

Pollutant Emissions of Vehicle Tyres and Pavement in Real Driving Conditions

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Abstract

Non-exhaust road transport emissions, particularly tyre-pavement abrasion, contribute to poor air quality. This original work is on particle emissions caused by tyre-pavement abrasion in real driving conditions in urban, suburban and motor-way areas. The collected data were analyzed according to the size, the number and chemical identification. Multivariate analysis provided clusters of size and chemical elements. Results showed the predominant particles (diameter $\varphi < 1 \mu m$ and $\varphi \epsilon$]1-2 µm]) for urban and suburban experiments, and]1- 3 µm] on motorways. A confirmed shift phenomenon at the close-to-the-source showed particles of large sizes [5- 10 µm]. The obtained clusters were: 1. Aluminosilicate, iron compounds, silicate free of aluminum (70%). 2. Ferrosilicon, silica, compounds of calcium, manganese, iron oxide, compounds of aluminum, sulfur, copper and zinc (29%). 3. A mix of various traces (1%). This paper will contribute to the development of new green technologies dedicated to improving sustainable road transport.

Keywords: Road Transport, Non-Exhaust Emissions, Tyres and Road Dust, Air Pollution, Environment

Introduction

Deterioration of air quality in every city in the world has become a major concern as environmental and health impacts have become highly significant [1-6]: there are more than 430,000 premature deaths every year in Europe and 7 million around the world per year [7-8]. In addition, chronic diseases such as asthma, cancers, pulmonary illnesses and heart diseases are associated with deterioration of air quality. It is proved that road transport has a negative contribution [9-13]. So far, little specified information exists on biological mechanisms and toxicology of non-exhaust particles [14-19] especially those having the potential to penetrate cell membranes, and / or penetrating the lung alveoli [5,17,20-23].

It is essential to emphasize that road traffic is a major contributor to non-exhaust emissions which are highly dependent on weather conditions such as wind speed and direction, ambient temperature, relative humidity (RH) and precipitation [3,24-25], distance from the road, topographical factors of the built environment in cities as well as road structures [3,26-28]. Depending on the variability of these parameters, various phenomena have occurred such as: atmospheric resuspension of particles, mixing – dispersion, coagulation, evaporation, dilution and turbulence, resuspension [2,6,12,29-32,34].

The interesting work by Kwak et al. [35] should be noted as he investigated the features of non-exhaust particles emitted on-road driving and in a laboratory study. They indicated that particles emitted by tyre wear are in the range of $2-3 \mu m$, while those by road surfaces are larger. Furthermore, particles from brake wear are in the size range of $1-10 \mu m$. They have shown that the concentrations of Fe and Ca were highest in the coarse fraction, and Fe, Ba, and Ti were most abundant in the fine fraction emitted during braking events.

Zhang et al. [36] analyzed the fraction of the PM_{10} : and $PM_{2.5}$: emitted by soil and cement dust, tailpipe exhaust, tyre and brake wear of road transport. The provided percentages seem to be far from the reality of this type of pollutant emissions by road transport.

Analysis by Harrison et al. [37] stipulated that the emitted non-exhaust particles appear in both the coarse $(PM_{2.5-10})$ and fine $(PM_{2.5})$ fractions, with a larger proportion in the former. Piscitello et al. [38] confirmed that the emission rate of the non-exhaust pollutants reached 90% by mass of total traffic-related PM emitted. They assessed emission factors as follows: 1 mg km⁻¹ veh⁻¹ to 18.5 mg km⁻¹ veh⁻¹ for brake wear, 0.3 mg km⁻¹ veh⁻¹ to 7.4 mg km⁻¹ veh⁻¹ for tyre wear, resuspended dust: 5.4 mg km⁻¹ veh⁻¹ to 330 mg km⁻¹ veh⁻¹. Authors [39-42] suggested that highest particle counts were observed in the size range of 0.25-0.5 μ m, and can be extended to the interval 0.25-10 μ m.

Crillery et al. [43] and Jeong et al. [44] confirmed that Cr, Ni, Cu, Zn, As, Cd, Sn, Sb, Pb are the major non-exhaust pollutants emitted by road transport. They also indicated that the combination of Cu and Zn can distinguish various non-exhaust traffic emissions, especially brake pads and tyres.

Effects of particle emissions by road traffic (exhaust and non-exhaust) on human health and environment have been regularly proven [1,3,45-48]. The major observed impacts are related to cardiovascular system (strokes and ischemic heart diseases), lungs inflammations, asthma, chronic lungs diseases, cancers, pulmonary fibroses, ... [12,49-54]. It would be very complex and useless to indicate all the references in this area, hence only those that seemed to be complete and provided factual evidence on these health impacts have been given. The literature describing health impacts and toxicological studies regarding road traffic emissions, among others, is very extensive and reflects the severity of transport particulate emissions. Many given analyses are based on particle sizes (from nano and ultrafine to large particles, volatile, semi-volatile, organic and inorganic particles, ...) but very rare analyses resulted in studies of these impacts according to the chemical composition of the emitted particles. In addition, environmental effects are numerous, for example: visibility impairment caused re-suspended particles and their dispersion [1,55-58].

The combined Scanning electron microscopy with energy dispersive X-ray analysis (SEM-EDX) is used for the identification of the chemical species collected in real driving conditions. The SEM-EDX is a quantitative method commonly used for obtaining chemical composition and spatial distribution of major and minor collected particles (images of the surface structures of the particles and chemical elements). It is a surface analytical technique with very high-resolution imaging capability (examination of surface morphology, topography, microstructure of environmental particles). SEM-EDX gave a quick nondestructive determination of the elemental composition of the collected particles on the membrane surface. SEM-EDX is a combination of two systems which can operate simultaneously. Scanning electron microscopy (SEM) provides images on the morphological features of the collected particles (surface), whereas Energy dispersive X-ray (EDX) gives the elemental composition of an area with a very high sensitivity and with a spatial resolution that could be less than 1 μ m². This technique is well known and used for a large scientific research, technical applications and innovations. The literature is very abundant in terms of application of this very efficient and effective method. We can mention the references [5,6,28] in our field of application.

This paper presents an original experimental work related to the particle's emissions by the abrasion of tyre-pavement in real driving conditions in urban, suburban and motorway areas. Analysis of the observed chemical species have been performed using the SEM-EDX which also gives an effective new method based on Multivariate data analysis (Hierarchical Classification on Principal Components) to investigate clusters of size distribution and pollutants identification (chemical characterization). Significant finding is presented, allowing to a better understanding of the non-exhaust pollution by road transport.

Size ranges, often given in the literature, revealed that the observed particle emissions could be a mixture with exhaust and non-exhaust emissions from road transport. In particular, the lower limit of the size intervals deserves further refinement in all those studies.

The first objective of this work is to specify the most preponderant granulometric intervals of these non-exhaust pollutants by road transport. The second one is to analyze the chemical composition of the emitted pollutants and their importance. In that respect, a significant experimental work and tools of data analysis were deployed. The quantity of acquired data and the most powerful analysis methods used provide an original work. One of the main challenges of this paper is to provide additional scientific information to complete the array of data (granulometry and chemical composition) already acquired and published in the open literature. The most important ones are briefly described above. In addition, the clustering, presented in this paper, could be essential to assess impacts of non-exhaust emissions (brakes, asphalt, tyres, dust resuspension) of road transport in cities, on air quality and human health., It could also be used in effective control measures. This work will certainly allow to obtain a significant advance in the implementation of regulations of not regulated pollutants. Moreover, it will certainly help decision-makers in the implementation of environmental policies on transport and air pollution.

Materials and Methods

Experimental Set-Up and Data Processing

Experiments have been carried out in and around the city of Lyon (Auvergne-Rhône-Alpes Region France) in three different experimental conditions (urban, suburban and motorway) during the summer period. The road traffic was very heavy in Lyon and its suburbs with about 179,300 vehicles a day on the ring road [59]. 70% of the vehicles in the suburbs travel less than 3 km of which around 60% are made by car [60]. As regards the motorway, 85 760 vehicles drive every day, **19 %** of which are trucks and 81% light vehicles [61]. Figures 1 (A, B, C) show the driving circuits where data have been collected in urban, suburban and motorway areas. Table 1 presents measurement days features including average meteorological conditions during experiments. Fifteen experiments were performed in real driving conditions: 5 in each area. The experiments were carried out on a total distance of 1,170 km: 220 km in urban streets, 320 km in suburban boulevards and 650 km on the motorway. The recorded data was collected on the same trips and refers to investigation of non-exhaust particle emissions (resuspension, tyre, brake and road surface wear) in real driving conditions. Speed of the vehicle and its exact position were collected using a global positioning system with a sampling of 1 Hz. During experiments, wind speed was less than 4 m/s. The duration of each experiment depended on the road type and the fluidity of the road traffic.



Figure 1 (A): Experimental areas where data were collected in urban, suburban and highway areas in real driving conditions. Urban trip (blue color in bold) - Source [https://www.google.fr/maps/]



Figure 1 (B): Experimental areas where data were collected in urban, suburban and highway areas in real driving conditions. Suburban circuit - Continuous yellow color in bold (Lyon eastern ring road) (modified picture from the original map by Tibidibtibo (2021)



Figure 1 (C): Experimental areas where data were collected in urban, suburban and highway areas in real driving conditions. Highway A43 (France) between the city of Bron (69500) and Romagnieu (38480) – France (source Google)

Road type	Distance (km) for each experiment	t° (°C)	Precipitation (mm)	Humidity	Wind speed (km/h)	Cloud cover	Pression (hPa)
Urban	2x20	25	< 1	63 %	7	13 %	1018
Suburban	2x32	26.5	< 5	48 %	14	21%	1019h
Highway	2x65	28	5	13 %	13	37 %	1017

Table 1: Features of measurement conditions: average meteorological conditions during experiments

Non-exhaust particles, emitted by tyres and pavements in real driving conditions, have been collected by two types of measurements. Simultaneous measure of particle sizes (granulometry) and a collection of particles on carbon adhesive tabs (=12 and 47 mm) and on polycarbonate Whatman* Nuclepore^{TT} Track-Etched Membranes ($\varphi = 25$ mm, pore size 0.4µm) have been carried-out. The later enabled their chemical analysis (composition and identification). Membranes were considered free of traces of the chemical elements Si, Sb, S, Fe, Mg, and Na. They ensured no contamination (high chemical resistance, good thermal stability, smooth flat surface for good visibility of particles). No distinction in this work can be made between what is emitted by the tyres or by the abrasion of the pavement. All measuring instruments were synchronized (injection of a square wave signal every 5 minutes).

The first recording data concerned PM concentrations which were measured by a light-scattering method. An optical particle counter 'OPC' GRIMMTM EDM 180 [62] (certificates and approvals: US-EPA, UK-MCERTS) was used to measure particles concentrations with a flow rate of 1.2 l/min ($0.1\mu g/m^3$ resolution) which have a diameter between 0.35 and 22.5 μ m. This OPC is a real-time measurement in ambient air of PM₁₀, PM_{2.5}, PM₁, total counts, and particle number distribution with no loss of semi-volatile compounds. Other features of this analyzer and its performance are:

- 31 sizes channels particle number (0 $3*10^{+6}$ particles/liter),
- Recorded parameters: temperature and relative humidity pressure wind speed,
- Fully automated monitoring system insensitive to vibrations,

• Total inlet flow analyzed in optical cell - excellent counting statistics and reproducibility at low and high dust concentrations (reproducibility >97% of total measuring range).

It has an efficient counting statistics and reproducibility of dust concentrations with low to high levels. Before every measurement campaign, zero calibrations were regulated on both the GRIMM and the GPS. Each measurement was followed by blank tests conducted during 15 minutes. Summer tyres, with a specific dimension of 185/75R16C XGreen, were used to study particle emissions by tyres and pavement abrasive contact. The inlet sampler pipe was fixed in the vertical axis of the wheel between 2 to 5 cm from the pavement, and 4 cm behind the vertical plane of the tyre situated at a height of 2 to 5 cm. This is the minimum distance at which data can be collected the closest to the ground. Figure 2 shows the experimental configuration. During this experiment, the exhaust pipe was located outside on the left side of the vehicle of avoiding a mix data collection (exhaust and non-exhaust).



Figure 2: experimental configuration of the experimental set-up related to data collection (distance pavement to data collection point)

Control tests were carried out to ensure that the exhaust and non-exhaust mixture of emissions was not collected by the measurement system. Indeed, as shown in figure 3, the sampling probe was placed in the centre axis between the wheels and at a distance of 5 cm, then 13 cm and 24 cm from the ground. These three ground distances confirmed the absence of exhaust particles in the collected data. However, measurements in this position showed that resuspension-induced particles were found as well as particles emitted by the brakes. The position of the sampling, due to the screen effect by the double wheels, guaranteed that 98% of collected particles were emitted by abrasion due to contact with tyres



Figure 3: Control set-up showing screen effect of the collected data by abrasion of pavement and tyres

This paper does not report on the study of the complex effects of the vehicle speed versus concentrations of particles in near field and related to air quality, but deals with the analysis of non-exhaust particles at the emission point (as close as possible to the source). Previous work [63-65] confirmed the link between the increase of the particle emissions with the vehicle speed, and also supported that, at distance of 3 m from the emission source, particle levels decreased with speed (faster deposition – dilution effect – dispersion).

On the one hand, the analysis of the collected particles on membranes was carried out with the well-known SEM-EDX which provides chemical identification of the collected particles. The objective was to analyze the composition of particles collected from dust sampling in real driving condition on the carbon membrane.

The JSM-6510LV (JEOL Ltd), which is a high-performance SEM for fast identification of the collected data, was coupled to an EDX system (Oxford Aztec-DDI X MAXN 50) to analyze our collected particles. The different particles were analyzed under the same experimental conditions in order to be able to compare their EDX spectra: retro-scattered electrons, intensity of 20 kV, working distance equal to 12mm, pressure = 20 Pa, magnification x500. We conducted these analyses with a high resolution of 3.0 nm at 30 kV which delivered the finest structures of the TRWP particles emitted and their chemical composition. The resolution is several hundred times greater than the optical microscope, with a focal depth several dozens of times greater than the optical microscope. This combined technique allowed the analysis of the most diverse inorganic materials. We obtain images on the morphological features of the collected particles surface, elemental composition of each analyzed area of the carbon membranes. The used SEM-EDX is equipped with INCA software (ETAS®) that allowed analysis, by individual identification spectra, of the collected particles (flash programming, analysis, calibration data management, automated optimization). For each sample we automatically analyzed an average of 1,035 particles corresponding to a membrane average surface of 8 mm². Each particle was classified in a set according

to its chemical composition and the result is given in the form of a spectrum. This is an iterative processing method allowing to a chemical identification of all particles. Traces were, in general, considered as contaminants. Once the particle identification sets have been constructed, statistical analysis was carried out with the statistical software R (FactoMineR package) dedicated to multivariate data analysis [66-68].

The use of the Hierarchical Classification on Principal Components (HCPC) to data analyzing and clustering of data vs. size and chemical compounds proved to be very reliable and provided a ranking of the different clusters in terms of predominance and chemical elements identification. In addition to the INCA software, the application of the HCPC method for the collected data provided structure of the data in terms of a partition on each granulometry interval, an identification of chemical elements, and consequently gave a hierarchy structure on the obtained cluster of particles. This method allowed the following analysis process:

- Original data: Extraction of particle size ranges.
- Assignment of intervals to identified clusters or groups of particles.
- Homogenization of the clusters according to their predominance: importance of their presence or not in a regressive way (clusters were homogeneous).
- Calculation and construction of the clusters or the tree of clusters.
- Result with a hierarchy tree by granulometric intervals and by chemical element class (to obtain the percentages of predominance of each cluster).
- Checking errors and their propagation, and providing means, min, gravity centers, ...
- Clustering numerical data with R [66-69] followed the well-known steps:
- Import dataset in a new data matrix data needing scaling before clustering.
- Build this matrix of the scaled data and apply the Ward's minimum variance method hierarchical clustering algorithm [70].
- Identify and assess percentages of elements inside the clusters.
- Choose the number of clusters that were relevant [71].
- Built the tree and interpret the obtained clusters using the principal component analysis of FactoMineR package.
- Cut the tree into three to ten clusters with weights depending on the aerodynamic diameters of particles and their chemical group.
- Interpret the partition of each cluster versus the obtained percentages of each chemical elements or group of elements.

Based on the previous steps, data analysis of results was grouped by type of route (urban – suburban – motorway trips), granulometry interval and percentage of identified chemical elements. Metal wrenching and abrasion of brakes were not considered. Whenever braking occurred, the corresponding data was systematically deleted. Images (Figure 4) show the observed brakes metal wrenching and abrasion during braking (clusters of particles, fibres, large metal dimensions, and a high-rate production of non-exhaust particles).



Figure 4: Observed metal and fiber elements removal and wrenching during braking on highway

This paper used the chemical elements of brakes emissions previously identified by Khardi et al. [28], particles emitted from the exhaust, resuspension and other contaminations, etc., being a necessary condition for identifying the emitted particles coming from the abrasion of tyres and pavement.

After clustering steps, inertia of chemical species has been checked to analyze the homogeneity of the classification in their corresponding clusters. Percentages of pollutants (chemical species i), belonging the groups (U, PU, H), were assessed in the all measured samples. Thus, we calculated the $v_{\rm test}$ by applying the following formula [68-72] for species i (i ε groups) to verify the homogeneity degree of clusters:

$$v_{test[i]} = \sqrt{N_{[\cup_{I}]}} \times \frac{f_{i/[I]} - f_{[\cup_{I}]}}{\sqrt{\frac{N_{i/[I]} - N_{[\cup_{I}]}}{N_{i/[I]} - 1}} \times f_{[\cup_{I}]} \times (1 - f_{[\cup_{I}]})}}$$

where:

i: chemical specie (pollutant) - $f_{i/[I]}$: frequency of the i specie in the set I (I= U, PU, H) $f_{[UI]}$: frequency of the i specie in the all sets of data (U and PU and H) $N_{i/[I]}$: the relative number of i specie in the set I (U, PU, H) versus the size of I

 $N_{[U_I]}$: the relative number of i specie in the all sets of data I ([UI = [U + PU + H]) versus the total size of the sets U, PU and H v_{test} is well-known to be sensitive to the cluster sensitivity analysis. It is used in this paper to categorial variables i in terms of the relative percentages.

This is a necessary and sufficient condition when groups are different and the measured species are identical. The reproducibility is therefore not to be verified afterwards.

Results and Discussion

This paper presents two complementary results on particles emissions due to the abrasion of tyres and pavement in real driving conditions. The first type involves particle size analysis and the second a chemical characterization of those emissions due to tyreground contact. The following two sections provide the obtained results from analysis of the collected data on tabs described in previous sections (numbers and sizes of particles and their chemical identification). As previously described, HCPC was used to process the collected data (clustering vs. size and chemical nature of the collected elements). It should be remembered that analysis of particle size data is largely simpler than the chemical identification of particles and the use of a hierarchical scheme providing clusters.

Number-Size Distribution Analysis

This section presents the main obtained granulometric results for the two measuring points: the first one is at a height of 5 cm from the ground (point 1), in relation to the tyre-pavement contact point. The second point corresponds to the closest point to the ground, at about 1 to 2 cm (point 2) from the tyre-pavement contact point. Concerning the size analysis of the collected particles for the three groups (urban, suburban, motorway), 7 clusters are identified for the point 1 and 4 for the point 2, which correspond to particles groups with aerodynamic diameters φ , as follows:

Point 1:

- Cluster 1: φ < 1 μm,
- -Cluster 2: $\phi \, \epsilon$]1-2 μm],

- Cluster 3: φε]2-3 μm],
- Cluster 4: φε]3-4 μm],
- Cluster 5: φε]4-5 μm],\
- Cluster 6: φε]5-10 μm,
- Cluster 7: φ > 10 μm.

Point 2:

- Cluster 1.2: $\phi < 5 \ \mu m$,
- Cluster 2.2: φ ε]5-10 μm,
- Cluster 3.2: $\phi \; \epsilon \;]10\text{--}100 \; \mu m,$
- Cluster 4.2: φ > 100 μm.

Figure 5 presents the obtained results relating to the collected particule number versus the sizes for the first point and the three homogeneous groups (U: urban, PU: suburban, H: motorway). Regarding the first position of data collection (point 1: 5 cm from the tyre/pavement contact point), analysis shows a similar behaviour between urban and suburban measurements for the three homogeneous groups 1, 2 and 3: On the one hand, the predominant size is in the decrease order corresponding to the three first clusters: 1. Cluster1 ($\varphi < 1 \mu m$). 2. Cluster 2 ($\varphi \epsilon$]1-2 μm]). 3. Cluster 3 ($\varphi \epsilon$]2-3 μm]). On the other hand, the predominant sizes for the experimental groups carried-out on motorway are as follows in terms of emitted quantities: 1. Cluster 2 ($\varphi \epsilon$]1-2 μm]). 2. Cluster 3 ($\varphi \epsilon$]2-3 μm]). On the other hand, the predominant sizes for the experimental groups carried-out on motorway are as follows in terms of emitted quantities: 1. Cluster 2 ($\varphi \epsilon$]1-2 μm]). 2. Cluster 3 ($\varphi \epsilon$]2-3 μm]). 3. Cluster 4 ($\varphi \epsilon$]3-4 μm]). Cluster1 ($\varphi < 1 \mu m$) is present but with small quantities compared to Cluster 6 ($\varphi \epsilon$]5-10 μm]). At point 2, analysis shows that emissions due to road tyre-pavement contacts are predominant in the range of particle size between 5 and 10 μm (Cluster 2.2) for the three urban, suburban and motorway routes (figure 6). Emissions in urban areas are fairly predominant compared to suburban and motorway ones for cluster 1 to 5. An increase of 10% of particle size under 5 μm has occurred for urban route compared to the other two. In addition, no difference was observed between suburban and motorway experiments in terms of emission dynamic behaviour but tyres-road emissions are in favour of Cluster 2.2.



Experimental groups (U, PU, H)

Figure 5: Particule number collected vs. size intervals for the three groups - Point 1



Figure 6: Particule number emitted during all combined groups vs. size intervals - Point 2

For the closest point to the roadway, the particles emitted have large sizes.

It is essential to underline our observation that the behaviour, in terms of emissions, is identical between the urban, periurban and motorway expriments. The number of the emitted particles are equal between U_1 , U_2 and U_3 , similarly for the peri-urban and peri-urban profiles. This is the reason why figure 6 presents the number of particles without index corresponding to the group of experiments (or to the group to which they belong). The simple explanation is that, at this position, a tearing phenomenon due to this strong abrasion took place and was certainly associated with the resupension. Particles of weaker sizes were present but were not collected enough simply because they were dispersed certainly due to significant turbulence. Thermal effects would have been responsible for this dispersion at a very short distance from the source. Moving closer to the source can explain some phenomena but it is difficult to conclude, with the absence of dispersion studies, on the pollutant plumes in the air in relation to the practiced speeds.

At very low heights from the ground, the emitted particles have large sizes and the small ones are quickly dispersed with the turbulence. This size effect could lead to the generation of secondary pollution at short distances for small particles with "a shrinkage phenomenon" of the large particles probably taking place. However, this closest-to-the-source experimental approach would definitely help tyre and pavement manufacturers to better design their new products (less abrasive and less polluting - more sustainable and more efficient). Finally, it should be noted that experimental work was not carried out as a function of the vehicle speed. Further research is needed in this way to entirety understand this specific pollution.

The conclusion is that the major significant emitted particles have sizes below 2 μ m for urban and suburban trips and 3 μ m for motorway experiments. There is a size shift between (urban-suburban) and motorway experiments. Larger sizes are not present because they are deposited on the pavement. This finding completes the obtained results by Iijima et al. [73] in terms of size distribution (1–10 μ m and 2–3 μ m). Our results confirm analysis by Liu et al. [39] which identified the variations in particle size range, in particular in 0,25–10 μ m interval. This size range revealed emissions that could be a mixture with exhaust and non-exhaust emissions. The lower limit of this range interval deserves further refinement in both studies. For this, the use of particle analyzers with finer sizes would be an essential asset. In addition, Harrison et al. [37] confirmed in their analysis of the non-exhaust emissions, from abrasion of the brakes and tyres and wear of the road surface, as well as from resuspension of road dusts, that the emitted particles have to be in both the course (PM_{2.5-10}) and fine (PM_{2.5}) fractions, with a larger proportion in the former. Our results agree with those obtained in the open literature [5,35, 37-39,46-48,53,74-75]). Our clustering could be essential to assess particle impacts on air quality and human health and, in addition, could be used in effective control measures.

Chemical Analysis of Collected Particles

Data analysis shows a wide variety of chemical elements emitted in real driving situations for urban, suburban and motorway environments.

For chemical analysis, the systematic presence of carbon (C) and oxygen (O) was noticed in all analyzed tabs. In order to identify the tyres-pavements particles, carbon and oxygen C and O were excluded from the statistical classification.

In this paper, neither the production modes of the emitted particles nor their shapes were analyzed because this would require very complex additional experimental work. Individual analysis is very expensive in terms of computational time. The modes and shapes that could be subject to further works are:

- Nucleation mode (particles having a spherical shape with ϕ < 30 nm),
- Accumulation (particles having an angular morphology with 100 nm < ϕ < 200 nm),
- \bullet Coarse mode (particles aggregated with 1 $\mu m < \phi).$

Clusters were obtained through a HCPC hierarchization tree in terms of the identified chemical elements. Analysis of data gave a hierarchical classification of the main classes or clusters and provided the gravity centres of the categories of pollutants from the most preponderant to the least important. The major and distinguished clusters are:

- Cluster A: the most abundant pollutants,
- Cluster B: the less abundant composition of the chemical elements,
- Cluster C: percentages of traces of less significant pollutants which origin is often complex to explain.

Observed results for urban, suburban and motorway driving routes indicated that the main common elements, presented in the first row of the table 2, are the aluminosilicate, silicate free of aluminum, silica, AlO, CaO, FeO, SiFe, FeCr, PCa, and series of compounds of [Al, Ba, Ba+Zr, Ca, Cl, Cu, Fe, Ti, Zr, Zn, S, Ni]. They are the most preponderant in terms of real driving emissions. Traces of some elements, presented in the second and third rows of the table are generally combined chemical elements, many of which are either contained in the tyres and pavement composition or could be considered as contaminants.

Urban roads	Suburban roads	Highways						
Common compounds:								
– Aluminosilicate	Aluminosilicate							
 Silicate free of alumin 	Silicate free of aluminum							
– Silica – AlO - CaO - F	eO – SiFe – FeCr – PCa							
 Series of compounds of 	of [Al, Ba, Ba+Zr, Ca, Cl, Cu, Fe, Ti, Zr, Zn, S	, Ni]						
Series of compounds of	Series of compounds of:	 Compounds of Mn 						
Combined compounds of:	– [Sn, Ti, W FeCr]	- Combined compounds						
[Mg, Mn, Pb, Sn, W]	– Cr+Sba	of elements [Cr+Zr+Al+W +Cu +						
	– Ni+Sn, Zn+MoO+Ce+Cl	PCa]						
	– Zn+Ti+SBa							
Combination elements of:								
– PClFe+Ba+Cr+Zr								
– SBa, SiTi, TiO								
– SBa+SiTi+Sn								
– AlO+NaCl+SiTi								
 Compounds of: 								
– Ce+Cr+Cu+Ni+Pb								
– Cl+Sba+Sn+Zr								

Table 2: Chemical elements emitted by abrasion phenomena due to contact tyres-pavement during driving (urban - suburban - highway)



Figures 7 (A, B, C) present the number of the identified particles versus the total recorded number during urban, suburban and motorway trips for three experimental groups in real traffic conditions. The corresponding percentages are given in tables 3, 4 and 5.

Figure 7 (A): Normalised number of particles emitted during urban (A) experiments and their chemical composition



Figure 7 (B): Normalised number of particles emitted during suburban (B) experiments and their chemical composition



Figure 7 (C): Normalised number of particles emitted during highway experiments and their chemical composition

On the one hand, chemical analyses confirmed, for all routes, the predominance elements such as: Aluminosilicate, compounds of Fe, silicate free of aluminum, SiFe, FeO, Silica. These elements are representative of the abrasion due to the contact with tyres. The aluminosilicates were highly emitted during the motorway experiments, while the emission rates during urban and suburban ones were similar. The number of the particles of the compounds of Fe were widely emitted in the following descending order: 1. Suburban, 2. Urban, 3. Motorway. On the other hand, chemical analysis showed the presence of the following chemical elements for the three driving situation:

- Urban: compounds [SBa, Sn, Zr], compounds of [Mn, Ca, S, Ti] and CaO,
- Suburban: compound of Ca, CaO, compounds of [S, Al], and PCa,
- Motorway: compounds of Cl, [Cu, Ca], FeCr, [Al, Zn], CaO.

Urban experiments were characterized by the presence of metallic elements, which were certainly emitted by brakes and / or resuspension phenomena. The presence of Cl and its compounds of Cl for the motorway route experiments was mainly due to the residues of strong pavement salting during the winter period. Tables 3, 4 and 5 present the normalised number of particles emitted for urban, suburban and motorway experiments and their chemical composition.

U1		U2		U3	
Aluminosilicate	47,5%	Aluminosilicate	41,3%	Aluminosilicate	49,1%
Silicate free of Al	10,4%	C[Fe]	16,2%	C[Fe]	17,8%
C[Fe]	9,8%	Silicate free of Al	9,2%	Silicate free of Al	7,6%
Silica	8,6%	SiFe	6,7%	SiFe	6,5%
SiFe	6,2%	[SBa+Sn+Zr]	5,1%	FeO	4,7%
[Ca]	3,7%	C[Mn]	4,6%	C[Cl]	3,6%
FeO	3,2%	Silica	3,5%	Silica	2,6%
CaO	2,8%	FeO	3,3%	CaO	1,7%
C[Ti]	2,8%	CaO	3,2%	C[Ti]	1,4%
C[S]	2,3%	C[Ti]	2,2%	C[Zn]	0,9%
PCa	0,8%	C[Zn]	1,4%	C[S]	0,8%
C[Al]	0,4%	C[S]	0,9%	C[Al]	0,8%
C[Cl]	0,3%	PCa	0,8%	PCa	0,6%
C[Cu]	0,3%	C[Al]	0,7%	C[Mn]	0,5%
SiTi	0,3%	[Ce+Cr+Ni+Pb]	0,4%	C[Cu]	0,3%
C[SBa+Sn+Zr]	0,2%	C[Ca]	0,2%	C[Ca]	0,2%
FeCr	0,2%	C[Cl]	0,1%	[SBa+Sn+Zr]	0,2%
C[Zn]	0,1%	C[Cu]	0,1%	SiTi	0,1%
Other traces	0,1%	SiTi	0,1%	[SBa+SiTi+Pca+Sn]	0,1%
				FeCr	0,1%
				C[Sn]	0,1%
				C[Ni]	0,1%
				Ba – SBa	0,1%
				C[Cu]	0,1%

Table 3: Collected and identified pollutants during urban experiments in real

driving conditions (average percentages - C[X] = compounds of the X element)

PU1		PU2		PU3	
Aluminosilicate	54,2%	Aluminosilicate	35,8%	Aluminosilicate	46,4%
C[Fe]	12,5%	C[Fe]	23,3%	C[Fe]	18,4%
Silicate free of Al	6,4%	Silicate free of Al	6,9%	C[Ca]	7,4%
Silica	5,9%	SiFe	6,4%	SiFe	5,7%
SiFe	5,3%	C[Ca]	5,3%	Silicate free of Al	5,5%
C[Ca]	5,2%	C[S]	5,0%	CaO	4,2%
CaO	3,1%	CaO	4,4%	Silica	3,2%
FeO	2,9%	C[Al]	3,8%	FeO	2,7%
C[S]	1,2%	FeO	3,4%	PCa	2,1%
PCa	0,9%	Silica	2,2%	C[S]	1,6%
C[Ti]	0,5%	C[Cu]	1,2%	C[Zn]	1,3%
Ba - SBa	0,5%	C[Zn]	0,9%	C[Cl]	0,6%
C[Cl]	0,4%	C[Cl]	0,4%	C[Cu]	0,3%
C[Al]	0,4%	PCa	0,4%	FeCr	0,2%
C[Zn]	0,2%	FeCr	0,3%	C[Cr]	0,2%
C[Cu]	0,2%	[PClFe+Cr+Zr]	0,2%	Ba - SBa	0,1%
FeCr	0,1%	C[Ti]	0,1%	C[Ni]	0,1%
C[Cr]	0,1%		·		

 Table 4: Collected and identified pollutants during suburban experiments in real traffic (average percentages - C[X] = compounds of the X element)

H1		H2		H3	
Aluminosilicate	53,7%	Aluminosilicate	66,6%	Aluminosilicate	34,4%
C[Fe]	12,6%	C[Cu]	6,9%	C[Cl]	19,3%
Silica	7,9%	Silicate free of Al	6,7%	C[Fe]	14,1%
SiFe	7,4%	C[Cl]	6,7%	Silicate free of Al	6,9%
Silicate free of Al	5,8%	C[Fe]	3,3%	Silica	6,0%
C[Ca]	4,0%	CaO	2,6%	SiFe	5,0%
FeO	3,0%	FeCr	2,6%	C[Cu]	4,9%
CaO	2,0%	C[Al]	1,5%	C[Al]	3,4%
PCa	0,9%	SiFe	1,3%	C[Zn]	3,3%
C[Ti]	0,7%	Silica	1,0%	C[S]	1,0%
C[S]	0,7%	C[Zn]	0,3%	PCa	0,6%
C[Al]	0,5%	C[S]	0,2%	CaO	0,3%
C[Cl]	0,2%	AlO	0,2%	C[Ca]	0,3%
C[Zn]	0,1%	FeO	0,1%	C[Ba]	0,2%
FeCr	0,1%			FeCr	0,2%
AlO	0,1%			AlO	0,1%
[Cr+Zr+Al+W]	0,1%				
FeCr	0,1%				
compounds [Cr+Zr+W]	0,1%				

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Table 5: Collected and identified pollutants during highway experiments in real traffic (averagepercentages - C[X] = compounds of the X element)

Differences of the relative percentages between the three types of experiments (urban – suburban – motorway) confirm, apart from the peak related to the explained Cl compounds, the same behavioural trend between the three pathways. Tables 3, 4 and 5 indicate the obtained data for the performed experiments. Compounds of Ti were particularly present in urban areas but almost absent in suburban and motorway experiments. HCPC analysis provided percentages of the measured and identified pollutants vs. their clusters for urban, suburban and motorway groups (higher to lower percentages) as follows (Table 6):

A. Cluster A: Aluminosilicate, compounds of Fe, Silicate free of Al are confirmed to be predominant,

B. Cluster B: SiFe, Silica, compounds of (CaO, Ca, and Mn), and FeO, compounds of (Al, S, Cu, and Zn), PCa,

C. Cluster C: a mix of various traces which can have their importance and consequences (impacts) according to the emitted quantities.

	Aluminosilicate	C[Fe]		Silicate free of Al		
Cluster A	47%	15%		8%		
(70%)						
Cluster B	SiFe	Silica	CaO	C[Ca] C[Mn]		C[Mn]
(29%)	6%	4%	3%	3% 3%		3%
	FeO	C[Al]	C[S]	C[Cu]	C[Zn]	PCa
	3%	2%	2%	1,0%	1%	1%
Cluster C	Traces of other pollutants					
(1%)	1%					

Table 6: Results - Clusters A, B and C. (C[X] = compounds of the X chemical specie)

This distribution is schematized in figure 8 and shows the importance of cluster A, then B. This is not because cluster B is at 29% that it should not be considered in the framework of the technological developments aiming to control - to reduce - these non-exhaust-emissions and their impacts.



Figure 8: Distribution of pollutants emitted by the abrasion of tyre-pavement in real driving conditions

In addition, the mix of various traces of other chemical pollutants are a combination of species: [Ce+Cr+Ni+Pb] - [PClFe+Cr+Zr] - [Sba+SiTi+Pca+Sn] - [Ba - SBa] - C[Ba] - C[Cr] - C[Ni] - C[Cr+Zr+Al+W] - C[Cr+Zr+W] - C[Sba+Sn+Zr], ... The traces of these recorded chemical elements, characterized by very low percentages, are considered as insignificant. However, in order to quantify the total mass of emissions of these elements for the traffic in France, an overall analysis should be carried out to assess the impact on health and the environment.

Pearson and Spearman correlations applied to the set data according to the different variables (urban, suburban and motorway routes, the same vehicle equipped with the same tyres, the weight of the vehicle, nature of the pavement, ...), showed that correlations were significant (with p < 0.02). In addition, analysis of the inertia of chemical species i (called X in the previous sections) has been carried-out to confirm the homogeneity of their classification in their corresponding clusters (and thus their belonging). Assessment of v_{-} test was done especially for the comparison between and within clusters. Characterization of the clusters has then

been performed, and the significance test calculated:

- ν_test is equal to 1.92 for urban sets of data, 1.97 for suburban, and -1.87 for motorway (p ε 5%).

The inertia of chemical species, in the three clusters, is definitively homogeneous, and the cluster hierarchy is therefore considered significant (v_test ± 2).

Finally, it should be remembered that this finding completes and provides more additional information to the work by Iijima et al. [73] which indicated that metallic elements are (K, Ti, Fe, Cu, Zn, Sb and Ba), and are largely emitted. They have also shown that the shapes of the elemental mass distributions of (Ti, Fe, Cu, Zn, Sb and Ba) were similar to the total mass distributions of the brake abrasion dusts.

Conclusion

This paper presents an original experimental approach combining collection of particles emissions due to the abrasion of tyres and pavement in real driving conditions. The SEM-EDX spectroscopy carried out identified the chemical elements. The successfully applied Hierarchical Classification on Principal Components provided clusters of predominant particles and their chemical nature.

Analyses showed that the predominant particles simultaneously emitted by the abrasion of tyres and pavement had a diameter $\varphi < 1 \ \mu m$ (the more significant), $\varphi \epsilon \]1-2 \ \mu m$] for urban and suburban experiments, and $\varphi \epsilon \ [1, 3 \ \mu m]$ for motorways. In addition, HCPC showed three different clusters in terms of predominance of the identified chemical elements by SEM-EDX: 1. Aluminosilicate, compounds of Fe, Silicate free of Al (Cluster A, 70%). 2. SiFe, Silica, compounds of (CaO, Ca, and Mn), and FeO, compounds of (Al, S, Cu, and Zn), PCa (Cluster B, 29%). 3. A mix of various chemical traces (Cluster C, 1%).

Further research, including the re-suspension phenomena, is therefore needed and should take into account the nature of road surfaces and tyres as well as their physicochemical features. Moreover, a consequent metrological development must be carried out to separate the sources of particles emitted by tyres and pavement and their resuspension. Significant complementary scientific work is necessary on exposure-response relationships to contribute to the development of health guidelines. As a result, appropriate regulations as well as public and environmental policies would provide the necessary support for the application of emerging technologies with a double objective of population well-being and environmental improvement.

Declarations

Ethics Approval

Not applicable

Consent to Participate

Not applicable

Consent for Publication

Not applicable

Availability of Data and Materials

The datasets used and analyzed during this current research are available from the corresponding author on reasonable request.

Competing Interests

Authors hereby declare that they have no known competing financial interests or personal relationships that in any way could have influenced the work reported in this paper.

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