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# Suspended particulate characterization using a cascade impactor and X-ray fluorescence.

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## 1. Introduction

Air pollution has become one of the biggest environmental and public health problems in large urban areas. The process of industrialization and urbanization of cities together with population growth has caused an increase in the level of air pollution in these places[1].

Several pollutants present in the air that are suspected of generating harmful effects on human health and the environment, including particulate matter (PM). PM can be understood as a complex mixture of solid and liquid components, which vary substantially in composition and size, depending on the emission source and meteorological conditions. The composition and size of these particles determine the potential for deposition in the respiratory tract and health effects, which is associated with an individual's exposure to this pollutant. The smaller the particle size, the greater its potential to penetrate deeply into the respiratory tract. According to Pires (2005), oil refining activities are responsible for 42% of industrial air pollutant emissions in the Metropolitan Region of Rio de Janeiro. In Brazil, oil refining plays an important role in the economy, accounting for around 30% of the total energy consumed in the country, and practically all the energy consumed in the transport sector.

This study has been carried out in the Duque de Caxias City. This city is located in the metropolitan region of Rio de Janeiro and has a petrochemical complex, which includes the Duque de Caxias Refinery (REDUC). So, the purpose of this study was to identify the chemical elements present in airborne particulate samples from this location using the X-ray fluorescence spectrometry (XRF) technique, In addition, it was possible to estimate the aerodynamic diameters of suspended particulates and point out possible sources for the detected elements.

## 2. Methodology

The collection of suspended particulate matter was performed using a cascade impactor (PIXE International Corporation). The impactor is composed of a set of concentric rings with 10 different aerodynamic cutting diameters: 16 µm – 0.06 µm. The flow was 1 L/min and controlled by a digital flow meter. The sampling points were carried out in homes close to REDUC using cellulose acetate membranes, with a diameter of 25 mm and porosity of 0.2 µm (UNIFIL). For each sampling point, internal measurements (inside the houses) and external measurements (outside) were performed. All sampling began in the morning (9:00 am) and ended 10 hours after. .X-ray fluorescence measurements were performed using the commercial XRF system (ARTAX 200). The X-ray tube has a fine focus (0.6 mm collimator) and a Molybdenum anode. The system uses an SDD type XFlash detector (Silicon Drift Detector) with an 8 µm thick Beryllium window, 10 mm² detector sensitive area. The ARTAX 200 has a laser beam for alignment and a CCD-type color camera with a resolution of 500 x 582 pixels. Experimental XRF measurements were performed under the following conditions: 45 kV, 500 µA, acquisition time of 300 and no filter was used in the incident X-ray beam. The XRF spectra of each analysis performed are acquired and evaluated by the SPECTRA software, version 5.3, provided by the manufacturer itself. Figure 1 shows the experimental setup for particulate collection and the XRF system.

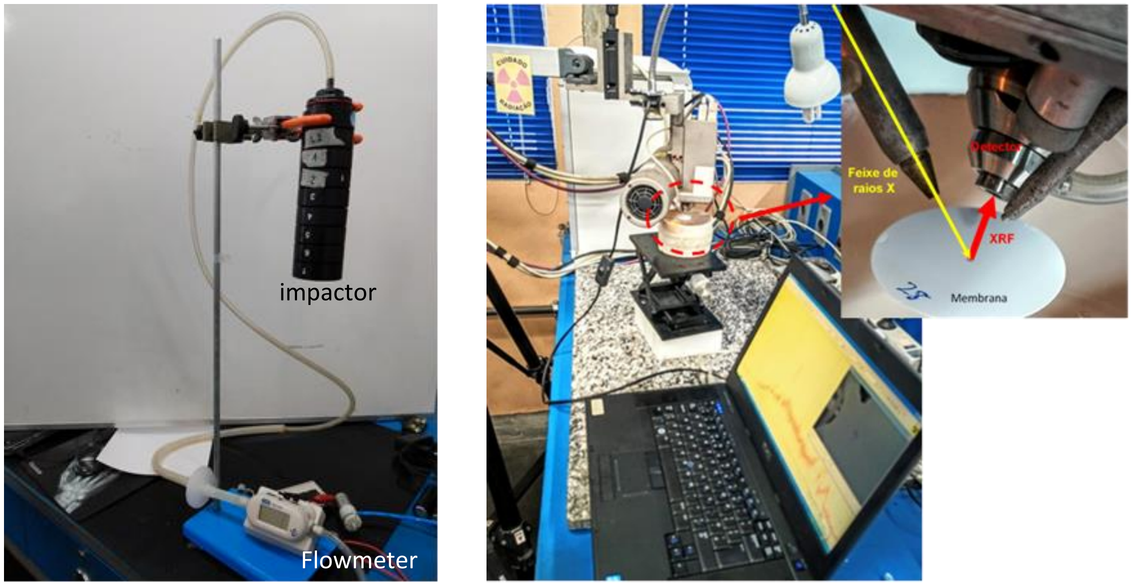


Figure 1: a) Particulate collection system and b) XRF system.

## 3. Results and Discussion

The elements detected in the XRF measurements were: Si, S, Cl, Ar, K, Ca, Ti, Mn, Fe, Zn and Mo. The chemical element Ar is one of the constituents of atmospheric air (about 1.0 %) and molybdenum (elastic scattering) is the target material of the anode of the X-ray tube (figure 2).

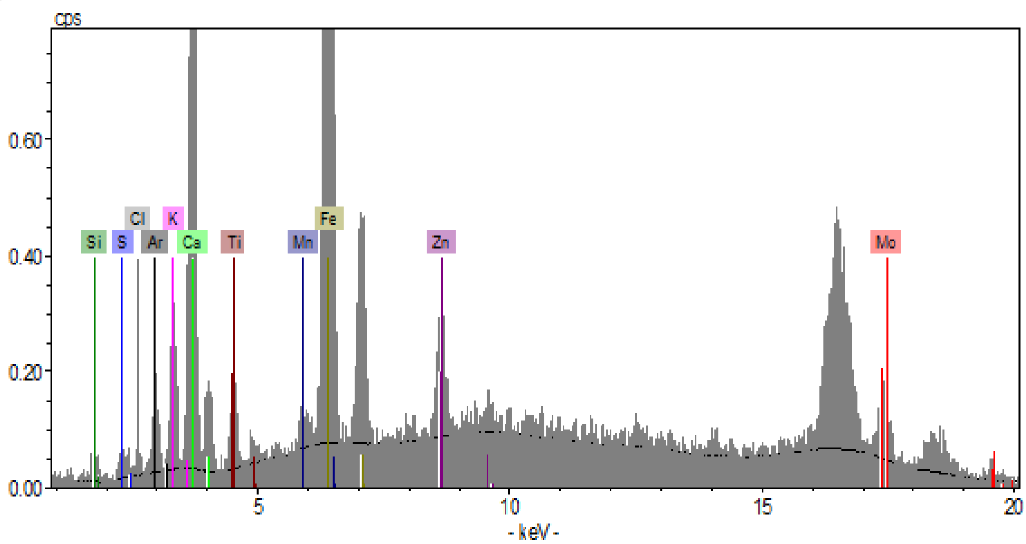


Figure 2 - XRF spectrum of the B out point with an aerodynamic cut diameter of 2.0 µm

The areas under the XRF peaks (counts) for each of the sampling points (inside - in and outside - out homes) were obtained to determine the contribution of each element present in the particulate. The results obtained were represented from dendrograms that allow a better way of grouping the elements and their possible sources. In figure 3 (A in), the dendrogram shows a strong correlation between all detected elements, Si, S, Cl, K, Ca, Ti, Mn, Fe and Zn. However, Zn, Si, S and Cl were in the same grouping, which may show that they are from the same source, probably industrial. Ti was close to this grouping, with a slight difference. This metal is used in metallic alloys and paints [3]. Another important grouping was formed by the elements Ca, K and Fe, which may be due to the re-suspension of the ground [6], because this point is close to an unpaved vehicular traffic. This same pattern was observed at sampling point B in. In the sampling point, A out, the dendrogram shows a very strong correlation between Ni and Zn and close to them S, this may be evidence that these elements come from the same source, in this case, an anthropogenic source. The possible source of Ni may be associated with the proximity of a scrap recycling company that operates close to the residence of sampling point A.

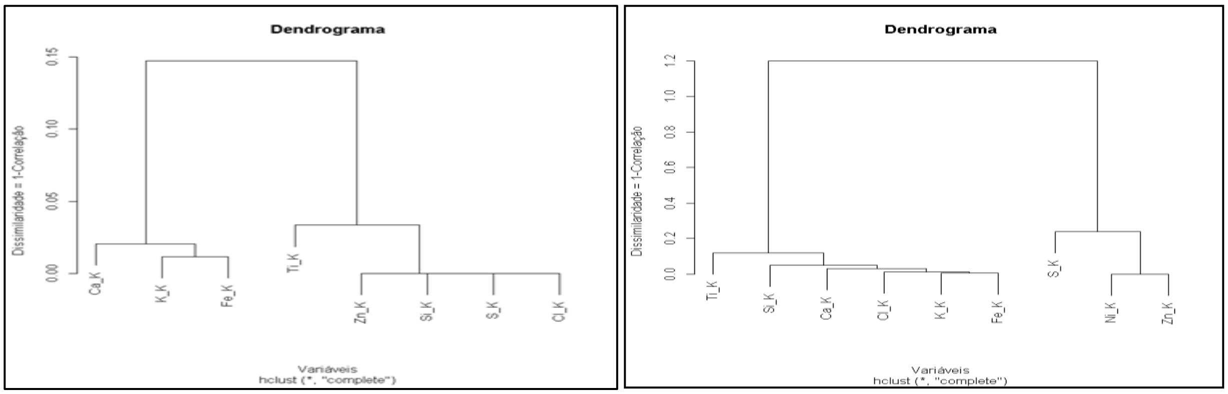


Figure 3 - Dendrogram of the elements of point A in (left) and A out (right)

In figure 4B (in), the elements Si, Cl, K, Ca, Ti and Fe were detected. The dendrogram shows the strong correlation between Si and Ti, which are possibly due to the suspension of particulates associated with clay in ceramic flooring. The Chlorine element was far from the other two groups, in this case, it is very likely that it comes from the use of cleaning products that contain chlorine in their composition, because according to the resident, the floor cleaning is done with these types of product. In figure 4B (out), we observe the elements Si, S, Cl, K, Ca, Ti, Mn, Fe and Zn. Its dendrogram shows the highlight of the S element, which may be linked to the origin of intense vehicular traffic, fossil fuel burning in the vicinity and industrial processes in the region [4]. It is still possible to observe the grouping between Mn and Zn, which are probably due to the suspension of particulates resulting from vehicle traffic [6].

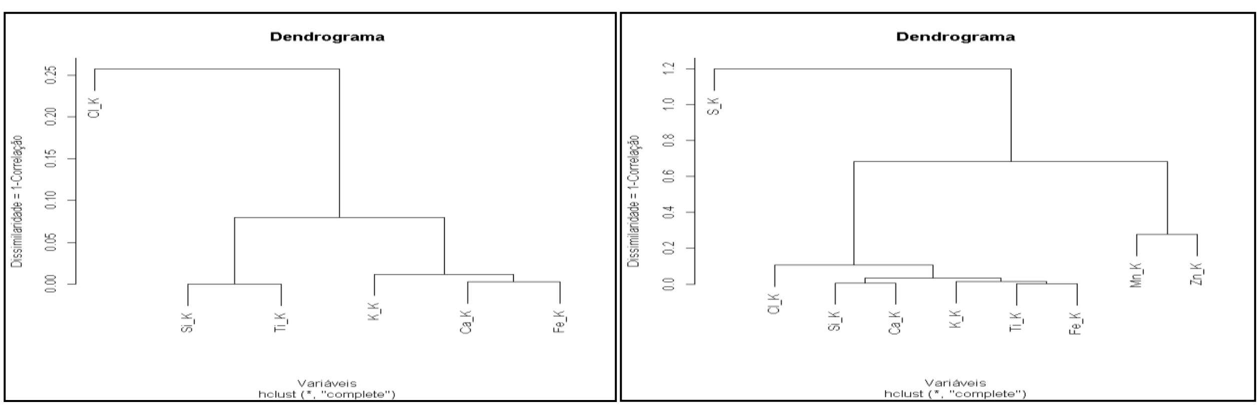


Figure 4 - Dendrogram of the elements of point B in (left) and B out (right)

Finally, in figure 5 C in, the elements Si, S, Cl, K, Ca, Ti, Fe and Zn were detected. The dendrogram shows the highlight of the S element, which may be linked to a punctual, internal, unidentified source. In figure 5 C out, the dendrogram shows the highlight of the Zn element, with no close correlation, which in this case, most likely, comes from materials associated with the wear of car tires, since this sample was collected at the exit of a garage maintenance of automobiles. In addition, during the collection period, the resident said he made cuts in galvanized steel structures, ie, coated with Zn, it probably also contributed to the zinc group. The S element is most likely due to its proximity to a polymer industry and REDUC, as there is no heavy vehicle traffic at the site. Cl, for example, may be due to its proximity to Guanabara Bay; on the other hand, Ca, K and Fe, related to soil resuspension [2]. Unlike the other points, Si and Ti were not detected. At this sampling point, the floor was not ceramic, and the walls of the residence were not finished with any type of paint. According to the literature, ceramics have Si in their composition and paints can have Si and Ti [3].

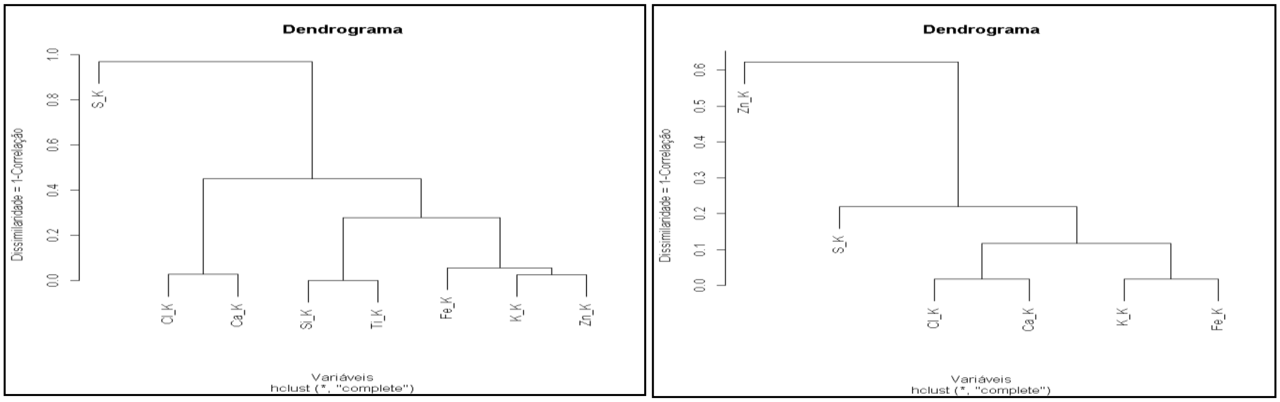


Figure 5 - Dendrogram of the elements of point C in (left) and C out (right)

## 4. Conclusions

It was possible to detect the following chemical elements in suspended aerosols deposited on membranes by impaction and analyzed by XRF: Si, S, Cl, K, Ca, Ti, Mn, Fe, Ni and Zn. Correlations between some elements were observed, making it possible to identify the possible sources of these elements. Besides that, with the use of the impactor, it was possible to identify the size of the aerodynamic diameter of suspended particulates. From the data obtained, it was possible to verify the presence of some chemical elements whose origins are natural and others from anthropogenic sources, such as vehicle traffic, wear of metallic constituents and industrial processes. We believe that this study has been important because: a) it expands the applications of the X-ray fluorescence technique; b) contributes with more information in the literature, in general, about air quality in the region studied; c) motivates future research in other regions with large urban and industrial concentrations.

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