal gives the total carcinogenic risk (TCR):

$$TCR = \sum CR_{n\_ing} + CR_{n\_drm} + CR_{n\_inh} \tag{9}$$

Values of TCR below 1×10^{-6} are considered negligible, values in the range between 1×10^{-4} and 1×10^{-6} are taken as tolerable and values above 1×10^{-4} are deemed to cause harm to human health, requiring the adoption of measures by the authorities (USEPA 2010).

Data treatment and indexes

Particulate emission factors (EFs) were estimated based on an empirical relationship by Amato et al. (2011):

$$\mathrm{EF} = 45.9 * \mathrm{DL10}^{0.81} \tag{1}$$

where DL10 represents dust loadings expressed in mg PM_{10} m^{-2}. EFs are expressed in mg veh^{-1} km^{-1}.

Moreover, with the aim of assessing the presence and the intensity of the anthropogenic contribution to elements, enrichment factors (EnF) were calculated:

$$EnF = \frac{\left(\frac{C_n}{C_{ref}}\right)_{sample}}{\left(\frac{C_n}{C_{ref}}\right)_{UCC}} \tag{2}$$

The concentration of an element is represented by C_n and the concentration of the reference crustal element, C_{ref}, normalised to the ratio of these elements in the average upper continental crust (UCC). In this study Al was used as the reference for normalisation (Alves et al. 2018; Cunha-Lopes et al. 2022). UCC average concentrations were retrieved from (Wedepohl 1995) since standard background values for soil and dust are not available for this region of Portugal for all elements. Five contamination categories, from minimal enrichment to extremely high enrichment, can be recognised (Yang et al. 2016).

Results and discussion

PM_{10} dust loadings and emission factors

With the aim of obtaining averaged road dust PM_{10} loadings (DL10), duplicate samples of both quartz and Teflon filters were considered for calculations. Samples from A-29 motorway in the rural stretch had an averaged DL10 of 0.66 ± 0.05 mg PM_{10} m^{-2}. As regards instead samples from A-29 motorway in the suburban stretch, one sub-sample had values twofold the other sub-samples and the difference could be attributed to local geographical specificities such as pavement inclination and the subsequent accumulation patterns on the lower ground, or to the pavement non-homogeneity. Therefore, averaged DL10 for the suburban stretch accounted for 1.49 ± 0.72 mg PM_{10} m^{-2}. The comparison with other sampling locations in Portugal shows that values of this study are generally in line with the ones obtained for asphalt paved roads in urban centres such as Porto (0.48 ± 0.39 mg PM_{10} m^{-2}) (Alves et al. 2018), Viana do Castelo (0.53–1.87 mg m^{-2}) (Casotti Rienda et al. 2023a) and Aveiro (0.98 and 2.24 mg PM_{10} m^{-2}) (Casotti Rienda et al. 2023b). Other European cities with much higher population and traffic had similar values: Paris (0.7–2.2 PM_{10} m^{-2}) (Amato et al. 2016) and Zurich (0.2 to 1.3 mg PM_{10} m^{-2}) (Amato et al. 2011).

As previously mentioned, studies regarding motorway environments are scarce and most of them focus on heavy metals and their pollution indexes. The only example of motorway where sampling was carried out with the same methodology and with comparable values is from Birmingham (UK), accounting for 3.78–21.8 mg PM_{10} m^{-2} (Pant et al. 2015). Some other studies focused on freeways and orbital roads, but sources appeared to be much stronger, and accumulation of dust is clearly visible in the DL10 obtained. For instance, samples from the ring-road of the city of Barcelona accounted for 12.8–73.7 mg PM_{10} m^{-2} (Amato et al. 2011). A ring-road integrated in such a metropolitan area usually suffers from heavy traffic, traffic jams and frequent use of brakes. The A-29 motorway object of the present study is an example of an open road infrastructure, with rare traffic congestion episodes and with rare construction obstacles that allow a rather efficient dispersion due to the strong and persistent westerly winds from the Atlantic Ocean.

Emission factors reflected the trend of dust loadings, since DL10 is part of the equation. On the one hand, the A-29 motorway in VN Gaia (suburban) had average EF of 66 mg veh^{-1} km^{-1}, in the range of 37–111 mg veh^{-1} km^{-1}. On the other hand, EF for the A-29 motorway in Ovar (rural) reached an average of 33 (31–36) mg veh^{-1} km^{-1}, similarly to the 33 mg veh^{-1} km^{-1}obtained in the urban road tunnel of Braga (Portugal) (Alves et al. 2018). Among the few emission factors available for motorways obtained with other sampling methodologies, we may find the 48 mg veh^{-1} km^{-1} of an interurban freeway in Reiden (Zurich, Switzerland) (Bukowiecki et al. 2010). Moreover, vertical profile concentrations calculated after sampling passive deposition at a kerbside location in the ring-road of Barcelona lead to estimated emission factors of 22.7 ± 14.2 mg veh^{-1} km^{-1} (Amato et al. 2012b).

Organic carbon (OC) represented 78.4–92.3% of total carbon, leaving a rather small share to elemental carbon (EC). Nonetheless, the carbonaceous content barely reached 14% of the filter PM_{10} mass for all samples. These values are in line with most studies carried out with the same methodology, yet higher than the 7.91 ± 4.80% of a major motorway in Birmingham (UK), the only example available in literature that also showcases OC/EC results (Pant et al. 2015). Further details can be found in Table 1.

In all samples, the sum of elements, expressed in their respective oxide species (Al_2O_3, MgO, Fe_2O_3, TiO_2, K_2O, etc.), represented the highest contribution to the total PM_{10} mass, with overwhelming mass fractions of 807–835 mg g^{-1} (80.7–83.5%). Values are prominently higher, 2-4 times, than results from active lanes of cities such as Porto (Alves et al. 2018), Lisbon (Cunha-Lopes et al. 2022), Bogotá (Ramírez et al. 2019) and Barranquilla (Ramírez et al. 2020). They are also higher than the mass fractions of 61.4 ± 8.6% obtained in Aveiro-Ílhavo (Casotti Rienda et al. 2023b), and the 70% abundance in Viana do Castelo (Alves et al. 2020), both in urban lanes. These results might be interpreted in a context of a lower contribution from exhaust particles, as well as a lower emission from tyre and brake wear, due to the smoother driving conditions on the motorway compared to urban roads. On top of that, very reduced dust loadings combined with a dominance of mineral components should indicate a stronger geogenic contribution. Typical crustal elements such as Si, Al, Ca, Fe and K (in this very order) accounted for almost three quarters of the total element mass for all samples (39.5, 19.6, 8.6, 7.3 and 2.1%, respectively). In samples from Aveiro and Ílhavo, those percentages were very similar, indicating an equivalent contribution from regional background dust sources. Nonetheless, in the nearby city of Porto, mass fractions (wt%) of those elements were much lower, even though the most abundant elements were the same (Alves et al. 2018). In this study, enrichment factors (EnF) for Si, Al, Ca, Fe and K were always below 2, which corresponds to the category of minimal enrichment.

The presence of calcium with high abundances, apart from a strong mineral contribution, might also be due to abrasion-led resuspension processes. High mass fractions were detected in road dust from very trafficked areas of Lisbon (Cunha-Lopes et al. 2022), where construction and demolition activities that release great amounts of Ca enriched dust are frequently registered. Besides, Ca has been reportedly considered an indicator of the abrasion of pavement materials, both pavement aggregates and bituminous bender (Fullová et al. 2017). On the contrary, the presence of Fe is usually attributed to both ferrous mineral dust and traffic sources (exhaust and non-exhaust), but mostly from brake wear that is emitted during the use of pads, but it is also accumulated and can be eased off from rims during movement (Amato et al. 2009; Grigoratos and Martini 2015; Hagino et al. 2016). In this campaign, Fe might be also ascribed to the non-exhaust source. Nonetheless, both elements showed enrichment factors in the lower level (minimal enrichment) (Table 2).

Among the well-established tracers for non-exhaust emissions in road dust, Cu (0.7 mg g^{-1} PM_{10}) and Zn (1.6 mg g^{-1} PM_{10}) were the most relevant in this study, showing very high and significant enrichments, respectively. The literature about road dust in motorways corroborated high concentrations of Zn (Adamiec et al. 2016) and Cu (Al-Shidi et al. 2020), far more abundant than other elements, yet lower than in other urban environments. Another study compared the samples from the urban motorway with a road tunnel in the same city, finding in the latter much higher element concentrations (Pant et al. 2015).

Samples were overall moderately enriched in other elements such as Cr, Pb, S and V. Their presence in road dust have been attributed to different sources. For many years Pb was attributed to vehicle exhaust, but after leaded fuels

Table 1 Organic carbon content in road dust samples collected in various Portuguese, European and southern American cities with the same methodology

Reference	City	OC/PM_{10}
Road dust studies in Portugal		
This study	A-29 Motorway (average)	9.38 ± 1.18 %
Alves et al. (2018)	Porto	7.14 ± 3.48%
Alves et al. (2020)	Viana do Castelo	5.56 ± 1.24%
Casotti Rienda et al. (2023a)	Indoor parking lots, Aveiro	13.8 – 30%
Casotti Rienda et al. (2023b)	Aveiro and Ílhavo	11.6 ± 2.87%
Cunha-Lopes et al. (2022)	Lisbon	10.4 ± 0.03%
Road dust studies in other countries		
Amato et al. (2011)	Barcelona (Spain)	11.7 ± 1.8%
	Girona (Spain)	10.9 ± 5.2%
	Zurich (Switzerland)	21.4 ± 12.5%
Pant et al. (2015)	Birmingham (UK)	7.91 ± 4.80 %
Ramírez et al. (2019)	Bogotá (Colombia)	13–29%

Table 2 PM_{10} mass fractions (wt %) of element oxides

Mass fractions of element oxides (μg g^{-1})						
Element	A-29 (Suburban)			A-29 (Rural)		
	Avg	Min	Max	Avg	Min	Max
Na	8,162	7,831	8,493	10,410	6,283	14,537
Mg	10,055	9,337	10,773	11,071	6,201	15,940
Al	212,694	202,450	222,938	180,180	108,647	251,713
Si	416,845	406,060	427,631	372,991	230,193	515,788
P	3,353	3,330	3,376	6,812	2,544	11,079
S	9,645	8,320	10,970	12,267	7,090	17,444
Cl	1,944	1,648	2,240	5,656	3,828	7,484
K	22,773	22,117	23,430	19,956	13,240	26,673
Ca	75,496	56,489	94,503	96,403	71,043	121,764
Ti	3,574	3,437	3,710	3,317	2,247	4,387
V	309	273	344	13,486	1,335	25,638
Cr	224	215	234	469	175	763
Mn	714	685	742	709	470	948
Fe	65,464	61,976	68,953	79,657	39,687	119,627
Ni	21	18	23	86	32	139
Cu	742	714	769	1,618	664	2,573
Zn	1,588	1,564	1,612	4,516	1,602	7,430
Se	9	8	10	41	13	69
Br	23	15	31	65	10	120
Rb	317	301	334	177	149	205
Sr	133	79	186	150	78	222
Y	58	39	77	354	33	676
Zr	266	220	312	442	205	678
Mo	138	135	141	752	134	1,370
Ba	499	331	666	897	273	1,520
Pb	162	141	183	143	102	183

(gasoline with PbO_4 addition) were phased out in most developed countries, Pb abundance has been decreasing. Nonetheless, lead was also used among other materials for wheel weight balancing, but it has been replaced by zinc weights, whereas it can still be released by tyre wear and by lubricating oil in use, apart from bearing wear (Beddows et al. 2016; Zhu et al. 2008). All around the world, there are evidences of Pb being still present in various road environments from diverse urban contexts (Acosta et al. 2011; Adamiec 2017; Zannoni et al. 2016), including urban motorways in Islamabad (Pakistan) (Faiz et al. 2009), Muscat (Oman) (Al-Shidi et al. 2020) and Istambul (Türkiye) (Sezgin et al. 2004), as well as in the interurban Katowice-Chorzów Batory motorway (Poland) (Adamiec et al. 2016). On its side, chromium has appeared in most of the aforementioned studies and its presence could be ascribed to non-exhaust sources, including degradation of vehicle components such as wrist pins and connecting rods. Another element typically related to vehicle NEE is titanium. In this study, Ti was present in all samples, in the range 2.2–4.4 mg g^{-1} PM_{10}, but its enrichment was minimal. In other studies, the presence of titanium was documented but not significant conclusions were drafted (Adamiec et al. 2016; Pant et al. 2015). However, in a Polish motorway, concentrations of Ti were found being 3 times higher in an interurban motorway than urban roads. The geo-accumulation index confirmed that samples were extremely polluted, way above the natural background (Adamiec et al. 2016).

Risk assessment

For all-samples, the non-carcinogenic risk score for both children and adults was calculated for a series of elements whose coefficients were available in the literature (see Tables S1 and S2). Results for those two categories must be presented separately due to the difference on their and respiratory systems. The non-carcinogenic health quotient (HQ) for children was systematically higher than the one for adults for the same exposure route, for all routes (ingestion, dermal contact, and inhalation), in alignment with other studies (Casotti Rienda et al. 2023b; Keshavarzi et al. 2015). Al, Fe, Ti and Zr, had the highest HQ_{ing}, HQ_{drm} and HQ_{inh} among elements for most samples (See Table S2). In order to establish a clear geographical variability of the non-carcinogenic risk level, the number of samples should be larger. Overall, HQ were above 1 for children, whereas they were well below 1 for adults, indicating a rather low non-carcinogenic risk (see Table S3). In the available literature the inhalation route is the most commonly addressed, but the calculations done for the other two routes showed consistent health quotient for the other two routes (ingestion and dermal contact). The calculation of the joint effects of all elements, namely the health index (HI), as sum of all individual HQs, would instead provide a different scenario. As concerns adults, the value would fall below the limit of 1, whereas for children the combination of elements would easily make the value fall within the category of potentially harmful.

The carcinogenic risk for heavy metals was instead calculated for a small number of elements, namely, Cr, Ni and Pb, and only for the inhalation route, since slope factor values for the other two routes are not available in the literature (Table 3). Whereas Ni and Pb fell within the safety limit of 10^{-6}, Cr was above the threshold of 10^{-4}, the acceptable target proposed by USEPA (Candeias et al. 2020), meaning that those concentrations may pose health concerns for humans. The summed carcinogenic risk would not be representative considering the reduced number of elements, for which data are available.

Ecotoxicological bioassay results

The bioluminescence inhibition bioassay with *Aliivibrio fisheri* had been previously applied to showcase potential toxicological effects of vehicle related aerosols (Aammi et al. 2017; Romano et al. 2020) and, more specifically, of road dust samples from parking lots (Casotti Rienda et al. 2023a) and from active lanes of urban roads of the Aveiro region (Casotti Rienda et al. 2023b).

In this study, all water-diluted sample extracts provoked toxicity, reaching values of EC_{50} of 39 and 24%, for the urban and suburban stretches, respectively. It means that 39 and 24% of the sample concentration is capable of creating

Table 3 Carcinogenic risk for selected elements, for adults (AD) and children (CH)

	A-29 motorway (Suburban)		A-29 motorway (Rural)	
	CR (CH)	CR (AD)	CR (CH)	CR (AD)
Cr	6.05E-04	3.41E-04	1.26E-03	7.13E-04
Ni	1.54E-07	8.71E-08	6.41E-07	3.61E-07
Pb	7.19E-08	4.06E-08	2.27E-08	1.28E-08

the ecotoxicological reaction under scrutiny (bacteria's death). Besides, the EC_{50} expressed in μg mL^{-1}, offered results of 74 and 20, respectively. As concerns the second batch of samples, the PM_{10} loadings dissolved in water was half compared with the first batch, leading to the conclusion that even small amounts of PM and PM-bound components, after a thorough dilution in aqueous means, can cause clear ecotoxicological effects.

Of potentially toxic elements, Al and Zn were detected in high concentrations (Al in the range of 202,450 and 222,938 μg g^{-1} for the suburban, 108,647 and 251,713 μg g^{-1} for the rural sites while Zn in the range of 1,564 and 1,612 μg g^{-1} for the suburban and 1,602 and 7,430 μg g^{-1} for the rural sites). Al toxicity has been scarcely addressed, in fact, an early study reported very low toxicity to *Aliivibrio fischeri* (George et al. 1995). On the contrary, the Vibrio assay has proven sensitivity to Zn (Fulladosa et al. 2005; Mohseni et al. 2018), or to Zn-containing mixtures (Kováts et al. 2022). Moreover, possible synergistic effects of heavy metals has been widely studied: Tsiridis et al. (2006), for example, detected synergism between Cu and Zn when assessing heavy metal toxicity on *Aliivibrio fischeri*. A further analysis should be accomplished with a higher number of samples, as well as a higher number of analysed components, including not only heavy metals but also organic compounds that are generally correlated with toxicity on bacteria (Casotti Rienda et al. 2023a; Kováts et al. 2021).

Conclusions

Loadings and chemical patterns of inhalable road dust were investigated for two motorway stretches of Northern Portugal, one in a suburban context and another one in a rural area. Locally obtained data are necessary for completing emission inventories and provide input values for source apportionment methodologies. Therefore, this study intends to complement previous data gathered from other urban environments so that a larger variety of road systems are covered, including dust loadings, emission factors and heavy metal concentrations, as well as potential risks for human health (carcinogenic and non-carcinogenic) and ecotoxicological effects on ecosystems. In the motorway environment traffic has specific driving patterns, usually at higher speeds and with a lower grade of braking and wheel movements, which is expected to lead to lower traffic-related emissions, mostly from brake and tyre wear.

Generally, PM_{10} from road dust of the motorway was predominantly composed of mineral material, encompassing traditional crustal elements and heavy metals. Compared to other studies of road dust in Portugal, PM_{10} dust loadings were overall lower than average, ranging from 0.66 to 1.49 mg m^{-2}. Consequently, emissions factors were also lower than average, from 33 to 62 mg veh^{-1} km^{-1}. The carbonaceous content represented 14% of the total PM_{10} mass, whereas the highest contribution to the mass was given by mineral matter, highly influenced by Si, Al, Ca, Fe and K. The chemical composition was able to showcase a varied composition with elements that are typically tracers of non-exhaust emissions, such as Cu and Zn, mostly, but also Mn and Cr, among others. Besides, the enrichment factors helped corroborating the vehicle-related sources.

The calculations based on the methodology by the US Environmental Protection Agency allowed estimating the non-carcinogenic and carcinogenic risks of selected elements as a consequence of different exposure routes (inhalation, ingestion, and dermal contact). Elements with relatively low concentrations, such as Ti, Rb and Zr, presented the highest non-carcinogenic risks for human health, whilst carcinogenic risks for both adults and children were estimated for Cr. Despite the low amounts of particulate matter in the aqueous solution used for the ecotoxicological screening with the *Aliivibrio fisheri* bioluminescence test, it was possible to prove the environmental toxicity of the chemical mix in the samples.

The present results should draw the attention of researchers and policy makers on all road environments, not only the most often discussed urban centres, but also most remote areas that may suffer from different source contributions or other processes of generation and transport of pollutants. Further research should be carried out on various motorway sections to cover other geographical conditions and traffic fleets and thus increase the representativeness of the data, taking into consideration how many motorways intersect with urban grids, where most socio-economical human activities have place.

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Data availability Not applicable.

Declarations

Ethical approval Research did not involve any experiment on humans.

Consent to participate Research did not involve any experiment on humans.

Consent for publication Research did not involve any experiment on humans.

Conflict of interest The authors declare no competing interests.

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