Evaluation of methods for characterizing particulate matter emitted by brake systems

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ABSTRACT

Particulate matter (PM) emissions from brake systems pose significant public health risks due to their association with respiratory and cardiovascular diseases. While Scanning Electron Microscopy (SEM) is a well-established method for PM analysis, it is expensive and requires specialized technical expertise. This study presents a comparative analysis of optical microscopy and SEM for particle counting. Experiments were conducted using a full-scale inertial dynamometer with commercial brake components. For optical microscopy, PM was extracted from filters using mechanical vibration, and the resulting images were processed automatically using ImageJ software. SEM images were analyzed manually using the same software. The comparison revealed substantial differences in the detection of fine and ultrafine particles between the two methods.

Keywords: Brake system; Particulate matter; Microscopy; SEM; Particle counting.

INTRODUCTION

The increasing demand for higher load-carrying capacity and greater vehicle speeds has led automotive engineers and designers to pursue more sustainable solutions in the automotive industry. Particulate matter (PM) emissions from vehicles have become a growing concern due to their contribution to air pollution.

In Brazil, it is estimated that PM-related deaths may reach 250,000 between 2012 and 2030, considering only the state of São Paulo. In Europe, air pollution is responsible for 80% of premature deaths related to cardiovascular diseases and strokes. Beyond contributing to air pollution, a major risk associated with PM lies in its toxicity, which varies depending on its source composition [1-3].

Vehicle-emitted PM can be categorized based on its source as combustion-related or non-combustion-related. Non-combustion emissions generally arise from tire, road, or brake wear. One of the main ways to categorize PM is by its aerodynamic diameter, ranging from PM_{10} (up to 10μm), $PM_{2.5}$ (up to 2.5μm), $PM_{1.0}$ (less than 1μm), and ultrafine PM (less than 0.1μm). The aerodynamic diameter is defined as the diameter of a sphere with a density of 1 g/cm³ used to describe how a particle behaves as it moves through the air. It does not represent the actual physical size of the particle, but its aerodynamic behavior. Brake systems are responsible for 55% of PM with aerodynamic diameters smaller than 10 μm and 21% of PM with diameters below 2.5 μm [4]. The friction between brake pads and the disc leads to component wear and consequently to PM emissions [5-6].

Given increasing transport demands and the global effort to limit environmental impact from the automotive sector, several institutions have been working to implement new regulations and targets to reduce PM emissions—such as the upcoming Euro 7 regulations, expected to take effect in 2030 [7]. In this context, advancing studies on PM emission reduction and monitoring from brake systems, as well as identifying suitable methodologies for PM collection and analysis, has become essential.

Scanning Electron Microscopy (SEM) is one of the most well-established techniques for PM analysis, capable of generating nanometric-scale images. Despite its robustness, SEM remains expensive, with limited availability, and requires highly trained personnel for its operation [8].

ImageJ is a widely used computational tool for processing and quantifying particles in images obtained via both SEM and optical microscopy. Prior to analysis, images must be converted to binary format. For the purpose of aerodynamic diameter estimation, particles were assumed to be spherical. ImageJ offers two thresholding approaches: manual and automatic. The automatic thresholding method enables particle detection and counting without user intervention, while the manual approach allows users to adjust pixel intensity values as needed [8,9].

In addition, ImageJ includes a watershed function, used to separate overlapping particles, and a thresholding function that distinguishes particles from the background based on pixel intensity. This separation is based on the calculation of an Euclidean distance map, identifying erosion points, which are then dilated until they reach the edge of a particle or intersect with another growing region [8].

This study aims to contribute to the advancement of research on PM counting methodologies. Specifically, it presents a comparative analysis between results obtained through optical microscopy and Scanning Electron Microscopy (SEM) for PM quantification.

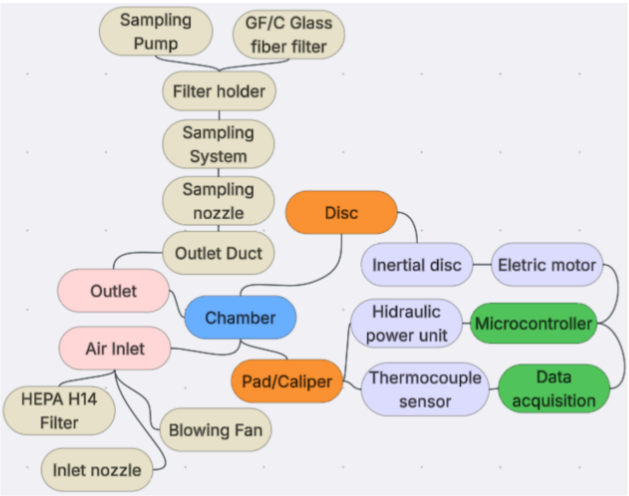
METHODS

The collection and extraction of particulate matter were carried out using the test bench developed by the Acoustics and Vibrations Laboratory at the Universidade Federal de Santa Catarina - Centro Tecnológico de Joinville.

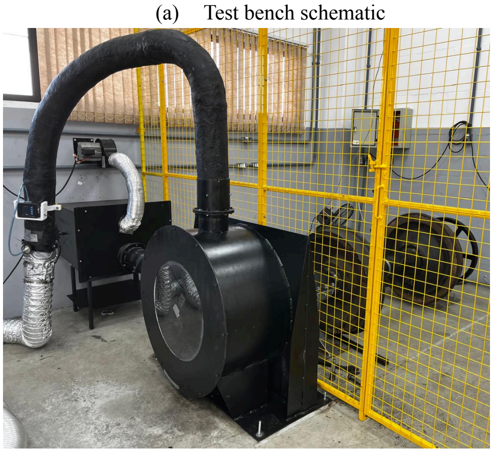
Collection and extraction of particulate matter (PM)

For the analyses conducted using optical microscopy and Scanning Electron Microscopy (SEM), particulate matter was collected from a test bench. The test bench consists of an inertial dynamometer equipped with a inertia disc and an enclosure chamber surrounding the brake system. The chamber features a controlled and filtered airflow. Figure 1(a) shows the schematic model of the test bench, indicating its stages and components. Figure 1(b) presents a photo of the test bench installed in the laboratory where the experiments were conducted.

Figure 1 - Test bench schematic (a) and Particulate matter collection system (b)



(a) Test bench schematic



(b) Particulate matter collection system
Source: Authors, 2025.

The tests were designed based on parameters representative of the current automotive market, as inertia. To ensure relevance, materials comparable to those used in light-duty vehicles were selected. A mid-size SUV was chosen as the reference model for this study.

The collection system was designed according to [6], comprising the brake system enclosure chamber, a filtration system prior to the chamber, and piping to connect the system. The sampling duct presents geometric limitations according to [7], where the diameter used was 4 mm, representing 1.78% of the cross-sectional area of the outlet duct, which has a diameter of 30 mm. These limitations are applied to allow the collection of particles smaller than 10 micrometers. To ensure proper airflow through the system, a sampling pump was used, providing a controlled flow rate in the range of 1 to 5 liters of air per minute and capable of delivering up to 10 kPa of suction pressure [8].

The initial speed applied during the tests was equivalent to 70 km/h in a real vehicle with a tire model 215/65 and rim 16, while the final speed was 30 km/h.

Before initiating the pressure cycles, a bedding procedure was carried out to standardize the friction material. Following this, tests were performed under a pressure of 30 bar, with a average temperature of the brake pad of 130 °C and a total of 500 braking events. The pad temperature was monitored using a thermocouple installed directly on the brake pads. Two filters were collected during the tests for optical microscopy analysis, and two additional filters were collected for Scanning Electron Microscopy (SEM) analysis. Figure 2 shows a filter with particulate matter deposited on its surface.

Figure 2 - Filter after collection

Source: Authors, 2025.

The cycle parameters are shown in Table 1, where Ri (RPM, Revolutions Per Minute) represents the initial rotation, R*f* (RPM) represents the final rotation, T*brake* (°C) represents the temperature of brake pads, and Qtd *fren* (-) represents the number of braking events. The pressure was controlled by a pressure switch connected to the hydraulic power unit.

Table 1 - Test Parameters for Each Cycle

Cycle	R*i* (RPM)	R*f* (RPM)	Pressure (bar)	T*brake* (°C)	Brake applications
Bedding	550	0	15	⩽90	200
P2	550	0	30	⩽130	500

Source: Authors (2024).

After the cycles, the filters were analyzed using optical microscopy and Scanning Electron Microscopy (SEM), with the aim of comparing the two techniques. For each analysis, different filters were used, repeating the same process during the cycles. The filter corresponding to the settling cycle was not analyzed, as it serves only to adjust the leveling of the pads. In both analyses, the obtained images were processed using the ImageJ computational software.

This approach was chosen due to one of the most common classifications of PM, which categorizes particles by sizes up to 10.0 µm, 2.5 µm, 1.0 µm, and ultrafine particles.

Optical microscopy

For particulate matter counting through optical microscopy analysis, the collected filters were placed on clean glass slides, similar to the method presented by [10]. The environment was maintained at 19°C during the extractions. Particulate matter was transferred to the slides via mechanical vibrations induced by a speaker placed on the filter.

The speakers were connected to a frequency generator with predefined values, decreasing in increments of 2,000 Hertz, starting from 15,000 Hz. The filter was exposed to each of the frequencies for five minutes. Afterward, the slides were transferred to the microscope, where images of three different regions were captured at a magnification of 200x.

The image processing was performed using the ImageJ software with a thresholding mechanism, which involves image treatment for particulate matter counting. The software uses the "Analyze Particles" function, which counts the number and area of the particles present in the image. The fiberglass present in the images was excluded from the count, as it was distinguishable by the circularity of the particles.

Using the "Analyze Particles" function, the aerodynamic diameter of the particles was calculated from their surface area, considering them as spheres with the same behavior in Brownian motion for approximation of their shape. The distribution and standard deviation were calculated and presented in graphical form.

Scanning electron microscopy (SEM)

Particulate matter counting using Scanning Electron Microscopy (SEM) is widely employed to quantify particles, providing information on particle size, shape distribution, and morphology at the nanometric scale. The analysis can also reveal information about particle origin, which is often not accessible through basic analyses [13-15].

The collected filters had their centers cut out where particulate matter is most concentrated and were mounted on carbon plates for stability. The filters then underwent gold sputter coating, a technique in which samples are coated with a thin layer of gold to enhance image quality and allow for surface structure analysis. Afterwards, the filters were analyzed under the scanning electron microscope, with images captured at 5,000x magnification.

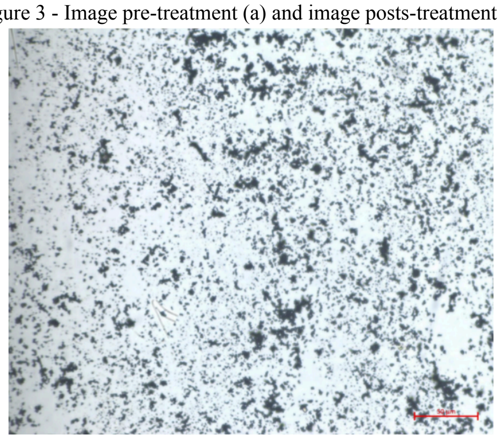
The SEM images were processed using ImageJ computational code. An manually thresholding mechanism was applied, along with the watershed function. To minimize errors, the auto-threshold function was used.

The Analyze Particles function was employed, as in the optical microscopy analysis, excluding fiberglass particles by identifying their distinct shape. The data obtained were processed through an algorithm capable of calculating the aerodynamic diameter of the particles. Their distribution and standard deviation were calculated and presented in graphical form.

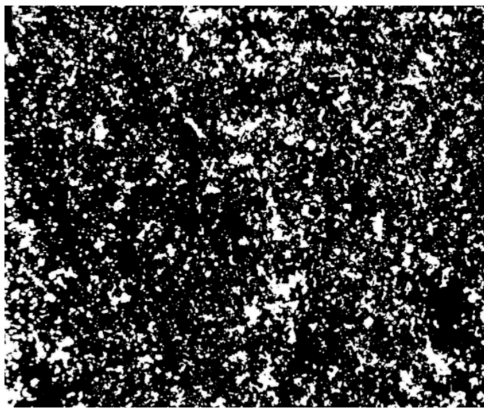
RESULTS

Using the optical microscopy method, six images were obtained and processed with ImageJ software. Figure 3 shows the images at 200x magnification before and after processing in ImageJ.

Figure 3 - Image pre-treatment (a) and image posts-treatment (b)



(a) Image pre-treatment

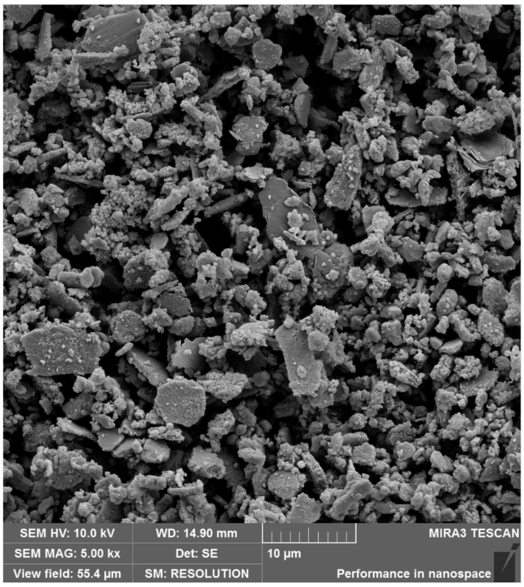


(b) Image post-treatment

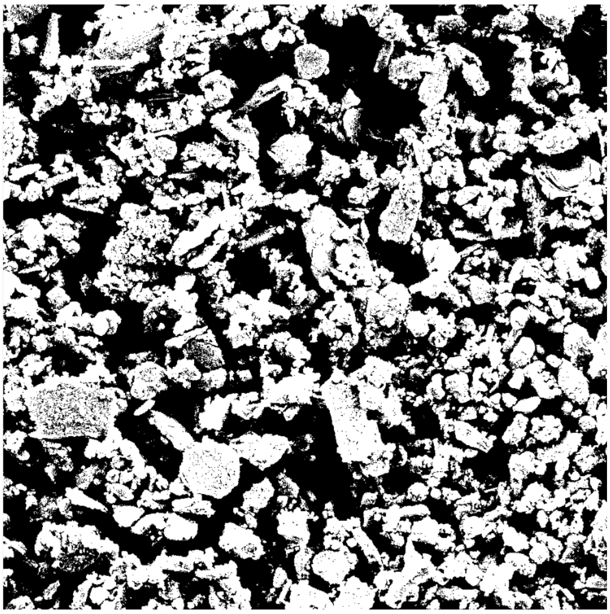
Source: Authors, 2025.

In the method using Scanning Electron Microscopy (SEM), six images were obtained for each filter—two images per region, with three regions analyzed per sample. For each of the three filters, images were acquired at 5,000x magnification. Figure 4 shows the 5,000x magnified SEM images before and after processing in ImageJ.

Figure 4 - Image pre-treatment (a) and image posts-treatment (b)



(a) Image pre-treatment



(b) Image post-treatment

Source: Authors, 2025.

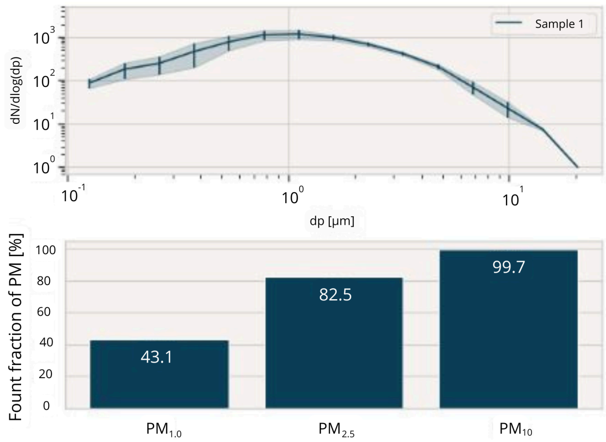
A significant visual difference can be observed between the images, with the SEM images appearing sharper and more detailed compared to those obtained through optical microscopy.

Average distribution and particulate matter percentage

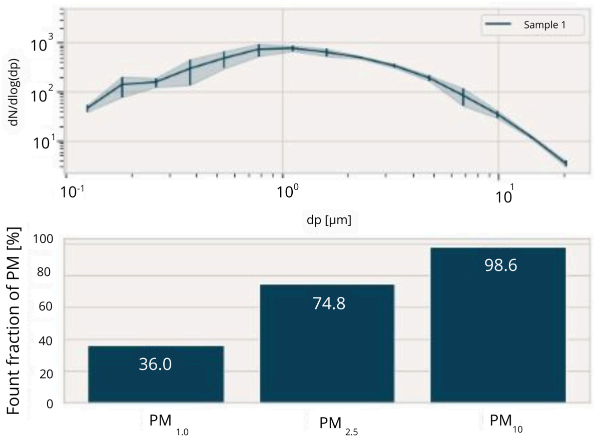
After image processing, the images were analyzed by an algorithm to calculate the aerodynamic diameter of the particles and to generate graphs of their standard deviation and distribution. Two graphs were produced for the analyses conducted using optical microscopy—one for each collected filter.

The graphs generated from the filters analyzed via optical microscopy indicated a peak in particulate matter concentration in the $PM_{1.0}$ range, as shown by the peak in the dN/dlog(d_p) graphs (normalized particle number distribution) versus d_p [µm] (aerodynamic diameter), as shown in Figure 5.

Figure 5 – Filter 1-OP (a) and filter 2-OP (b)



(a) Filter 1-OP



(b) Filter 2-OP

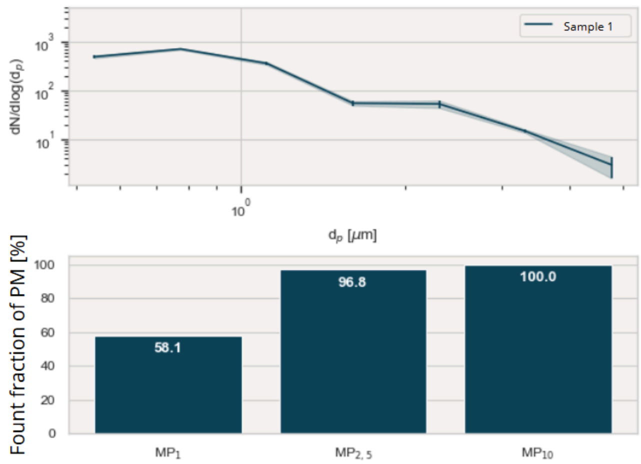
Source: Authors, 2025.

The percentage of particulate matter (PM) according to its aerodynamic diameter showed an average of 43.1% and 36.0% for $PM_{1,0}$, 82.5% and 74.8% for $PM_{2,5}$, and 99.7% and 98.6% for PM_{10} across the two collected filters. The percentage of PM by aerodynamic diameter was calculated as the cumulative sum of the proportion for a given aerodynamic diameter and the percentages of all finer particle sizes. The variation in the percentage of particulate matter between the graphs can be explained by the particle extraction method, which results in particle losses during the process. Moreover, fine particles are more susceptible to loss during extraction, which explains the 7.1% variation observed between the graphs.

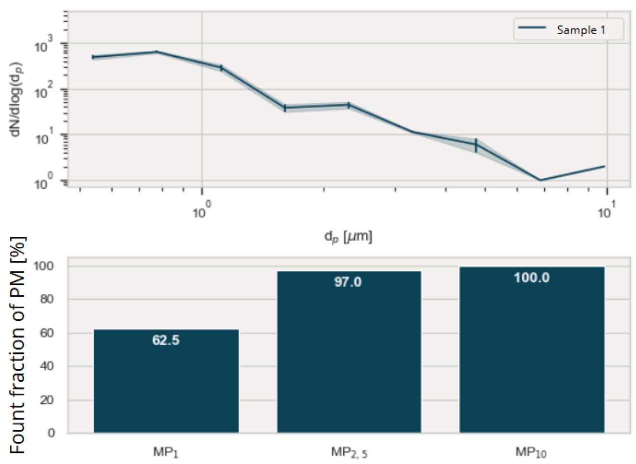
Based on this calculation method, it is evident that the highest emission rates are associated with finer particles ($PM_{1.0}$), which are among the most harmful to human health and the environment. Particles with aerodynamic diameters equal to or smaller than 1.0 µm are inhalable and capable of reaching sensitive systems, such as the respiratory system—penetrating deep into the pulmonary alveoli—and the circulatory system [10].

Figure 6 presents the graphs obtained from the scanning electron microscopy (SEM) images. These graphs show behavior distinct from those generated by optical microscopy analysis, indicating a greater presence of particulate matter with aerodynamic diameters smaller than 1.0 µm, thus classified as ultrafine particles. The graphs show the two filters at 5,000x magnifications in sample Cycle P2.

Figure 6 – Filter 1-SEM (a) and Filter 2-SEM (b)



(a) Filter 1-SEM



(b) Filter 2-SEM

Source: Authors, 2025.

From the SEM graphs, it is possible to observe that the percentage of particulate matter classified as $MP_{1,0}$ is significantly higher compared to the percentage of $MP_{1,0}$ in optical microscopy analyses, being more than double its value.

This fact can be explained by the loss of fine and ultrafine particulate matter during the extractions carried out with mechanical vibrations in optical microscopy analyses. During the vibrations, particles with smaller aerodynamic diameters were dispersed in the air, preventing their capture by the microscope and, consequently, their counting through the ImageJ software.The microscopy method using SEM may contain inaccuracies in counting $PM_{1.0}$ particulate matter, as it can mistake the particle's arrangement for its diameter, overestimating the result due to noise generated during image post-processing.

The optical microscopy method with extraction using mechanical vibrations is significantly more economical than the analysis using scanning electron microscopy. The mechanical vibration method can be adapted according to the analysis goal, indicating potential for analyzing larger particles. Just like optical microscopy, SEM-based microscopy also has limitations in counting fine particles, which, although better visualized with SEM, still require further refinement for accurate quantification.

CONCLUSION

This study achieved its objective by performing a comparative analysis between optical microscopy and scanning electron microscopy methods for particulate matter counting. The optical microscope may show values lower than the actual ones due to particle losses during extraction, especially for $PM_{1.0}$. SEM, on the other hand, may show $PM_{1.0}$ values higher than the actual ones, as ImageJ can mistake fine particles for the particle arrangement.

Moreover, the study presented different mechanisms in the ImageJ computational code for processing the images obtained from the analyses, adapting them for each of the methods used. In both methods, the highest percentage of particulate matter occurs near the aerodynamic diameter of 1 micrometer.

This study enables future research aimed at reducing particulate matter emissions, optimizing analysis methods, and providing the automotive industry with more cost-effective alternatives for particulate matter analysis and counting. Additionally, it opens the door for studies related to the chemical characterization of particles in SEM.

Furthermore, this work contributes to some of the 17 Sustainable Development Goals (SDGs) set by the UN, such as SDG 3 (Good Health and Well-being) and SDG 9 (Industry, Innovation, and Infrastructure), considering the environmental, social, and economic pillars. The study demonstrates feasibility and applicability within the automotive sector.

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