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PM_{10} Resuspension of Road Dust in Different Types of Parking Lots: Emissions, Chemical Characterisation and Ecotoxicity

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Abstract: The thoracic fraction of road dust (PM_{10}) was measured for the first time in Portugal in parking areas, both outdoors and indoors, with the aim of completing existing studies carried out in active lanes of various roads. An in situ resuspension chamber was used to collect a total of 23 samples in three parking areas of Aveiro, whilst the laboratory procedures included determination of carbonaceous content (OC and EC) by a thermo-optical technique, elemental composition by ICP-MS and ICP-OES after acid digestion, and the *Aliivribrio fisherii* bioluminescent bacteria ecotoxicity bioassay. Dust loadings (DL10) obtained were 18.5 ± 9.8 mg PM_{10} m^{-2}, in outdoor parking, and 1.8–23.7 mg PM_{10} m^{-2} for indoor parking, corresponding to emission factors of 476 and 75–589 mg veh^{-1} km^{-1}, respectively. OC represented 9–30 % of PM_{10} for the indoor parking areas. However, for the outdoor samples, the high iron oxide content jeopardised the OC-EC separation. In those samples, carbonates accounted for 10.0 ± 3.3% of the PM_{10} mass. The analysis of elemental components focused on major elements (Al, Ca, Fe, K, and Mg) as well as minor elements. The total mass fraction of element oxides accounted for 27.1% (outdoor) and 23.6–34.3% (indoor). Σ_{PAH} calculated for all parking areas accounted for 8.38–36.9 µg g^{-1} PM_{10}. The ecotoxicological bioassay showed that all aqueous solutions were toxic to bioluminescent bacteria, whereas no clear correlations could be made with specific component groups, with the exception of Σ_{PAH} and EC_{50}.

Keywords: nonexhaust emissions; road dust; parking lot; heavy metals; ecotoxicity; rare earth elements; PAH

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1. Introduction

Air pollution is responsible for 7 million deaths globally per year [1], and it is considered one of the top five causes of mortality [2] due to heart diseases, stroke, cancer, low-respiratory inflammation, and infections, among others [3,4], as well as neurodevelopmental and neurodegenerative diseases [5,6]. Furthermore, outdoor air pollution has been classified as carcinogenic to humans (Group 1) [7]. The dominant contribution of traffic emissions in urban areas and their effect on exceedances of limit values have been extensively demonstrated [8–10]. For that reason, vehicle-related pollution has been the focus of researchers and decision-makers for the last several decades, meanwhile the awareness of the potential effects of particulate matter on air quality, human health, and ecosystems is rising as long as sampling technology becomes more advanced and accurate. Generally, among the most targeted pollutants is particulate matter with aerodynamic diameter of lower than 10 µg (PM_{10}) or the smaller fractions ($PM_{2.5}$ and PM_1), as they are deemed to be a serious threat to human health [11].

Non-exhaust emissions (NEE) from road traffic occur irrespective of the type of vehicle and its mode of power, contributing to the total ambient PM burden associated with human morbidity and premature mortality (Casotti Rienda and Alves, 2021 [12] and references herein). In a context where exhaust emissions are progressively diminishing thanks to more stringent regulations alongside the technological updates achieved in the automotive industry towards net-zero emission technologies, it remains clear that the contribution of NEE has overtaken or will soon overtake exhaust emissions from motor vehicles, reaching up to 90% of road traffic emissions [13,14]. The whole amount of NEE particles is basically determined by four processes: brake, tyre and road surface wearing down, and road dust resuspension [15]. Their individual contribution and their chemical profile vary depending on multiple factors, such as brake type and conditions, vehicle weight, driving patterns, available dust on surfaces, and weather conditions (e.g., humidity and wind dispersion), among others. Nevertheless, uncertainties associated with NEE are still a burden to its proper inclusion in emission inventories. Among the NEE, road dust (RD) resuspension carries an intrinsic uncertainty due to ambiguous quantification methodologies and approaches.

There are no straightforward data about the contribution of RD to the total PM mass in an urban environment, but there are some exceptions that should draw attention to it. For instance, the kerbside PM_{10} increment in central London due to RD resuspension via tracer-derived calculations was estimated to be as much as 1.5 µg m^{-3} [16]. Another approach was followed by Weinbruch et al. [17], who estimated the PM_{10} kerbside increment in Ruhr (Germany) due to abrasion and resuspension using electron microscopy, attributing to abrasion and resuspension 0.8 and 4.2 µg m^{-3}, respectively. The contribution of RD to total primary emissions from all sources was estimated to be in the range of 16–54% (PM_{10}) and 9–56% ($PM_{2.5}$) in three Latin American capital cities (Mexico DF, Bogotá, and Santiago de Chile) [14]. Notwithstanding, the dynamics of RD deposition, removal, and distribution depend on a complex combination of factors. First of all, the moisture content of dust is a key parameter that determines its capacity to bounce after the passage of a vehicle on the pavement [18,19]. In countries with arid climates or marked dry seasons (i.e., the Mediterranean basin, central Asia and Middle East, and some regions of Northern America), the scarcity of precipitation and low humidity at the pavement enhance the resuspension emissions [20]. In those cases, RD becomes a compelling source of air pollution that may lead to exceedances of the daily limit values established in the legislation. Contrarily, regions with severe winters, such as the Nordic countries, are affected by different phenomena: RD is held responsible for the worsening of air quality indexes during springtime, when a mixture of pavement wear and antiskid road treatments (sand and salt) emerge after snowmelt, leading to peaks of coarse particles ($PM_{10-2.5}$) [21]. An extensive list of references to RD studies from all continents [12] shows how many approaches and sampling procedures might be applied. Research studies have indeed focused mostly on urban areas, where the population progressively concentrates, leading to both an increased number and strong emissions sources. RD is not only a source of air pollution per se, but it can be a carrier of pollutants from other sources, either wind-blown or already deposited on the road surface. Bezberdaya et al. [22] inferred that PM_{10} particles of RD from an industrial city were an important carrier of organic compounds, such as benzo(a)pyrene, whilst the contamination of surrounding urban soils indicated a long-term anthropogenic impact. Candeias et al. [23] detected heavy metals from agrochemical additives in the RD of a residential suburb. Noticeable traces of inorganic fertilisers and pesticides were instead detected in other urban road contexts [24,25]. Even though most studies involve active lanes of diverse road environments, sometimes with comparisons to rather less polluted backgrounds [12], the focus on parking lots, courtyards, and green areas of cities has been gaining some attention, mostly to explore distribution dynamics that not only rely on passing traffic but also on the anthropogenic contribution to road dust accumulation [26,27]. This reasoning led to drawing attention to parking areas that are deemed to work as a reservoir for road dust from various sources that are deposited on the pavement surface and, therefore, contain a vast array of pollutants. Parking lots in busy areas of a city, such as a city centre or university campus, are the interface between vehicle traffic and walking trips, becoming a potential hotspot for higher exposure for pedestrians and residents to vehicle-related indoor air pollution. Moreover, spaces allocated to car parking adjacent to active lanes, as well as dedicated outdoor and indoor parking areas (including garages and malls), are intrinsically integrated into the urban grid. The population will therefore spend time in or close to these environments on a daily basis, even without being a vehicle user. Another potentially affected group is the parking attendants, who face exposure to dust resuspension during the hours of their working day in those specific environments. It should be noted that there is so far limited available literature on road dust in parking areas, but the most recent articles show a vast array of organic pollutants and potential carcinogens in indoor [28] and outdoor parking lots [29]. Huang et al. [30] detected instead substituted p-phenylenediamines (PPDs) in RD from underground parking lots of 10 local malls.

With the aim of capturing different realities of an interface between vehicle traffic and pedestrian urbanised areas, this work proposes a comprehensive chemical characterisation of the thoracic fraction (PM_{10}) of RD samples from three parking lots in the city of Aveiro (Portugal): one outdoor parking in the university campus, one indoor multistorey parking of a residential building also open to the public, and one underground unloading area of a shopping mall.

2. Materials and Methods

2.1. Sampling Sites

Three different parking areas were chosen in the city of Aveiro, Portugal (Figure S1). The central parking facilities of the University of Aveiro campus, where hundreds of users park their cars at the beginning of the working day, constituted the first sampling site, where six samples were collected. It is an open-air space with old pavement and small debris and sand often visible. Located next to the main road of the campus, it is surrounded by university buildings but still affected by the dominant winds coming from the eastbound side of the ocean, which favours the dispersion of pollutants. It was identified as P1. The second one is a multistorey indoor parking area in the city centre of Aveiro, where both residents of the building and external users can park (Figure S2). It hosts 289 parking spaces on four levels, three of which are open, from 8 a.m. to 10 p.m. to the general public; meanwhile, residents have 24-h access through a card. Rush hour is in the afternoon from 3 to 6 p.m., where users park their cars while running errands in the city centre. The two entrance doors are located at different levels, on opposite sides of the building, but the other levels are located in the underground, leading to very poor air ventilation. Internal ramps are quite tight; therefore, vehicles must circulate at a very low speed, but with frequent braking and steering actions. Cleaning of the parking pavement is performed on a regular basis (monthly), but the effects of outdoor weather conditions on the indoor air quality are unknown, most likely negligible, except on rainy days when there is the possibility of vehicles carrying mud on their tyres. It was identified as P2, and the number of samples collected was 10 (before cleaning) and 2 (after cleaning). The reference $P2_{AVG}$ includes both sample types (averaged), except when it is explicitly indicated otherwise (i.e., $P2_{BC}$—before cleaning and $P2_{AC}$—after cleaning). The third and last sampling was instead carried out in the unloading area of the Forum Aveiro shopping centre, where trucks, vans, and other vehicles park to unload their products, such as textiles and food. Food delivery motorcycles also park there, and waste is stored and sorted out there as well (Figure S3). No information was provided on pavement cleaning or on the types and numbers of commercial vehicles that pass through it daily. It was identified as P3, and five samples were collected.

2.2. Sampling and Physico-Chemical Determinations

Road dust was collected with a RD sampler designed by the Spanish National Research Council (CSIC) [31], and thereafter employed in numerous campaigns, including other Portuguese cities such as Porto, Lisbon, Aveiro-Ílhavo, and Viana do Castelo [32–34]. The device consists of a vacuum-based system that captures dust deposited on the ground with an air flow of 25 ± 2.5 L min^{-1} via a resuspension chamber, followed by an elutriation filter in stainless steel that allows the thoracic fraction (PM_{10}) to continue flowing until being collected on a filter, which was alternatively of either quartz fibre or Teflon. For each site, road sediments were collected within a 1 m^2-shaped template to achieve a homogeneous and standardised sampling procedure, at least for 3 replicates, 2 quartz-fibres, and 1 Teflon filter per location, according to the desired purpose of the laboratory analysis to be performed afterwards. Contrarily to most examples of RD collection performed on active traffic lanes, the squared-templates were located on the lanes that allow cars to move within the parking area, where contamination from tyre- and brake-wear particles is expected on top of other sources. All samples were collected during dry periods to replicate the technical conditions previously applied for sampling in active lanes [33,35]. Once samples reached the laboratory after sampling, they were stored for 48–72 h under stable temperature and humidity conditions, to prepare for weighing and allow gravimetric determination of the particulate matter.

The carbonaceous content, organic (OC) and elemental carbon (EC), of particulate matter was determined by a thermal-optical transmission technique, where it is converted to CO_2 after a controlled combustion process with a sequence of temperature steps overtime equivalent to the EUSAAR-2 protocol. For each sample, two replicates of two 11-mm punches each from quartz filters were analysed, and values were averaged. On the other hand, samples from the outdoor parking were analysed both after undergoing acidification via exposure to HCl fumes in order to eliminate carbonates ($CO_3{}^{2-}$), and without any further treatment. Carbonates, which were then estimated by subtraction, were instead neglected for indoor samples. In fact, while the pavement of the outdoor parking was composed of pervious concrete and crushed gravel, that of the indoor infrastructures was paved with impermeable materials not composed of carbonates. Details of the full procedure can be found in Pio et al. [36], whereas the specific method for estimating carbonates was also applied to ambient air PM_{10} in Coimbra [9]. Organic matter (OM) was calculated by multiplying OC by a factor of 1.8 to compensate for the limitations of the thermal-optical analytical technique to quantify oxygen, sulphur, hydrogen, chlorine, and other atoms present in the organic fraction [37,38].

Polycyclic aromatic hydrocarbons (PAHs) were analysed in quartz filters by gas chromatography-mass spectrometry (GC-MS) according to the standard MSZ 1484-6:2003 (Testing of waters. Determination of polycyclic aromatic hydrocarbons (PAH) content by gas chromatographic-mass spectrometry). During the extraction process, the method of Vouitsis et al. [39] was followed with a minor modification. Each sample was extracted in a 30-mL vial of dichloromethane (DCM) for 24 h, and then evaporated to a final volume of 1 mL at 40 °C with nitrogen stream was left. The extract was kept at −20 °C until measurement. A GC-MS with a single quadrupole (models 6890 and 5973, respectively, from Agilent Technologies, USA) was employed. Further details of the operating conditions and specificities of the instrument can be found in Hubai et al. [40]. For quality assurance and quality control purposes, internal standards (p-terphenyl-d14 and 2-fluorobiphenyl from Restek Corporation, USA) and surrogate standards (acenaphthene-d10, benzo(a)pyrene-d12, chrysene-d12, naphthalene-d8, perylene-d12 and phenanthrene-d10 from Restek Corporation, USA) were diluted in GC grade solvents (Sigma Aldrich, USA). Before the measurements, a standard mixture was used to establish each of the target chemical compounds as a five-point calibration (concentration range: 0.5–5.0 ng mL^{-1}). The mean recovery based on the extraction of a certified standard solution was in the range of 60–120%. All data were corrected for the average value of the blanks. The limit of PAH detection (LOD) in the extracts was 0.001 µg L^{-1}. Analytical determinations were performed by courtesy of the laboratory of the ELGOSCAR-2000 Environmental Technology and Water Management Ltd. accredited by the National Accreditation Authority, registration number NAH-1-1278/2015.

Teflon filters were instead fully allocated to the analysis of major and trace elements by ICP-OES (Agilent Technologies, model 5110) and ICP-MS (Agilent Technologies, model 7900), respectively [41]. The digestion of samples was performed with a mixture of acids (2.5 mL HNO_3: 5 mL HF: 2.5 mL $HClO_4$) [42,43]. For quality control, the analytical procedure was validated with the analysis of NIST-1633b (fly ash) standard reference material, whereas the digestion procedure was validated with the certified reference material 1648a. For the elements under scrutiny, accuracy and precision ranged from 5 to 10%. Major and minor element concentrations were transformed into their respective mass concentrations of the most common oxides based on stoichiometric proportions.

2.3. Ecotoxicity

An ecotoxicity assessment was performed with the aim of exploring which samples provoke a higher mortality of bacteria, and therefore, which chemical composition has the most severe effects on health. The chosen procedure is a bioluminescence inhibition bioassay with *Vibrio fischeri* (recently renamed *Aliivibrio fischeri*) bacteria. The protocol follows 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), and it has been adapted for analysing aqueous extracts of particulate matter from various sources [44–47]. Some cm^2 of quartz filter samples, as well as blanks, were grinded with an agate mortar, diluted with 2 mL of high-purity Milli-Q water, and transferred to a precleaned vial. The bacteria kit provided by Aboatox Co. (Finland), lyophilised and frozen, was rehydrated with the reconstitution solution and stabilised for 35 min at 12 °C. A 96-well plate was used to place 150 µL of the liquid sample extract, and all the dilutions were performed two-fold eleven times, as well as one control. All wells were then topped up with 150 µL of 2% NaCl solution containing bacteria in suspension to ensure the correct living medium.

The direct contact test (also known as the flash test) consists of a double measurement of bioluminescence, the first one happening 30 s after exposure to the bacterial suspension, and the second one after 30 min. The Ascent software provided by the manufacturer, Aboatox Co. (Finland), registered the bioluminescence inhibition before and after exposure, filling in a template formatted to calculate the EC_{50} and EC_{20} parameters (the amount of sample that causes 50 and 20% of bioluminescence inhibition, respectively). Since samples from different environments had different dust loadings and, therefore, the sample area in the aqueous extract contained different PM_{10} loadings, the parameter EC_{50} was calculated as PM_{10} μL^{-1} in addition to %. Additionally, the concept of toxicity units (TU) was utilised ($TU = 100/EC_{50}$) to further evaluate the toxicity level of the extracts [48–50]. A similar approach was applied by Evangelopoulos et al. [51], where EC_{50} was calculated in ng m^{-3} since the target was the ecotoxicity of ambient air PM.

2.4. Data Processing

The whole data processing was performed with Microsoft Excel software and its analysis tools for advanced features, with the exception of the specific software for the ecotoxicological bioassay. Additionally, indexes and calculations were deployed based on previous literature. For instance, emission factors (EF) were calculated on the assumption that they were as valid for a parking lot as they were for active lanes. In fact, the very concept of EFs stands on the principle that vehicles resuspend the available dust present on the pavement under the effects of the vehicle speed and weight, as well as of environmental conditions, such as relative humidity and wind speed. Nevertheless, there are various formulas to reach a numeric value that are derived from different sampling procedures [12].

Amato et al. [52] validated previously collected data to reach an equation that simply ties the emission factor, expressed in mg veh^{-1} km^{-1}, to the dust loading as follows:

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

where DL10 expresses the dust loading, as PM$_{10}$, at the specific site (mg m^{-2}).

The geoaccumulation index was deployed to assess the contamination level of heavy metals (including rare earth elements):

$$I_{geo} = \log_2\left(\frac{C_n}{1.5 \, * \, B_n}\right) \tag{2}$$

where Bn (or Ccrust) represents the crustal concentration of a certain element, retrieved from Wedepohl [53], and Cn stands for the concentration of that element in road dust, expressed in mg kg^{-1}. Values representing the contamination levels are categorised, depending on the Igeo, into seven classes [12].

3. Results and Discussion

3.1. PM$_{10}$ Road Dust Loadings and Emission Factors

The interpretation of the obtained dust loadings of PM$_{10}$ (DL10) is a fundamental step to allow a thorough comparison among sampling sites, or even within the same site. In this campaign, the DL10 mean values for the three parking areas (outdoor, multi-storey indoor, and indoor/shopping mall) were 18.5 ± 9.8 (P1), 20.6 ± 11.1 (P2$_{\mathrm{AVG}}$), and 22.3 ± 6.7 mg PM$_{10}$ m^{-2} (P3), respectively (Table S1). Nonetheless, DL10 in P2 dropped from 23.7 ± 8.1 mg PM$_{10}$ m^{-2} before cleaning to as much as to 1.8 mg PM$_{10}$ m^{-2} after the monthly cleaning, 13 times less, confirming the results by several studies that demonstrated the ability of street washing to both reduce the available dust loadings [33] and the mobility of dust load deposited [20,54,55].

While comparing these DL10 values with previous literature, for the same sampling methodology applied for active lanes of asphalted roads, one could verify that most cities in Europe had sensibly lower dust loadings: 0.48 ± 0.39 mg PM$_{10}$ m^{-2} in Porto [32], 0.53–1.87 in Viana do Castelo [33], 0.7–2.2 in Paris [35], or even 6.27 ± 2.35 mg m^{-2} in Lisbon [34]. On the contrary, Vanegas et al. [56] detected in Bogotá DL10 levels between 1.8 and 45.7 mg m^{-2}, averaging 11.8 mg m^{-2}. A previous campaign of RD sampling in the urban area of Aveiro showed yet lower dust loadings of 0.33–6.57 mg m^{-2} (unpublished results).

It is worth mentioning that both driving conditions and local dispersion/concentration dynamics may vary greatly from an active lane with moderate traffic to a closed parking area (P2 and P3) with very specific vehicle movements (low speed, frequent steering) and mostly standing with engines off. The case of P3 might be a mixture of the aforementioned situations, but the pavement was in quite a bad state with large amounts of loose debris.

Emission factors were calculated for the three parking lots, showing mean values of 476 ± 207, 516 ± 246, and 534 ± 203 mg veh^{-1} km^{-1}, in P1, P2$_{\mathrm{AVG}}$, and P3, respectively, but a much lower value of 75 mg veh^{-1} km^{-1} was obtained in P2$_{\mathrm{AC}}$ and a slightly higher value of 589 ± 166 for P2$_{\mathrm{BC}}$ mg veh^{-1} km^{-1}. As regards southern-European cities, values from active lanes of various roads range from the 24.0 mg km^{-1} veh^{-1} of Milan [57] and the 27.0 mg km^{-1} veh^{-1} of Turin [58] to the 41.2 mg km^{-1} veh^{-1} of Viana do Castelo [33] to the 70.8 ± 56.9 mg km^{-1} veh^{-1} in Aveiro-Ílhavo (unpublished results). On the contrary, high emission factors, yet lower than the three parking lots, were calculated for cities in southern Spain (dry weather), averaging from 158 to 347 mg km^{-1} veh^{-1} [59]. Heavy-duty vehicles were instead responsible for EFs of 262 mg km^{-1} veh^{-1} in a street canyon of Zurich [60], still about half of those estimated in Aveiro for a mixed fleet.

On the one hand, both dust loadings and emission factors can be used as indicators of how polluted an urbanised area can be, but solely regarding the total mass. On the other hand, only a detailed chemical composition may establish whether one sample is more contaminated in terms of harmful compounds for human health [61–63].

Contrary to other studies, where it was possible to reach 70–90% of mass reconstruction [64,65], the identified components reached up to 50–65% of the total PM_{10} mass (Figure 1). The two main factors were elements expressed in their oxidised form (Al_2O_3, MgO, Fe_2O_3, TiO_2, K_2O, etc.), and carbonaceous content (OM and EC). With regard to the outdoor parking (P1), elemental oxides contributed 22.8% to the total mass, whereas carbonates (CO_3^{2-}) represented 6.0–12.0% of the total PM_{10} mass. However, the laser performance was disturbed by some metal content (mostly Fe), hindering a proper OC-EC separation and preventing it from obtaining a valid value for OC. In indoor parking lots, carbonaceous content (OM + EC) was similar to the sum of elemental oxides ($\Sigma_{\text{ELEMENT OXIDES}}$) for $P2_{AVG}$ (28 ± 3% over 29.1 ± 7.9%), but significantly higher for P3 (41.2 ± 3.7% over 23.6 ± 6.1%). The only exception were data from P2 after cleaning, in which case element oxides contributed to the total mass more than carbonaceous content: 34.3% ($\Sigma_{\text{ELEMENT OXIDES}}$) and 29.1% (OM + EC). Moreover, the contribution of metal oxides to the total PM_{10} mass in P2 after cleaning accounted for 34.3% (1.57 mg m^{-2}) versus a lower 27.8% (20.5 ± 7.02 mg m^{-2}). This suggests that the accumulation time needed for organic constituents might be longer than the time needed for the metal fraction to bulk up.

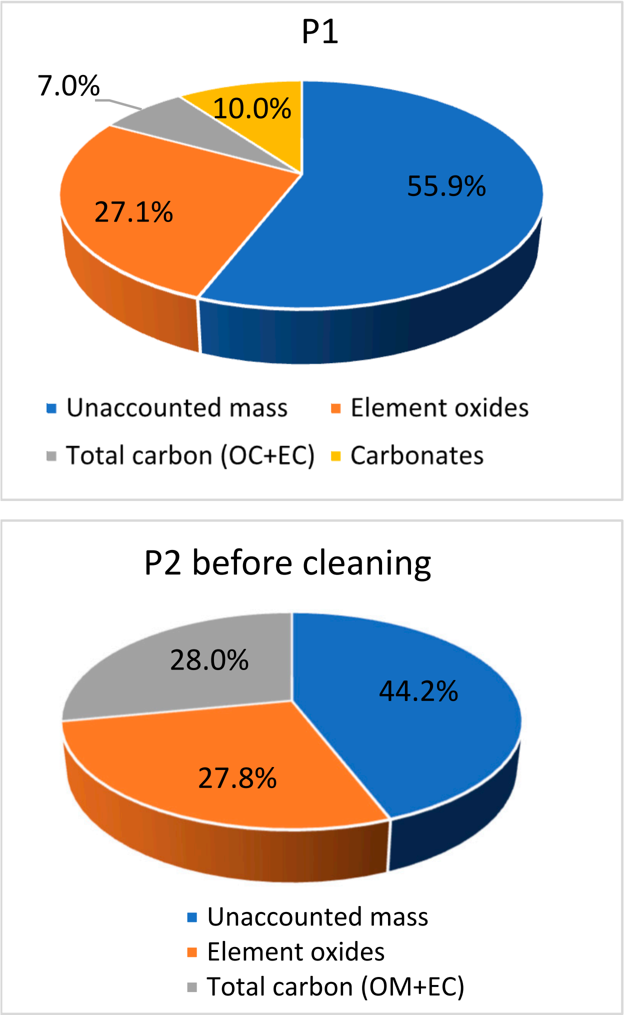

Figure 1. *Cont.*

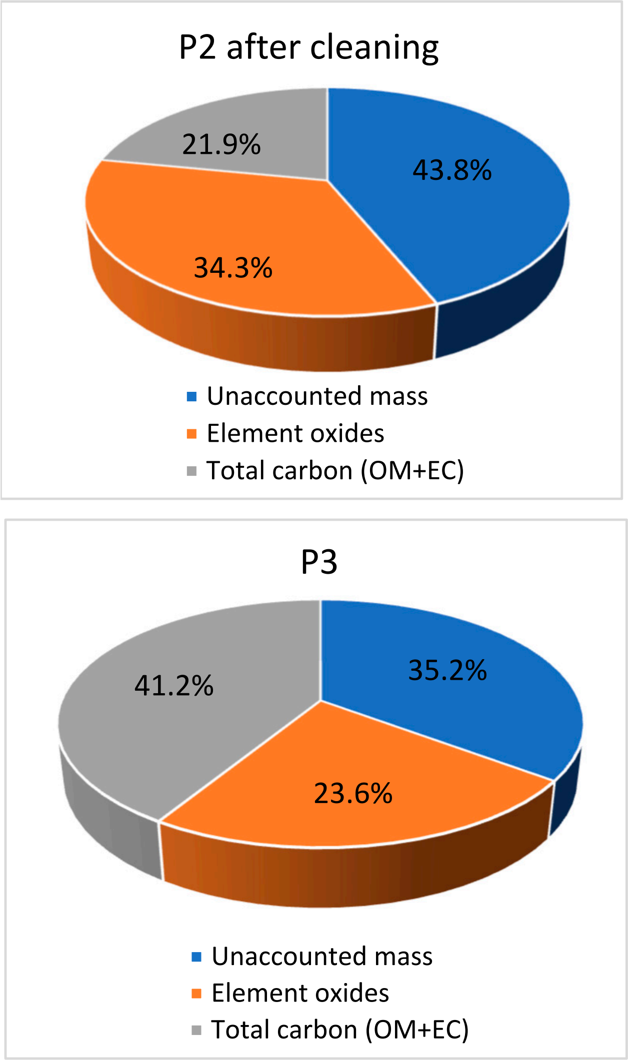

Figure 1. Mass reconstruction with main component groups.

Still, unidentified, or unquantified mass was a relevant portion that could not be completely justified, even though it could be ascribed to unanalysed constituents and to PM_{10}-bound water content [38,66,67]. The estimation of SiO_2 content can be carried out applying the formula that follows:

$$SiO_2 = 3 * Al_2O_3 \quad (3)$$

SiO_2, in fact, contributed to 4.4 ± 1.6% (P1), 9.9 ± 1.7% ($P2_{AVG}$), and 7.9 ± 2.4% (P3) of the PM_{10} mass. Moreover, SiO_2 content in $P2_{BC}$ and $P2_{AF}$ was very similar: 10 ± 2% and 9.8%, respectively. The reasons behind these differences in composition between indoor (P2 and P3) and outdoor (P1) parking are unclear. This compound is usually attributed to the abrasion and degradation of pavement, rich in crustal and crustal-like components, the tyre wear contribution, including amorphous silica (SiO_2) [68,69], and the progressive degradation and volatilisation of compounds from vehicle components (engine, fuels, lubricants, etc.) [70]. In this case, the higher mass fractions were detected in indoor parking with smoother and better conserved pavement, therefore the contribution of abrasion-related crustal materials must have been outperformed by tyre-related or vehicle-related components (tyre-pavement friction) but a thorough analysis of organic components should be performed to thoroughly disclose sources.

For indoor parking lots, $P2_{AVG}$ and P3, OC accounted for 13.8–30.0% and 15.1–20.6% of PM_{10}, respectively, markedly higher than Viana do Castelo (5.56 ± 1.24%) [33], Porto 7.14 ± 3.48% [32], and Lisbon (10.4 ± 0.03%) [34]. OC represented 9% of the PM_{10} mass in $P2_{AC}$. High vehicle emissions from both exhaust (engine combustion) and non-exhaust (mostly tyre and brake wear) should be responsible for these robust shares of organic carbon in the two locations, way higher than expected from the literature. EC, which partly shares vehicle sources with OC, is very representative in P3 (EC/PM_{10} 7.8–10.2%) and also both in $P2_{BC}$ and $P2_{AC}$ (EC/PM_{10} 10.8–15% and 5.7%, respectively). As concerns the outdoor parking facilities (P1), the high concentration of mineral elements (mostly Fe) might have disturbed the steadiness of laser acquisition during the thermal-optical carbon analysis, hindering a clear OC and EC distinction. Overall, total carbon (TC) accounted for 4.2–9.6% of the total PM_{10} mass, whereas carbonates contributed 6–13% (see Table 1).

Table 1. Carbon content in percentage and PM_{10} mass fractions.

Carbon Fraction	P1 (Campus UA)		$P2_{BC}$ (Indoor Multi-Storey)		$P2_{AF}$ (Indoor Multi-Storey)		P3 (Underground)	
OC (%)/ (mg g^{-1} PM_{10})	-	-	23.8 ± 6.4	132.7 ± 3.8	9.0	90	17.7 ± 2.8	176.9 ± 27.7
EC (%)/ (mg g^{-1} PM_{10})	-	-	13.3 ± 167	41.6 ± 3.8	5.7	57.1	9.3 ± 1.4	93.4 ± 13
TC (OC + EC) (%)/ (mg g^{-1} PM_{10})	7.0 ± 2.7	69.6 ± 26.9	30.1 ± 7.6	186.7 ± 34.3	15	147.1	27 ± 1.6	270.3 ± 15.6
Carbonates (%)/ (mg g^{-1} PM_{10})	10 ± 3.3	100.1 ± 32.6	-	-	-	-	-	-

Whereas samples from both outdoor and indoor parking showed marked but uneven contamination of major elements, samples from P2 regularly proved to be the most enriched in minor elements (Table S2). A high geogenic contribution for major elements in outdoor environments, where mass fractions are high but mostly attributable to natural sources, with the exception of Ca, showed a rather moderate contamination compared to the average crustal concentration from the literature. Indoor parking lots exhibited different patterns among them, including significant enrichments for minor elements, even after floor washing. The indoor parking in the residential building ($P2_{AVG}$) was way more contaminated if compared to the underground loading area of the shopping mall (P3). The reason could be found in the more demanding driving patterns required to circulate in the parking area, including steep ramps to change levels, leading to frequent use of brakes and steering wheels.

As per most studies of RD in urban environments [32,71], Al, Ca, and Fe were the most abundant elements in PM_{10} in all samples, in terms of mass fractions (wt %), followed by other major elements, such as Mg, K, P, S, Mn, and Ti. Vehicle-related metals in the group of minor elements were always present with relevant mass fractions, generally following the rule P2 > P3 > P1. That is the case for Zn, Ba, Cu, Cr, Sn (mostly P2), Zr, Sr, Pb, Rb, V, Li, Bi, Sb, Mo, Ni, As, Ce, Cs, Ga, La, and Nd. Whether applying the I_{geo}, instead, the contamination pattern changed to a rather different scale: Bi, Sb, Cu, Zn, Sn, Mo, Se, Cd, Cr, Li, Pb, and Ba, where some element oxides with lower mass fraction have instead a higher contamination level in comparison with geological background (Table S3).

Al and Ca in high concentrations in a closed environment, yet falling into the "uncontaminated" class of the I_{geo} index, are a symptom of the capacity of vehicles to transport geogenic dust all along their travel until parking. Silica is a typical mineral present in asphalt materials. It may provide important information about the processes of abrasion (friction and weathering) [52,72] and its subsequent distribution into the urban environment [73]. Nonetheless, the high enrichment in Ca might specifically indicate the presence of construction/demolition hotspots nearby [31]. Silicates, although mostly natural, may also derive from brake wear, such as zirconium silicate ($ZrSiO_2$), a friction modifier detected in RD from brake linings [74].

Heavy metals from vehicles are commonly attributed to non-exhaust sources (tyre wear, brake wear) since exhaust emissions have been decreasing their share. However, it is difficult to attribute to either a brake or tyre. For instance, brake wear is typically represented by Cu, Fe, and Sn [75,76], but Ba and Sb appear as tracers too [77]. The panoply of constituents that can be related to brake abrasion or specifically brake pad components, including SnS, Bi_2S_3, CuS, CuS_2, PbS, and Sb_2S_3, confirmed by previous studies in Portuguese cities [33], are very well represented in all samples of this study. The presence of Pb in the three parking areasmay be instead related to both vehicular and industrial emissions [24]. Zn, present in most urban environments with heavy traffic [78], is often found in association with carbonates and in the metal forms of ZnO and ZnS [79], but the threats derive from its mobility and bioavailability [80]. For this element, there is broad scientific agreement about its origins in tyre wear [31,81,82] and that aligns with extremely high contamination in indoor parking areas, where steering is very frequent, although performed at low speed.

Bismuth (Bi) is also often found in road dust, but it is not among the main elements or among those typically associated with one source. Indeed, it is a widely used metal in various industrial sectors, from synthetic fibres and rubber to lubricating greases, and it is also a superconductor for electromechanics [83]. In this campaign, it was the element with the highest geoaccumulation index ($I_{geo} > 7.4$) in all samples, and by far in $P2_{AVG}$, corresponding to 45 ± 13 $\mu g\ g^{-1}$ PM_{10}, while the rest of the samples were heavily-to-extremely contaminated (I_{geo} 4.3–4.4, 2.7 ± 2.1 $\mu g\ g^{-1}$ PM_{10} in P1, 3.7 ± 0.2 $\mu g\ g^{-1}$ PM_{10} in P3). Additionally, it was detected in Moscow, both in trafficked areas and, at a lower amount, in courtyards and parking areas [84]. In Viana do Castelo (Portugal), it was also found at highly enriched rates [33].

Rare earth elements (REE) are common in the upper continental crust, but they are becoming essential elements in the industrial sector, e.g., for battery manufacturing [85]. Among other traffic-related contributors, REE are present in tyre wear [68] and their presence has been also attributed to the metallurgical industry [86]. Additionally, a strong correlation was found between rare elements and African dust outbreaks [86,87]. Some REEs, such as Ce, La, and Zr, are often attributed to the same source since they have been increasingly used for catalytic converters to replace the use of lead in fuels, and it has been demonstrated that catalysers emit them together with platinum group elements (PGEs) [88]. Indeed, REE oxides are employed as stabilisers of the catalyst ceramic material and enhance pollutant oxidation [89].

In this study, the detected mass concentrations of Ce and La were 9.2–39.9 $\mu g\ g^{-1}$ and 6.4–15.6 $\mu g\ g^{-1}$, respectively, in line with the values from RD in Thessaloniki [90] and in a parking area in NW Germany, 38.0 and 18.8 $\mu g\ g^{-1}$, respectively, but less half of those in surrounding motorways [88,91], where higher contents were measured. Nonetheless, the La/Ce ratio of 0.5–0.7 was not much higher than the 0.46 attributed to the crust and the 0.41 detected in soils [89], indicating that the contribution of anthropogenic sources over a natural background must be carefully quantified and further studies are needed. According to the aforementioned literature, there is also a fairly well demonstrated association of Ce, La, and Zr with Fe/Mn oxides in road dust. Other than Zr, hafnium (Hf) was also detected in all samples, in the range 1.4–3.9 $\mu g\ g^{-1}$. It is indeed another traffic-related minor element, even though there is not much evidence about specific sources, and it used to appear accompanied by Zr [92,93]. Niobium (Nb)was detected in all samples (0.38–8.33 $\mu g\ g^{-1}$), but very few studies are available relating it to vehicle emissions [90].

Moreover, the use of element ratios is often seen in road dust studies to help differentiate sources on the basis of empirically known values that characterise, for instance, brake pads or other vehicle-related sources [94,95], yet their application and interpretation should be adapted to regional and local specificities. For instance, Cu/Sb in the range of 3.3–9.1 was associated with brake wear in PM in various European cities [96,97]. In this study, Cu/Sb values of 48.1 were obtained in P1, not very far from the value of 45 estimated for the upper crust [53]. For indoor parking spaces, the Cu/Sb ratio was 26.4 in $P2_{AVG}$ and

24.6 in P3. Slightly lower values were detected in the RD of London [96], but a handful of lower values for different cities can be found in Alves et al. [33].

As previously stated, Cu is an abundant component in brake wear, as are Fe and Sn, but it is also used in most brake linings. Hulskotte et al. [98] analysed dozens of brake pads and discs, concluding that brake discs mostly, but also brake pads, have high Fe contents (93% and 23%, respectively). In this study, the Fe/Cu ratio averaged 50.3 (P1), 44.0 ($P2_{AVG}$), and 58.3 (P3), in line with the values obtained in the RD of Aveiro-Ílhavo (65) (unpublished results), Oporto (49.3) [32], and Barcelona (37.2–52.1) [31]. Nevertheless, the Fe/Cu ratio significantly varies according to the brake formulation showingstrong geographical differences, reaching 182–211 in Beijing [99], New Delhi, and Birmingham [94].

The contribution of Fe from vehicle non-exhaust is corroborated by the Fe/Al ratio, 0.8 (P1) and 0.7 ($P2_{AVG}$ and P3), almost doubling the 0.4 tabulated for the upper crust [53], in line with values obtained in RD from active lanes of Aveiro-Ílhavo (0.37–1.63, average 0.8). As previously stated, among the most significant sources of road dust, asphalt-derived particles and construction-related dust are often deemed to be the top contributing factors to road dust [100]. In these samples, mostly P1, the presence of Ca was way above the crustal average, showing Ca/Al ratios of 15.2 (P1), 3.1 ($P2_{AVG}$), and 3.3 (P3). This indicates that a strong source of Ca contributes to heavy contamination, with extreme values detected in the outdoor parking area, possibly because of a severe degradation of the pavement.

3.2. PAH Content

Whereas in most studies of RD the elemental composition is targeted with a focus on key heavy metals, some pieces of research also aimed at studying the accumulation of organic components. Research studies have most often opted for the 16 priority PAHs due to their carcinogenicity, mutagenicity, and genotoxicity. Indeed, road dust is a source of organic pollutants [101]. The presence of PAHs in all fractions of particulate matter has been widely documented in road environments [12]. Sources can be either petrogenic, mostly related to fossil fuel storage, transport, and other industrial activities, or pyrogenic, due to the incomplete combustion and pyrolysis of organic components of wood, coal, and fossil fuels, among other activities [102,103]. Significant concentrations of 2–6 ring PAHs were found in the urban street dust of various urban hotspots in Guangzhou (China) with different land uses, and high-molecular-weight PAHs were predominant [104]. In Portugal, there are records of PAHs detected in the road dust of cities such as Viana do Castelo (37–926 $\mu g\ g^{-1}$ PM_{10}) [33] and Aveiro-Ílhavo (9.26–45.3 $\mu g\ g^{-1}$ PM_{10}) (unpublished results).

In this study, Σ_{PAH} doubtlessly represented a relevant contribution, accounting for 24.3 (P1), 8.38 ($P2_{AVG}$), and 23.2 (P3) $\mu g\ g^{-1}$ PM_{10}. Interestingly, $P2_{AC}$ showed the highest mass fraction (36.9 $\mu g\ g^{-1}$ PM_{10}). Considering that DL10 values after cleaning were much lower in comparison to all samples in the same parking, before the cleaning, these mass fractions denote either a rapid accumulation of pollutants, likely in the lower sizes of PM_{10} just a few days after the pavement was washed, or that the cleaning is rather ineffective for the organic fraction (see Table S4). Phenanthrene and naphthalene had the highest concentrations outdoors (P1), whilst indoors, those compounds were also complemented by a significant contribution from pyrene and fluoranthene ($P2_{AVG}$ and P3). These four organic compounds had also been detected in urban aerosols (ambient air PM_{10}) from various Portuguese cities [105].

3.3. Ecotoxicity

The utilisation of the bioluminescence inhibition bioassay with *Aliivibrio fisheri* to showcase potential toxicological effects of vehicle related aerosols has already been documented [44,106–109], but its application to road dust, especially from parking lots, is scanty. A first study applying this direct contact test to road dust collected from an urban environment showed little ecotoxicity [45]. Later studies have proven different outputs, suggesting that PM_{10} from road environments leads to a direct bacterial mortality when diluted in an aqueous extract [48,49]. In this study, all samples proved to be toxic, with average EC_{50} values of 65, 29, and 21% for P1, $P2_{AVG}$, and P3, respectively. There are, however, some clarifications to be made. The samples from the underground parking (P3) were overall the ones with the lowest EC_{50}, both expressed in percentage and in $\mu g\ mL^{-1}$, as well as the highest toxic units (TU) of 4.9, over 1.8 and 3.9 of P1 and $P2_{AVG}$, respectively (see Table S5). Therefore, high toxicity might not be explained by just a high DL10, but mostly higher contents of those components that are known for being lethal for bacteria, such as PAHs [46] or elements. Available literature indeed shows various results that could be summarised in two statements: there is a clear negative correlation between PAH content and EC_{50} and also a negative correlation between particle size and ecotoxicity [51]. In this study, a good correlation factor ($r^2 = 0.76$, inverse proportion) was found between PAHs and EC_{50} ($\mu g\ mL^{-1}$), indicating that an increased mass concentration of Σ_{PAH} leads to a lower amount of PM_{10} necessary to induce mortality in bacteria (measured as the reduction of 50% in the bioluminescence activity). Unlike Alves et al. [110], the correlation between PAHs and TU was rather poor, since the parameter is calculated on the percentage EC_{50}, regardless of the PM_{10} concentration present in the liquid extract. Additionally, it was not possible to establish correlations between EC_{50} and total element oxides, nor was it possible for EC_{50} and DL10. Interestingly, when the aqueous extraction carried smaller filter sections, implying lower amounts of particulate matter, the bioassay results showed a clear toxicity pattern with an EC_{50} below average. The optimisation of the procedure should therefore be achieved using the very same area of filter to compare all samples on the same ground, indicating that this analytical methodology is limited by the density of the extract and the capacity of the constituents of diluting in a water medium.

4. Conclusions

Sampling the thoracic fraction of road dust (PM_{10}) in a variety of geographical contexts and road environments enables a broader understanding of road dust generation, deposition, and resuspension processes at the regional and local scale. Data collected and presented in various studies in recent years have attempted to provide data for national databases of air pollution, and they already cover some cities all over the country. In this article, a similar approach was followed, but the road environment chosen was one with very specific driving conditions, namely areas of parking and loading/unloading, where the circulation of vehicles is expected to be at a very low speed, but with an increased rate of breaking and steering, enhancing the emission of pollutants (both organic and inorganic compounds). The chemical characterisation showed a different mass concentration of major and minor elements in their oxide form: they accounted for 15.7–17.8% of indoor parking lots and 22.8% of outdoor parking. The mass concentrations of major element oxides showed a very important contribution from Ca, Al, Fe, K, and Mg, which are typical crustal elements that are also generated through anthropogenic activities, namely vehicle traffic. Minor elements such as Cu, Zn, Cd, Sn, Sb, Pb, and Bi were also thoroughly scrutinised, trying to ascribe them to specific exhaust and non-exhaust emission sources. Nonetheless, with the aim of separating biogenic and anthropogenic sources as much as possible, the geoaccumulation index (I_{geo}) was calculated. Therefore, elements were categorised among contamination classes, drawing the attention to those elements whose mass concentrations were far higher than those expected in an average continental crust soil. This phenomenon unmistakably applies to Bi, Sb, Sn, Cu, Zn, and Mo, mostly in classes five and six (high-to-extremely and extremely contaminated). The examination of REE concentrations was instead legitimised by their growing use in vehicle parts (either catalytic filters or electronic parts), and they are quickly gaining momentum with the technological upgrade that the industry of electric vehicles is undergoing. The collected data, in terms of chemical profiling and the ecotoxicological effects, should both serve as input information for databases and emission inventories, and as a warning for technical stakeholders and decision-makers regarding the exposure of humans to traffic-related pollutants and their evolution keeping up with scientific and technological innovation.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/atmos14020305/s1, Figure S1: Map of Aveiro and indication of sampling sites. Source: Google Earth image. Figure S2: Multi-storey indoor parking. Figure S3: The shopping mall (FORUM Aveiro) and the entrance for loading trucks, vans, and other vehicles. Table S1: Dust loadings and emission factors. Table S2: Element oxide mass concentration for major and minor elements in the sampling sites. Table S3: The geoaccumulation index (I_{geo}) and the values obtained in the three parking areas. Table S4: PAHs content. Table S5: Results for the bioluminescence bioassay with *Aliivibrio fisheri* bacteria, including EC_{50} (% and mass concentration) and toxic units (TU).

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