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# Characterization of Particulate Matter from Brake Wears

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**ABSTRACT**
The present study aimed characterising non-tailpipe emissions of particulate matter (PM) from passenger cars, namely brake wear particulates (BWP) in terms of their structure, morphology, mean particle size and chemical composition. For this purpose, BWP formed in the brake systems of three types of vehicles, denoted herein as: BS1, BS2, and BS3, were collected and analysed. The sampling procedure was conducted after a predefined mileage (about 50000 km) under uncontrolled (real life) operating conditions. The relative contents of carbon, hydrogen, and nitrogen were determined using ultimate analysis. The morphological features of the particles and their size distribution were assessed by means of scanning electron microscopy coupled with energy-dispersive spectroscopy (SEM/EDS). The obtained results revealed substantial variations in the particles' morphology, size distribution (mean particle size was between 16 and 74 μm) and chemical composition, among the BWP samples, collected from the investigated brake systems. The present investigation provided foundation for continued monitoring the physicochemical properties of the BWP, generated in field studies or under well controlled laboratory conditions. This in turn should enhance the evaluation of their impact on air quality and public health.

**KEYWORDS**
Characterization of brake system PM, PM from brake pads, non-tailpipe emissions of particulate matter (PM)

## 1. Introduction

Air pollution from the transport sector remains one of the leading sources of fine PM in urban environments. While regulatory efforts have historically targeted the reduction of exhaust emissions, a growing scientific consensus underscores the substantial role of non-exhaust emissions—in particular those from brake wear—in overall particle load and toxicological risk [1-3]. These emissions span a wide range of aerodynamic diameters ($PM_{\geq 10}$, $PM_{2.5}$ and ultrafine fractions with aerodynamic diameter below 1 micron - $PM_{\leq 1}$) and include metallic and organic constituents, whose relative abundances, morphology, and redox activity govern their reactivity and biological effects [1-4].

The composition and microstructure of BWP are highly heterogeneous and dictated by tribological regimes and material chemistry [5,6]. Electron-microscopic and spectroscopic investigations of brake system's discs and pads consistently reveal multiphase morphologies (metals and metal oxides, carbonaceous species, sulphides/sulphates), fragmentation, and the formation of tribofilms that control emission behaviour [7,8]. Comparative studies across nano- and micrometer-size fractions show metal enrichment and distinct formation and transport mechanisms between fractions, with implications for toxicity and atmospheric transport [9].

In urban settings, brake wear accounts for a significant share of transport-related PM emissions, with emission intensity depending on material formulation, mechanical and thermal load, driving style, and environmental conditions [1,10]. Electrification, while eliminating exhaust emissions, does not remove non-exhaust emissions; the higher vehicle mass and specific brake management in hybrid and electric vehicles can modify emission profiles and sustain the relevance of brake dust to air quality [2,11].

Once emitted, particles undergo atmospheric transformations - agglomeration, surface oxidation, interactions with the gaseous composition (often photochemical process), and incorporation into secondary aerosols - that alter their aerodynamic properties and potential toxicity; a substantial share of the load is deposited in roadside sediments and in urban drainage systems, with consequences for the environment and population's expression to eco-toxicity [7,12] and sustainable urban management [2,13,14]. Of particular concern is the ultrafine fraction (d < 100 nm), normally generated during high-temperature processes, including combustion [15], and/or thermal decomposition of organic binders; this fraction exhibits high mobility, large specific surface area, and enhanced potential for cellular penetration and oxidative stress [9,10,16].

From a materials engineering perspective, friction systems are complex composites (organic/inorganic matrix, fiber's, fillers, lubricating and abrasive phases, metallic additives) that balance competing requirements: mechanical stability, noise-vibration-harshness (NVH) behaviour, thermal resistance, cost, and environmental compliance. Contemporary studies show that metallic reinforcement morphology, crosslinking chemistry, and surface engineering (e.g., alumina-based coatings) can modify wear mechanisms and reduce emissions in terms of mass and/or particle number [17,18], while the structural–tribological response of the disc–pad pair remains decisive for emissions [19]. Methodologically, dynamometer testing is indispensable for controlled reproduction of braking regimes, NVH, and emissions, yet discrepancies with real-world conditions are often observed due to the influence of climate, load, traffic, and material aging [20]. These observations are corroborated by direct in situ measurements during real driving [16] and by tests following the Global Technical Regulation (GTR) methodology for brake particles, enabling more comparable assessments [21].

From a regulatory standpoint, international and European frameworks are gradually integrating non-exhaust emissions with mass-based (mg/km) and particle-number limits for brake systems, and with an emphasis on harmonized measurement methodologies and reporting [2,21]. This reorients industrial and scientific efforts toward low-emission formulations and validated cross-laboratory comparable protocols that are relevant to real traffic.

Concurrently, advances in instrumentation and modelling enhance the precision of assessments: new automotive particulate emission measurement systems improve laboratory and field metrology [22], while finite element modelling of the tribological contact supports the analysis of local pressure/temperature fields that govern wear and particle release [23]. Parallels with the morphology and reactivity of exhaust aerosols (e.g., soot) serve as a useful reference framework for characterization and risk assessment [14].

In this context, the present study aimed at characterizing three different friction materials used in real passenger cars' braking systems. The non-exhaust BWP samples were collected after operating the vehicles in normal mode and predefined mileage. The specimens were designated as BS1, BS2, and BS3. Using SEM/EDS and elemental analyses, the chemical profile, structure, morphology, and particle size distribution were investigated. The environmental relevance of the studied products was discussed with respect to current regulatory trends and methodologies [7,8]. The results address the following issues: (i) the relationships between break wear composition – tribological behaviour – emissions; (ii) the differences across formulations and wear mechanisms; (iii) the implications for the design of low-emission brake materials and the harmonization of evaluation protocols.

## 2. Materials and Methods

### 2.1. Materials Used

For the purposes of this study, a sampling procedure was implemented after a predefined vehicle mileage of about 50,000 km. The passenger cars were operated under standard, real life driving conditions. A standard mechanical sampling procedure was applied for collecting the BWP. In order to avoid potential conflicts of interest, the results were denoted using abbreviations derived from the term brake system, as follows: BS1, BS2, and BS3. From each brake pad, sections of approximately 1 × 1 cm were cut from the active friction zone. The samples were cleaned with isopropyl alcohol, mounted on aluminium stubs, and fixed using carbon adhesive tabs.

### 2.2. Elemental Analysis

Quartered samples of the PM (BS1–BS3) were separated and subjected to elemental analysis. Prior to the analysis, the samples underwent preliminary acid treatment followed by decomposition through melting and inductively coupled plasma optical emission spectrometry (ICP OES). The elemental composition was determined using automatic EuroEA 3000 analyser.

2.3. SEM/EDS Analysis and Elemental Mapping

The structure and morphology of the collected particles was investigated using the SEM-Zeiss EVO 10 apparatus, manufactured by Carl Zeiss Microscopy GmbH, Carl-Zeiss-Promenade 10, 07745 Jena, Germany. In addition, particles' elemental composition was analysed using EDS detector, precisely Oxford Instruments EDS Xplore 30, from Oxford Instruments, Halifax Rd, UK. The apparatus was operating at an accelerating voltage of 15–20 kV and a working distance of 10 mm. Micrographs were acquired at magnifications of ×500, ×1000, and ×2000.

Particle mean size was estimated based on the SEM images using ImageJ software package. For that purpose particle boundaries were manually defined, and the Feret diameter was determined for 100 to 150 particles analysed per sample.

Elemental composition was estimated based on the EDS analysis using an Oxford Instruments EDS detector at an accelerating voltage of 20 kV and a signal acquisition time of 60 s. The mass percentages of C, O, Fe, Si, Zn, Ca, Al, and Cl were quantified. The results were averaged for each sample (three spectra were collected per sample). The elemental distribution maps of the main constituents (C, O, Fe, Si, Zn, Ca, and Al) were generated to assess the morphology and spatial distribution of the metallic and organic phases. The elemental composition results are presented as mean values (±) standard deviation. The EDS data were evaluated using graphical comparisons and tabulated data in order to identify differences among the investigated brake pad types.

## 3. Results and Discussion

The present study represents an experimental investigation of chemical composition, structure and morphology of the investigated BWP.

### 3.1 Elemental composition

The relative contents of carbon (C), nitrogen (N), and hydrogen (H) was determined by elemental analysis (according to the methodology, described in paragraph 2.2). The obtained results (see Table 1) correspond to the individual indicators and represent mean values from three parallel determinations for each type of brake system (BS) sample.

Table 1. Elemental analysis of the BS1, BS2 and BS3 samples

| Parameters Measured | Elemental Composition, (wt %) | | |
|---|---|---|---|
| | BS 1 | BS 2 | BS 3 |
| Carbon (C) | 11.38 | 12.09 | 12.77 |
| Nitrogen (N) | 0.67 | 0.73 | 1.07 |
| Hydrogen (H) | 5.33 | 6.15 | 6.49 |

The elemental compositions of the investigated brake wear samples (BS1–BS3) showed carbon content ranging from 11.38–12.77 wt %, hydrogen from 5.33–6.49 wt %, and nitrogen from 0.67–1.07 wt %. These values fall within the range reported for carbonaceous fractions of brake wear particles in previous studies [24-26]. Quantitatively, the fraction of C, H and N slightly increases in the order BS1 < BS2 < BS3. For example, Garg et al. [24] report substantial carbon contributions in airborne brake wear particles, highlighting the importance of organic binder degradation in particle formation. These quantitative trends support the interpretation of a more favourable emission profile for materials with higher organic content, to be further corroborated by SEM/EDS observations.

### 3.2. Particle Morphology and Size

The SEM images revealed distinct morphological differences between the three analysed friction material samples (Figure 1).

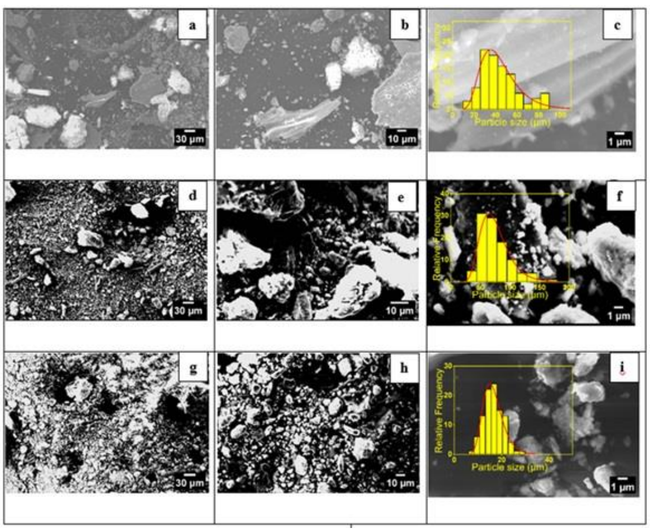



Figure 1. SEM images of: (a-c) BS1; (d-f) BS2; (g-i) BS3

The surface of BS1 exhibited a relatively fine and homogeneous structure, consisting of a compact organic matrix with uniformly dispersed small fillers and individual Fe and Si micro-particles. The average particle size was 40.40 ± 1.74 µm, which is typical for the non-asbestos organic (NAO) friction materials [25]. Such a structure favors smooth braking and reduced wear, but is usually accompanied by lower thermal resistance [26].

The results for the BS2 demonstrated a coarser and more porous morphology, with numerous metallic aggregates and a more pronounced texture. The measured average particle size was 74.04 ± 2.51 µm, indicating a predominant metallic phase with oxidized inclusions. This structure is characteristic of semi metallic pads, which is associated with high durability, but propensity for generation of larger number of abrasive particles during friction [27]. The presence of large metallic particulates and a heterogeneous structure suggested an increased risk of $PM_{10}$ and $PM_{2.5}$ emissions during brake system operation. The large number of particles observed in previous independent wear tests was attributed to condensation [24,28].

The surface of BS3 was characterized by a much finer and more uniform particle distribution, with an average size of 16.57 ± 0.41 µm. The SEM images indicated smooth transitions between organic and metallic phases, with no evidence of large agglomerates [28]. Accordingly, the observed morphology suggested similarity to hybrid or low metallic friction materials, in which the addition of small amounts of metallic fillers enhances thermal conductivity and friction performance without markedly increasing particulates generation. The determined relationship between the metal phase content and particle morphology supported the conclusion that, as the proportion of metals (Fe, Zn) increases, the structure becomes coarser and the potential for the formation of fine PM due to abrasion increases.

The average particle size was determined from the SEM micrographs using the ImageJ software (NIH, USA). Between 100 and 150 particles were measured for each sample, and the results were reported as mean ± standard deviation (µm). The mean particle sizes for all investigated samples were summarized in Table 2.

Table 2 Average particle size for BS1, BS2 and BS3, calculated from the SEM images

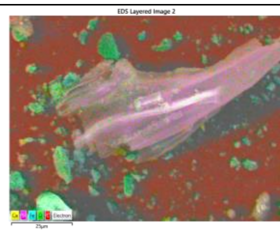
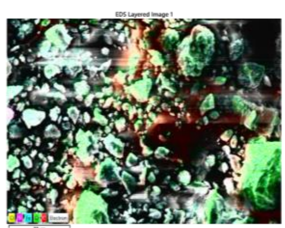
| Sample | Particle size ± SD (µm) |
|---|---|
| BS1 | 40.40 ± 1.74 |
| BS2 | 74.04 ± 2.51 |
| BS3 | 16.57 ± 0.41 |

The average size confirmed the following relation: BS2 > BS1 > BS3. Thus, it indicated that the increased metal content led to larger particles and potentially higher PM emissions.

### 3.3. Particle Chemical Composition and EDS mapping

The results from the EDS analysis (Table 3) confirmed substantial differences in the elemental composition among the three investigated brake system samples of particulates.

Table 3. The EDS analyses of the BS1, BS2 and BS3 samples

| Sample | C (wt %) | O (wt %) | Key elements (wt %) | Mapping |
|---|---|---|---|---|
| BS1 | 63.17 | 26.37 | Si (4.7) Fe (5.12) |  |
| BS2 | 40.03 | 23.26 | Fe (30.99) Zn (3.00) Cl, Ca, Si and Al ~ 1.5 |  |
| BS3 | 49.93 | 28.93 | Fe (19.23) Si, S, Ca, Cl and Al < 0.5 |  |

The BS1 samples were dominated by carbon (C – 63.17 wt %), while the metals Fe (5.12 wt %) and Si (4.70 wt %) were present in low amounts. These observations confirmed that the examined material represents an organic friction layer with a limited content of inorganic and metallic fillers [28].

The BS2 samples showed considerable proportion of Fe (30.99 wt %) and Zn (3.00 wt %), with lowest C and O relative content. Such metal concentrations are indicative for semi metallic material, in which metallic inclusions play a major role in frictional resistance, but also contribute to the potential air pollution.

In BS3, the Fe content was moderate (19.23 wt %), representing an intermediate value between BS1 and BS2, while C and O were 49.93 wt % and 28.93 wt %, respectively. These ratios were characteristic of hybrid materials that combine mechanical strength with enhanced environmental compatibility [27,29]. The EDS spectra and elemental distribution maps revealed a clear distinction between organic (C/O) and metallic (Fe/Zn/Si) phases. The BS2 exhibited high concentration of metallic aggregates, whereas BS3 showed metals that were more finely dispersed and uniformly distributed, suggesting a milder wear process and a lower release of metal rich BWP.

The results from the EDS mapping (Table 3) visualized the spatial distribution of the major elements in the three samples. In BS1, the elements like C and O covered the surface evenly, while Fe and Si appeared as separate small zones. This was an indication of good homogeneity and an organic-dominated structure. In BS2, Fe and Zn formed dense regions, confirming the predominant metallic nature of the material. Such morphology typically leads to a higher potential for the separation of metal particles upon friction. In BS3, the Fe was evenly distributed in the organic matrix, while Si and Al were finely dispersed. This combination is expected to provide balance between mechanical resistance and environmental profile [29].

### 3.4. Ecological interpretation

The obtained morphological and chemical data provoked a discussion about the potential emission risk of the friction materials during regular breaker wear system's operation under real life conditions (see Table 4). Particular attention was paid to the content of carbon, inorganic and non-metallic (e.g. chlorine, sulfur) and major metal components (e.g. Fe, Zn) and the particle sizes, and morphology, as such particles were expected to have direct impact on the formation of PM10 and PM2.5 fractions under open air atmospheric conditions. Details were presented in Table 4. The data were interpreted in accordance with current scientific evidences [1] and with the requirements of the Euro 7 regulation (Regulation (EU) 2024/1257), which for the first time introduces limits for brake emissions in mg/km. Accordingly, materials with low Fe, Zn and Cu content and fine morphology were considered the most favorable.

Table 4. Comparative characteristics of the investigated brake system samples (BS1–BS3)

| Parameter | BS1 (Cherry) | BS2 (Fabia) | BS3 (Škoda Fabia) |
|---|---|---|---|
| Mean particle size (µm) | 40.40 ± 1.74 | 74.04 ± 2.51 | 16.57 ± 0.41 |
| Dominant elements (wt %)C | C, O, Si | Fe, Zn, C | C, O, Fe |
| Material type | Organic (NAO) | Semi-metallic | Hybrid Structural |

| | | | |
|---|---|---|---|
| Structural homogeneity | High | Low | Very high |
| Expected emissions ($PM_{10}$/$PM_{2.5}$) | Low | High | Low–moderate |
| Euro 7 compatibility | High | Low | Excellent |

From this perspective, the following global conclusions were drawn:

- BS1 – showed low emission potential and thermal resistance;
- BS2 – higher durability, but also higher emission potential;
- BS3 – well-balanced between the two parameters, suggesting higher environmental compatibility.

In the context of the Euro 7 standard, the BS3 demonstrated the effects of employing brake pads with reduced unburned emissions and enhanced environmental compliance, positioning it as promising option among the analysed samples.

## 4. CONCLUSION

The present work aimed presenting a laboratory approach, aiming to characterise the non-exhaust PM generated by the wear of different automotive brake systems (the so called BWP), using a combined approach that includes elemental analysis and SEM/EDS microstructural investigations. The results indicated that higher C content was characteristic of organic and hybrid materials, whereas semi metallic pads exhibited a lower proportion of organic constituents. The conducted SEM/EDS analyses demonstrated that the chemical composition of brake components has a direct impact on the morphology and potential emission risk of friction materials. Organic and hybrid brake pads (BS1 and BS3) were distinguished by a finer and more homogeneous structure, a more uniform distribution of fillers, and a lower metal content, which promoted them as environmentally compatible and sustainable materials in accordance with the current environmental standards. Among the three investigated materials, BS3 was identified as the most balanced type, combining good mechanical wear resistance with a low potential for harmful emissions.

The present study confirmed that advanced electron microscopy is a powerful micro-analytical and non-destructive technique, capable for providing essential understanding of key chemical and physical properties of break wear PM in automotive braking systems. The chemical composition and microstructure of friction materials is being considered a key factor for studying the reducing non exhaust emissions.

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