Non-Exhaust Emissions from Brakes: Comparative Assessment of Physico-Chemical Properties in Nanometric and Micrometric Particulates

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Outline

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- Materials & Sampling
- Preparation & Analytical Probes

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- Concentration
- Particle Size Distribution
- Elemental Composition
- Metals Speciation
- Organic Compounds

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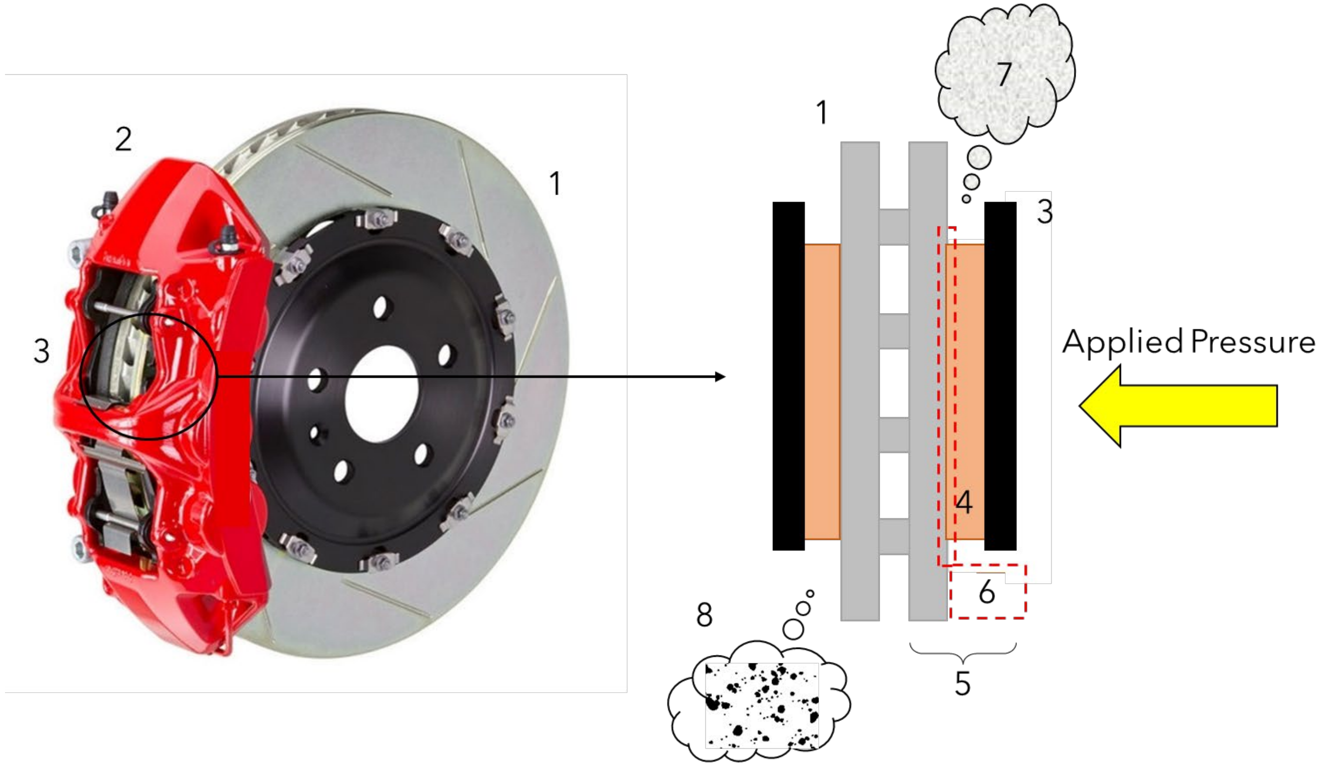
Introduction



Disc Brake System:

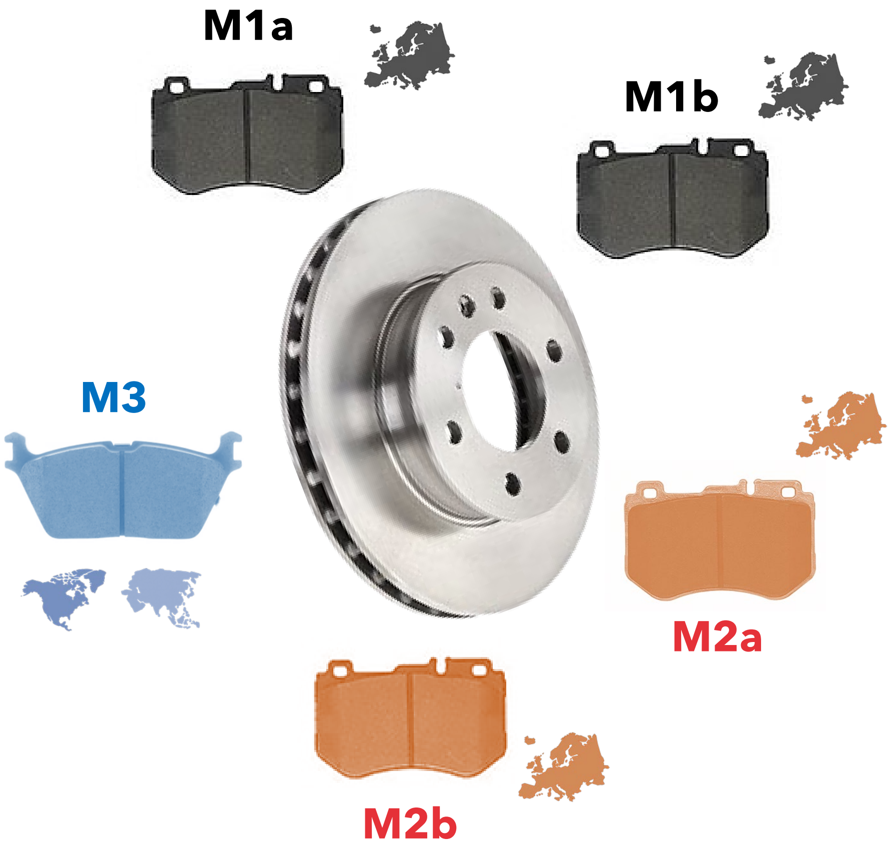
1- Braking Disc (**BD**)	**5-** Friction Couple **(FC)**
2- Caliper	**6-** Tribological Interface
3- Braking Pads	**7-** Non-Exhaust Emissions **(PMs)**
4- Friction Material (**FM**)	**8-** Non-Exhaust Emissions **(Debris)**

- 10% of $PM_{2.5}$ and PM_{10} emissions are due to road transport sector (up to 70% if EU urban environment is considered)
- Nowadays *Exhaust* and *Non-Exhaust* emissions from road transport are estimed to contribute at the same level
- Inside *Non-Exhaust* category, PM10 and PM2.5 generated from brakes are estimed to contribute respectively by 55% and 21%
- Substantial lacking of information on the **U**ltra-**F**ine **P**article fraction (**UFP**: d < 100 nm)

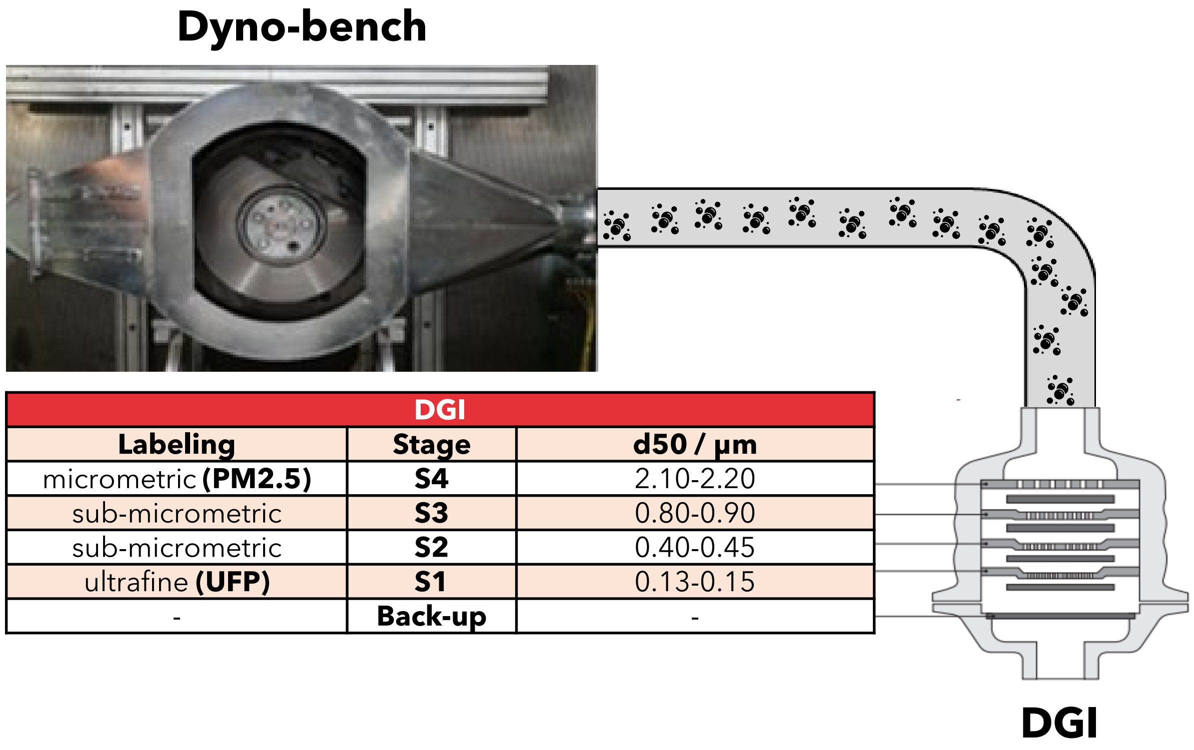
nPETS
Nanoparticle Emissions From The Transport Sector

Horizon 2020 – Grant n°954377

Experimental: Materials & Sampling



M1a, M1b: ECE R90 Low Steel/Low Met, Cu-free
M2a, M2b: ECE R90 Low Steel/Low Met, Cu-content
M3: NAO, Cu-free

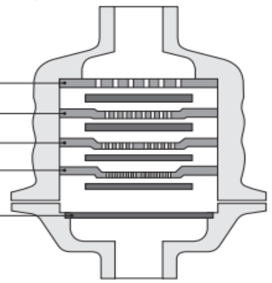


DGI		
Labeling	**Stage**	**d50 / µm**
micrometric **(PM2.5)**	**S4**	2.10-2.20
sub-micrometric	**S3**	0.80-0.90
sub-micrometric	**S2**	0.40-0.45
ultrafine **(UFP)**	**S1**	0.13-0.15
-	**Back-up**	-

- Dyno-bench compliant to UN GTR n°24
- Several repetitions of WLTP-Brake cycle, until sufficient material is collected (min. 0.3 mg) for UFPs

Experimental: Sample Preparation & Analytical Probes

DGI	
Stage	**d50 / µm**
S4	2.10-2.20
S3	0.80-0.90
S2	0.40-0.45
S1	0.13-0.15
Back-up	-

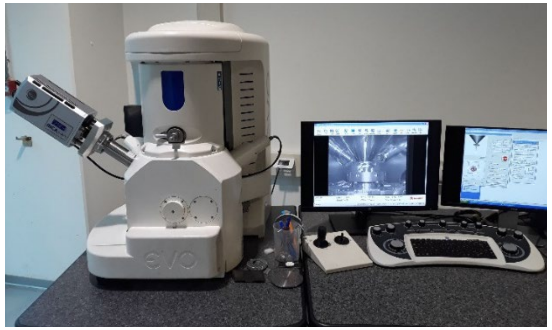


Solvent extraction

Centrifugation

Redeposition

GC-MS
Organic Compounds Determination



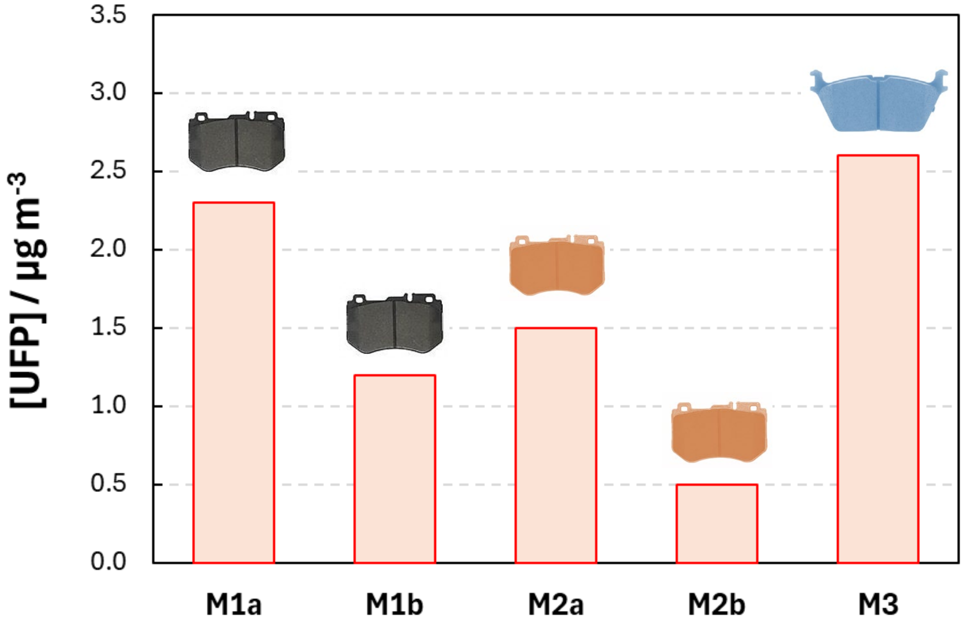
SEM/EDXS
Particle size & Semi-quantitative composition

XANES
(Speciation of *d-block* Metals)

Results: UFPs' Concentration

Friction Pair	UFP [mg]	Flux [$m^3 h^{-1}$]	Duration [h]	[UFP] [$mg\ m^{-3}$]	[PM10]* [$mg\ m^{-3}$]
M1a	1.46	5.76	109.8	**0.0023**	0.15-0.30
M1b	0.84	5.76	122.0	**0.0012**	
M2a	0.79	5.76	93.0	**0.0015**	
M2b	0.31	5.88	97.6	**0.0005**	
M3	1.00	5.76	67.1	**0.0026**	0.01-0.10

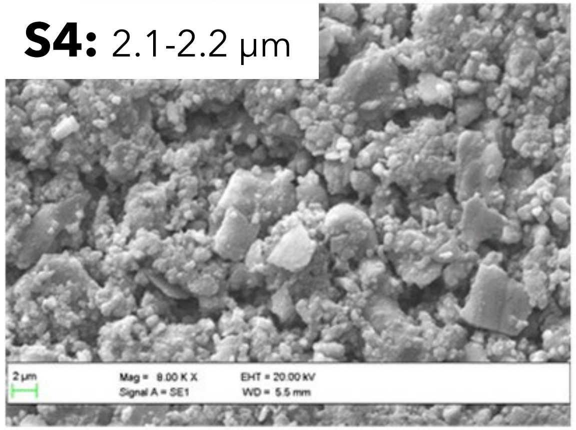
* averaged over corresponding emission tests on similar friction couples in identical experimental conditions



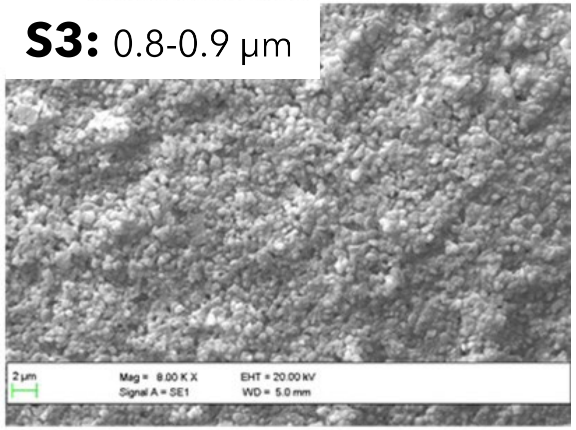
- Emission concentrations for UFPs in the order of µg m^{-3}
- M2b emits the lowest amount of UFPs (0.5 µg m^{-3}), M3 generates the highest concentration of UFPs (2.6 µg m^{-3})
- Lower PM10 emission of NAOs does not appear to be confirmed when assessing UFPs, reasonably explained by friction material compositional characteristics

Results: Morphology & Size Distribution

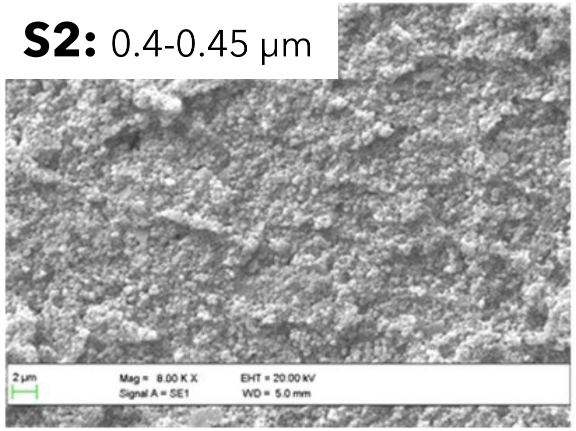
S4: 2.1-2.2 µm



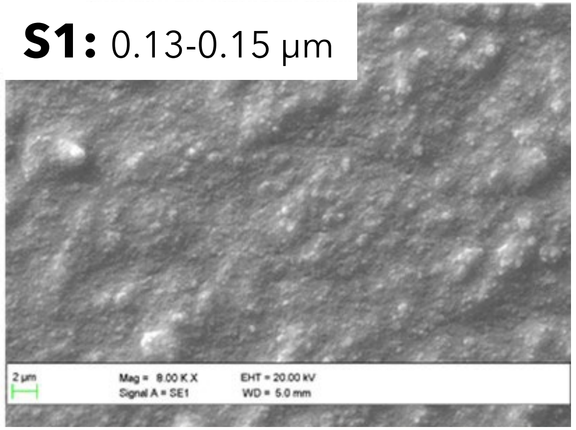
S3: 0.8-0.9 µm



S2: 0.4-0.45 µm

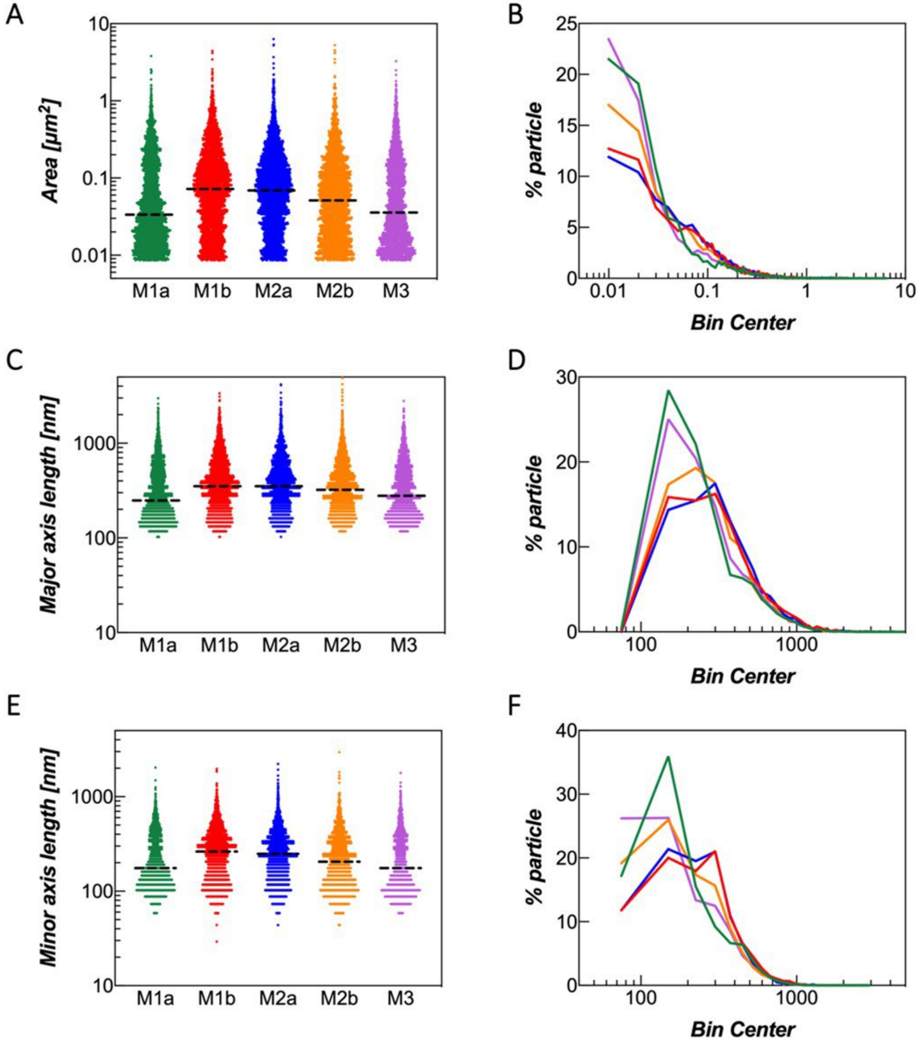


S1: 0.13-0.15 µm

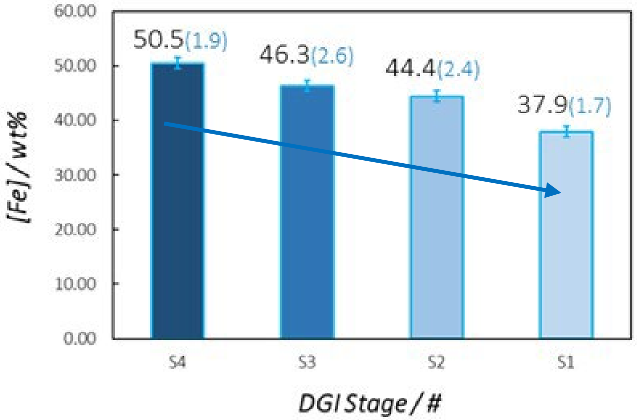


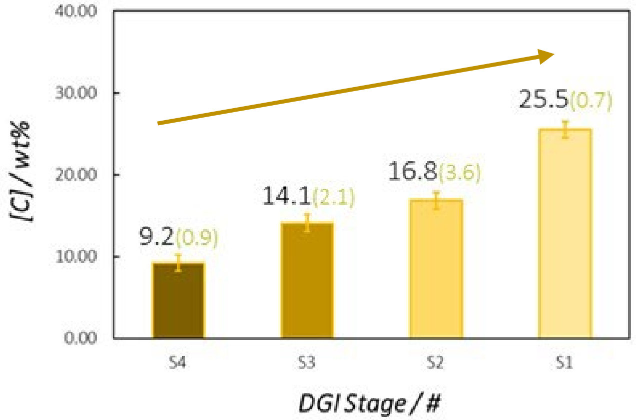
- Coherent size distribution for different stages
- M1a and M3 appear overall slightly finer on back-up filter

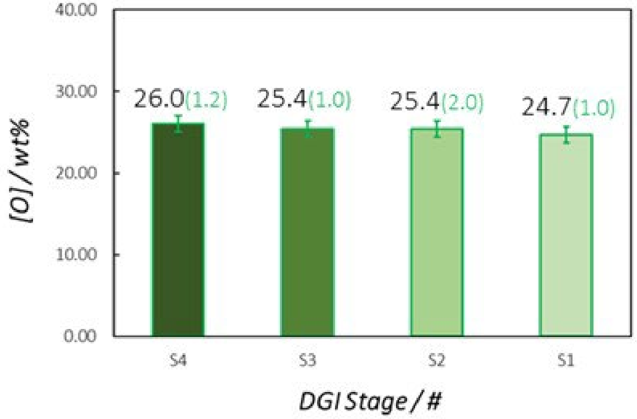
Back-up Filter

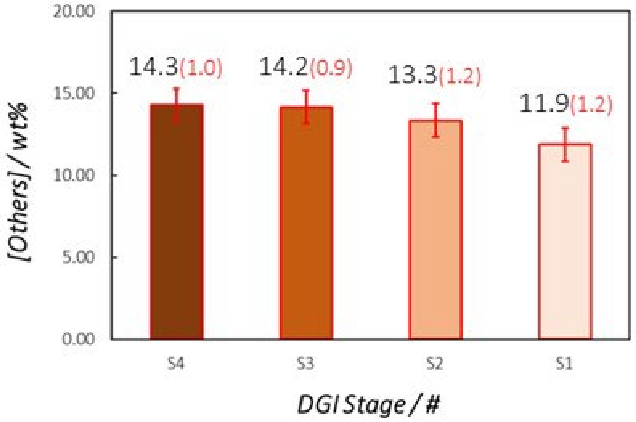


Results: Elemental Composition – M1b

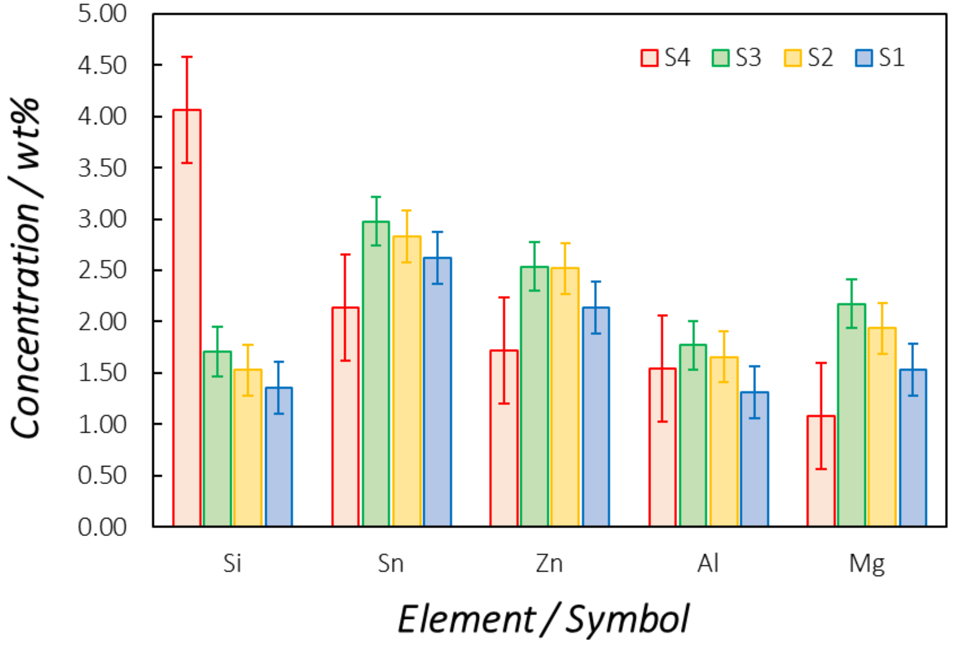






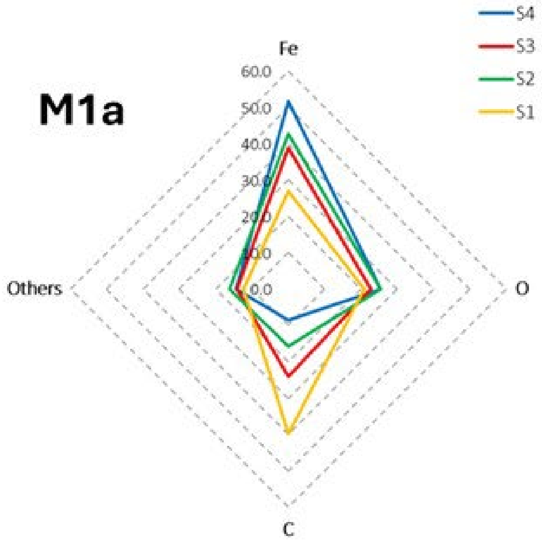


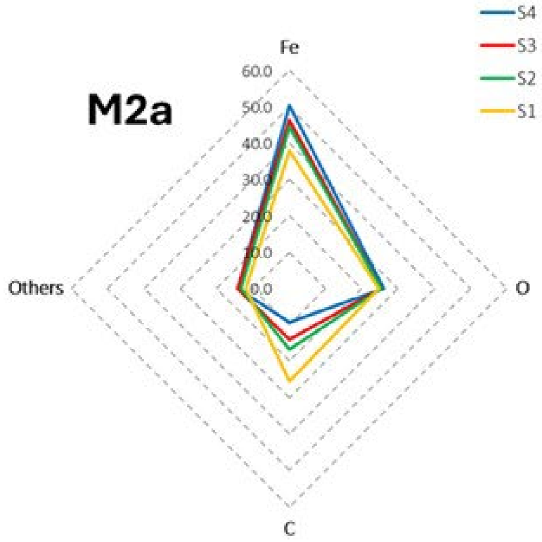
Secondary Elements

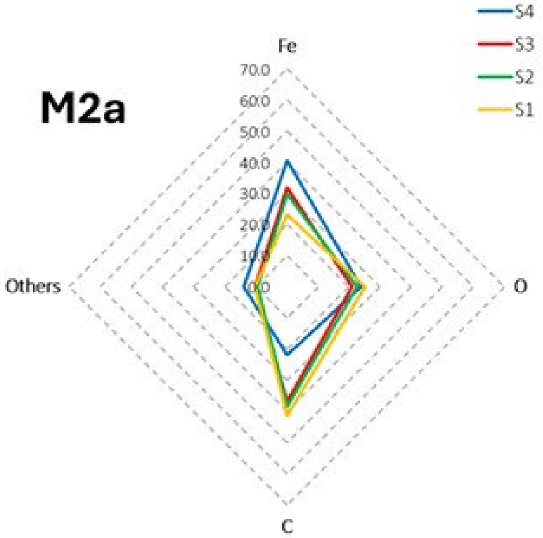


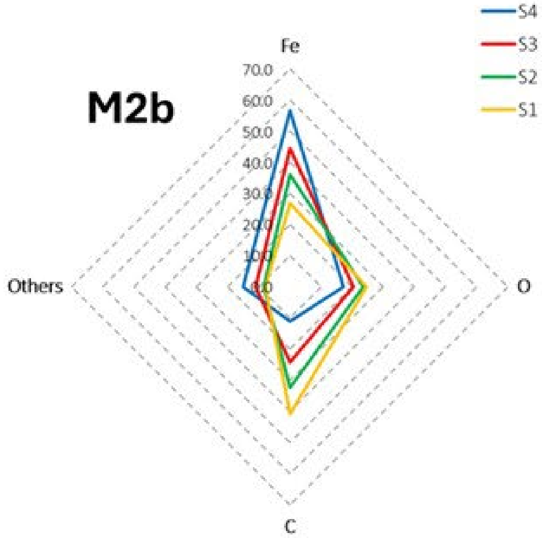
- Decreasing [Fe] and increasing [C] going towards finer fractions
- Decreasing Fe/O ratio going towards finer fractions (higher metal oxidation)
- Overall stable concentration of other elements (secondary & trace elements)

Results: Elemental Composition

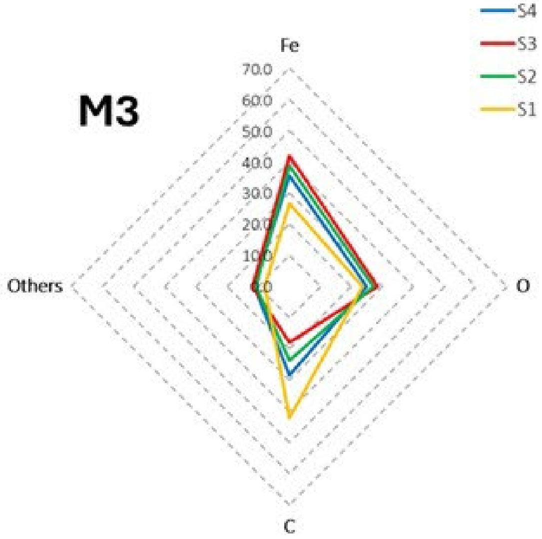

M1a


M2a


M2a

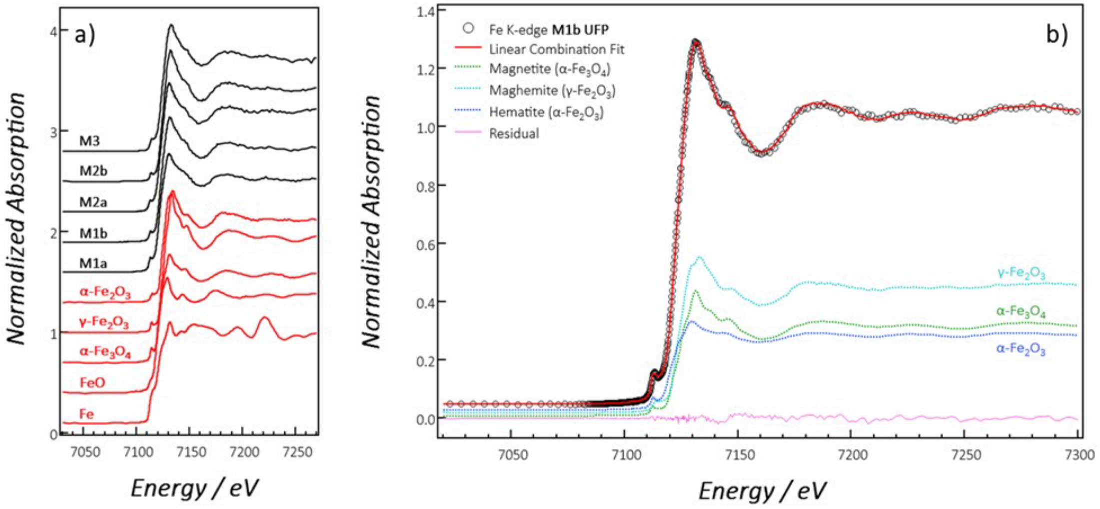

M2b

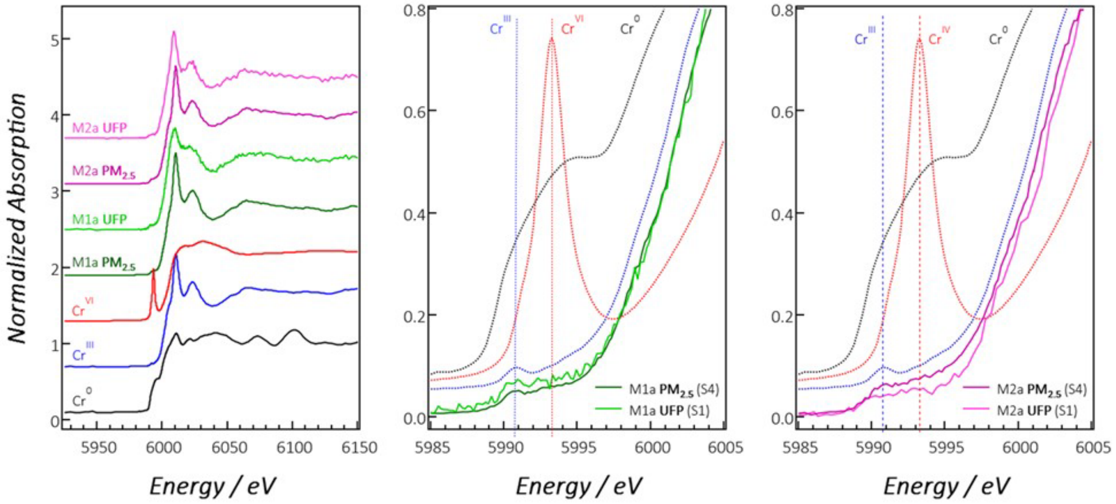
- ➢ General {trends} reproducible in all the five investigated particulates


M3

- Decreasing [Fe] and increasing [C] going towards finer fractions
- Decreasing Fe/O ratio going towards finer fractions (higher metal oxidation)
- Overall stable concentration of other (secondary elements)

Results: Phase Composition, *d-block* Metals

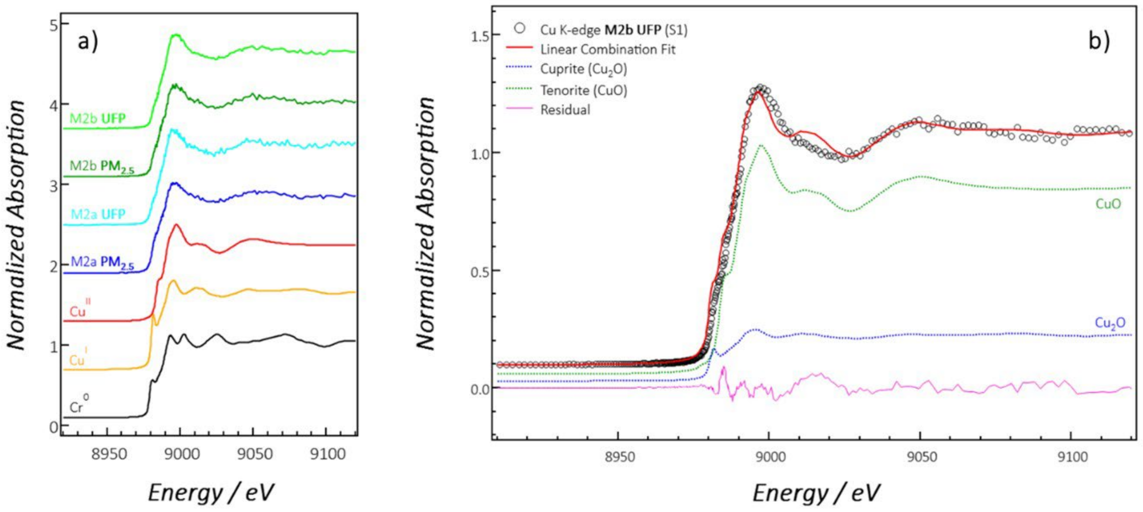


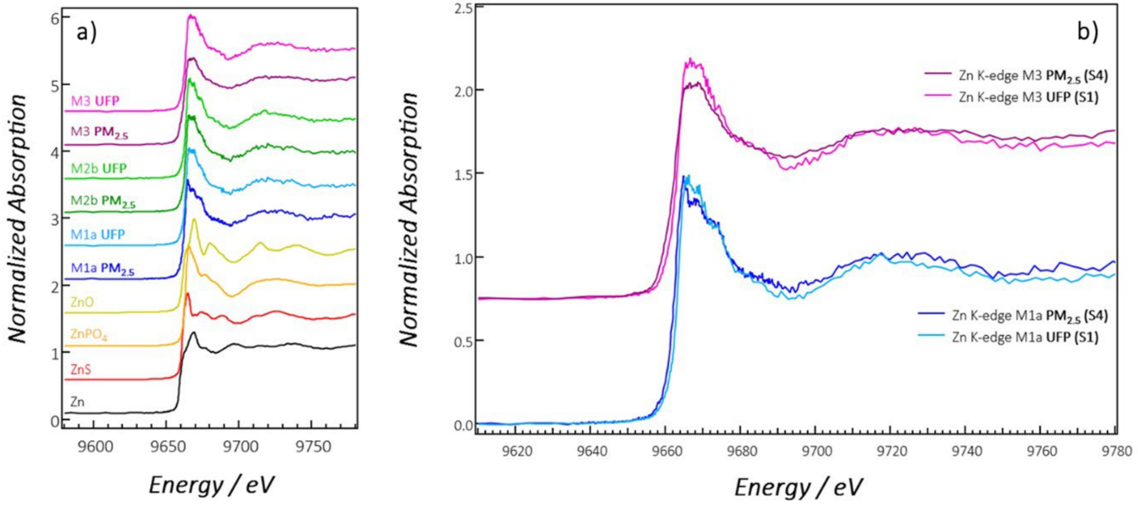


Sample / Phase (wt%)		Fe	FeO	α-Fe3O4	γ-Fe3O4	α-Fe2O3
M1a	**UFP**	0	10	68	22	0
	PM2.5	0	27	40	33	0
M1b	**UFP**	0	0	26	31	43
	PM2.5	11	38	30	21	0
M2a	**UFP**	0	0	18	50	32
	PM2.5	0	12	55	10	23
M2b	**UFP**	0	0	0	54	46
	PM2.5	0	0	41	20	39
M3	**UFP**	0	10	21	51	18
	PM2.5	0	0	41	57	0

- Magnetite (α-Fe_3O_4), Maghemite (γ-Fe_3O_4) and Hematite (α-Fe_2O_3) as dominant Fe bearing compounds
- Increasing oxidation of Fe moving towards finer dimensional fractions
- Chromium always found to exhibit +3 oxidation state: Chromite $FeCr_2O_4$ - as bearing compound

Results: Phase Composition, *d-block* Metals

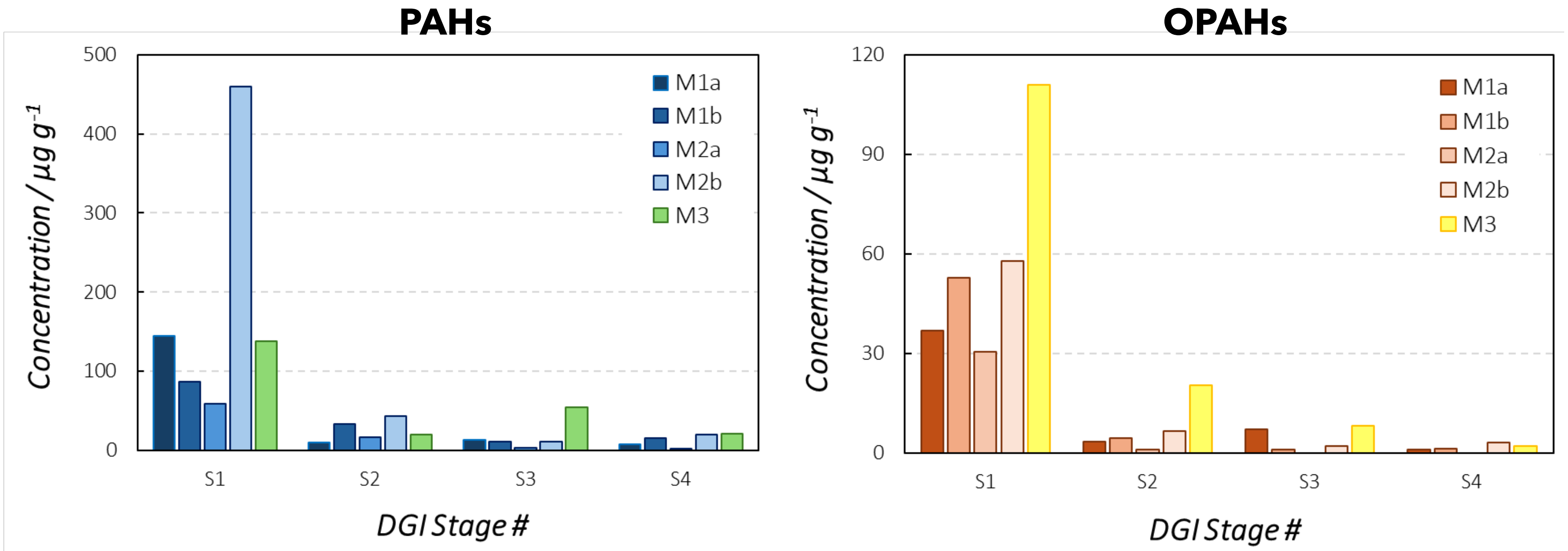




Sample / Phase (wt%)		Cu	Cu_2O	CuO
M2a	**PM2.5**	0	38	62
	UFP	0	30	70
M2b	**PM2.5**	0	21	79
	UFP	0	28	72

- Cu always oxidized, both Cuprite (Cu_2O) and Tenorite (CuO) as bearing compounds (ratio 1:2)
- Zn shows similar level of oxidation in both micrometric and nanometric particulates
- Several Zn bearing compounds: ZnS, ZnO and Zn-phosphates

Results: Organic Compounds

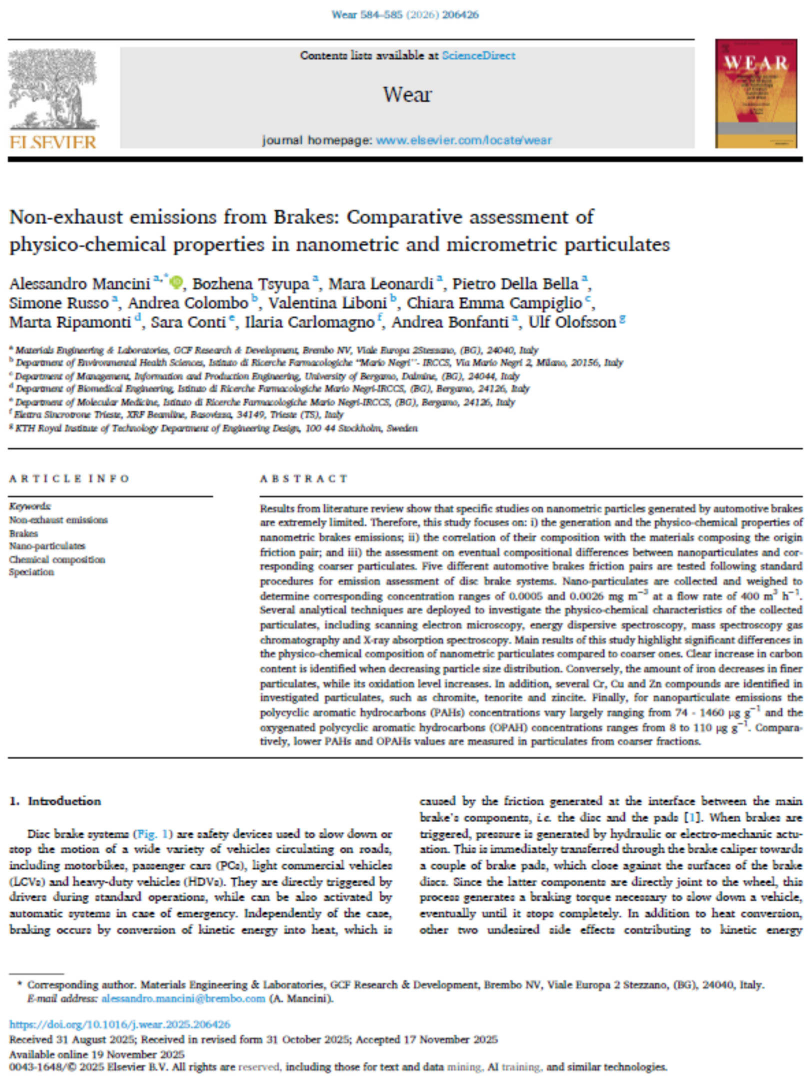

PAHs

OPAHs

- 24 PAHs and 9 OPAHs congeners found and measured, including lower molecular weight PAHs (LMW, containing 2/3-ring PAHs), middle molecular weight PAHs (MMW, containing 4-ring PAHs) and higher molecular weight PAHs (HMW, containing 5/7-ring PAHs)
- LMW and MMW –PAHs/-OPAHs contribute fore more than 80% of the total
- Comparatively, lower PAHs and OPAHs values are measured in particulates from coarser fractions, both sub-micrometric and micrometric.

Conclusions: Take-Home Messages

- Mass of brake-emitted nanoparticles three to four orders of magnitude lower compared to micrometric counterparts (under WLTP-Brake conditions)
- NAO friction materials do not appear to consistently reduce particulate emissions across all size fractions (similar level of nanoparticles than the highest emitters among the Low Steel / Low Met friction composites)
- The iron-to-oxygen ratio decreases progressively toward the finer fractions, *i.e.*, nanometric iron-based particles are more oxidized than their micrometric counterparts
- Carbon concentration increases progressively toward the finer fractions, *i.e.*, particles collected in the nanometric fraction show a higher concentration of carbon-based material (from brake pads)
- Several chemical compound are clearly identified in brakes nanoparticulates in addition to iron oxides, including: copper oxides, chromite, zinc sulphide and zinc oxides
- For nanoparticulate emissions the polycyclic aromatic hydrocarbons (PAHs) and oxygenated polycyclic aromatic hydrocarbons (OPAHs) concentrations vary largely: comparatively, lower PAHs and OPAHs values are measured in particulates from coarser fractions.



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Non-exhaust emissions from Brakes: Comparative assessment of physico-chemical properties in nanometric and micrometric particulates

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ABSTRACT

Results from literature review show that specific studies on nanometric particles generated by automotive brakes are extremely limited. Therefore, this study focuses on: i) the generation and the physico-chemical properties of nanometric brakes emissions; ii) the correlation of their composition with the materials composing the origin friction pair; and iii) the assessment on eventual compositional differences between nanoparticulates and corresponding coarser particulates. Five different automotive brakes friction pairs are tested following standard procedures for emission assessment of disc brake systems. Nano-particulates are collected and weighed to determine corresponding concentration ranges of 0.0005 and 0.0026 mg m^{-3} at a flow rate of 400 m^3 h^{-1}. Several analytical techniques are deployed to investigate the physico-chemical characteristics of the collected particulates, including scanning electron microscopy, energy dispersive spectroscopy, mass spectroscopy gas chromatography and X-ray absorption spectroscopy. Main results of this study highlight significant differences in the physico-chemical composition of nanometric particulates compared to coarser ones. Clear increase in carbon content is identified when decreasing particle size distribution. Conversely, the amount of iron decreases in finer particulates, while its oxidation level increases. In addition, several Cr, Cu and Zn compounds are identified in investigated particulates, such as chromite, tenorite and zincite. Finally, for nanoparticulate emissions the polycyclic aromatic hydrocarbons (PAHs) concentrations vary largely ranging from 74 - 1460 μg g^{-1} and the oxygenated polycyclic aromatic hydrocarbons (OPAH) concentrations ranges from 8 to 110 μg g^{-1}. Comparatively, lower PAHs and OPAHs values are measured in particulates from coarser fractions.

1. Introduction

Disc brake systems (Fig. 1) are safety devices used to slow down or stop the motion of a wide variety of vehicles circulating on roads, including motorbikes, passenger cars (PCs), light commercial vehicles (LCVs) and heavy-duty vehicles (HDVs). They are directly triggered by drivers during standard operations, while can be also activated by automatic systems in case of emergency. Independently of the case, braking occurs by conversion of kinetic energy into heat, which is caused by the friction generated at the interface between the main brake's components, *i.e.* the disc and the pads [1]. When brakes are triggered, pressure is generated by hydraulic or electro-mechanic actuation. This is immediately transferred through the brake caliper towards a couple of brake pads, which close against the surfaces of the brake discs. Since the latter components are directly joint to the wheel, this process generates a braking torque necessary to slow down a vehicle, eventually until it stops completely. In addition to heat conversion, other two undesired side effects contributing to kinetic energy

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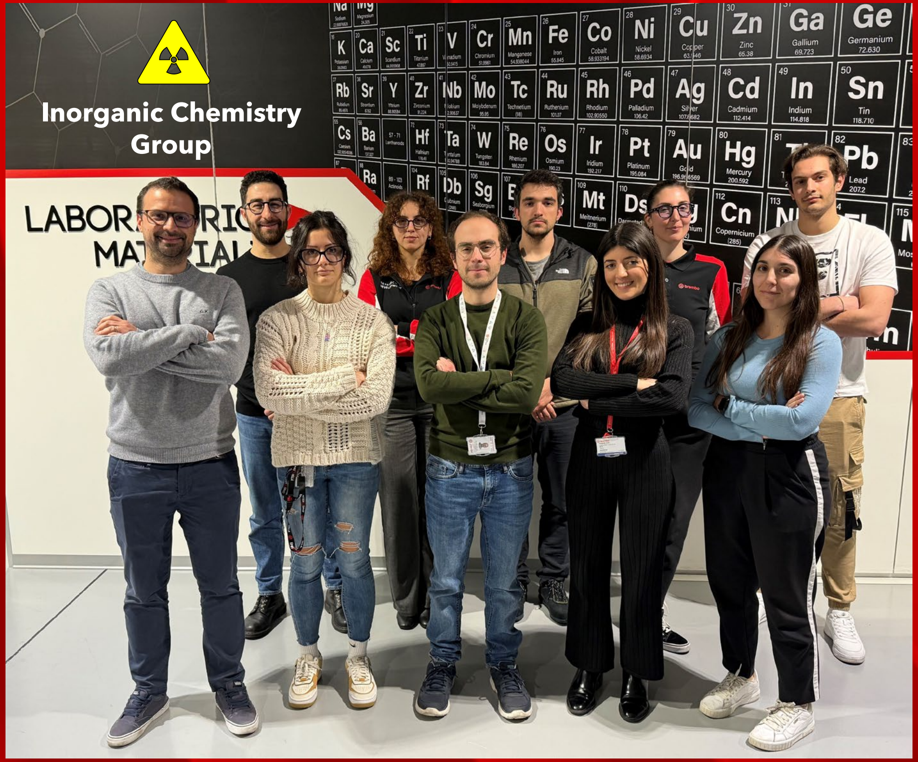

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THANK YOU