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RESEARCH ARTICLE

Elemental composition of $PM_{2.5}$ in the urban environment of San Juan, Argentina

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Abstract

This study contributes to the current knowledge about air pollution in the province of San Juan, Argentina. Sampling was carried out to measure the fine particulate matter in the atmosphere ($PM_{2.5}$) of the city of San Juan. $PM_{2.5}$ was collected continuously during the winter and spring seasons of 2014 and 2015, and the concentrations of 14 elements (Pb, Ca, K, Cd, Ni, Cr, Mn, V, Cu, Ti, Ba, Co, Sr, and Fe) were determined in $PM_{2.5}$ filters using the technique of X-ray fluorescence by synchrotron radiation (SR-XRF). The results revealed that $PM_{2.5}$ presented annual and seasonal variations, showing a higher concentration during the winter seasons. In addition, for the elements quantified in the filters, a multivariate analysis (Positive Matrix Factorization) was performed to identify the main sources of emission of these elements in the study area, with a series of components being obtained that corresponded to their compositions, which were assigned physical meanings. The first factor, which was the most important in contribution of the sum of the measured elements (45%), was determined mainly by the elements K, Ti, V, Mn, and Fe, which came predominantly from soil particles. The second factor contributed 30% to the measured species in $PM_{2.5}$, with higher Ba and Zn content perhaps being related to emissions from vehicular traffic. Finally, the third factor, in which Pb, Cr, and Ca predominated, may be an indicator of industrial activity and contributed 25% of the sum of the measured elements of $PM_{2.5}$. The results of this study provide the first PM composition database in the province, and this can now be used in the development of mitigation and prevention programs.

Keywords Fine particulate matter · Trace elements · Air pollution · Source of emission · San Juan · Argentina

Introduction

Airborne particulate matter is a pollutant of large concern because of its high concentration levels and its adverse effects on human (Vecchi et al. 2007). The particulate matter (PM), which is composed of a broad class of chemically and physically diverse substances, can be very variable in size, chemical composition, formation mechanism, and origin (Callén et al. 2009). The health risk increases with the exposure to small particles, which are able to penetrate deep into the lungs, and even read the alveolar region (Pope et al. 1995). In addition, PM is associated with cardiovascular problems and premature mortality (Bobak and Leon 1999; Callén et al. 2009). To explore further this latter issue, some authors examined the relationship between long-term exposure to fine PM air pollution and postneonatal infant mortality (Pope 2000; Woodruff et al. 2006), and found that the PM10 fraction of aerosol particles (diameter smaller than 10 μm) and especially the $PM_{2.5}$ fraction (diameter smaller than 2.5 μm) were the primary causes of increased death rates (Braga et al. 2005; Xia and Gao 2011).

The main sources of ambient PM at urban roadways are vehicle exhausts, emissions from tire and brake wear, and the re-suspension of road dust (Bathmanabhan and Madanayak 2010). Element aerosols in the urban ambient atmosphere are produced by various anthropogenic and natural sources. Of these, the anthropogenic ones include combustion of fossil

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fuels and wood, exhaust emission from vehicles, industrial activities, energy production, construction activities, and waste incineration, while natural ones include volcanic activity, wind-eroded soil dust, forest fires, and sea salt spray (Allen et al. 2001; Wang et al. 2005; Mohammed et al. 2017).

The industrial development of mining in the province of San Juan (Argentina) has been carried out unforeseen, without any environmental protection for the disposal of effluents and emissions. Related to this, the province does not make measurements of $PM_{2.5}$, which is why it is very important to carry out atmospheric studies in the province that can provide us with the first data indicating the pollutants present in the atmosphere. We cannot minimize the impact on health if we do not even know the amounts of PM in our atmosphere or how different sources contribute (Fang et al. 2000). Moreover, regulation of the PM_{10} mass of particles requires a better knowledge of both the $PM_{2.5}$ and PM sources and their behavior in the atmosphere (Callén et al. 2009).

The objective of the present study was to determine the concentrations of $PM_{2.5}$ in San Juan, its seasonal variations, the concentrations of elements in $PM_{2.5}$, and their relation with the sources present in the area. This work should be considered as a first attempt to address the lack of information on PM in San Juan city, with our results making it possible to compare the atmospheric situation with other cities of Latin America, and providing a benchmark for future studies in the region.

Materials and methods

Study area

The selected study area is located in the city of San Juan, Province of San Juan, Argentina, at 31° 30′ S latitude and 68° 34′ W longitude (Fig. 1). This city is characterized by a completely urbanized landscape with approximately 500,000 inhabitants. To the east of the city is the agricultural sector, where vines, vegetables, forestry, and fodder flourish. Also, among other activities, we can highlight industry, with one of the most important of the province being located here, which is dedicated to the production of calcium carbide (DINREP 2015). In addition, the traffic in this area is characterized by all types of vehicles (cars, trucks, buses, etc.).

During the years 2014 and 2015, $PM_{2.5}$ sampling campaigns were carried out in the winter (July and August months) and spring (October and November months) seasons, in order to evaluate the seasonal and annual variations of PM in this area, and also to identify the main emission sources.

$PM_{2.5}$ concentrations

The concentrations of $PM_{2.5}$ were measured with Harvard impactors operating at 16 L min^{-1} (Air Diagnostics and Engineering Inc., Naples, ME, USA). Briefly, during the sampling periods, particles were collected once a week on 47-mm PTFE filters (1 μm pore) to give a total of 32 samples, with filters always being replaced at the same time of day (11–12 a.m.). The particle mass was determined by gravimetric analysis using analytical microbalances with a precision of 1 μg. The teflon filters were conditioned for 48 h in a room at constant temperature (21 ± 2 °C) and relative humidity (35 ± 5%) conditions before performing the pre- and post-weighings.

Element concentration

The concentrations of K, Ca, Cr, Mn, Fe, Co, Ni, Cu, Zn, Br, Sr, V, Ti, Pb, and Ba in the $PM_{2.5}$ filters were determined by total reflection X-ray fluorescence (TXRF) using synchrotron radiation. The extraction of the elements from the teflon filters was performed in an ultrasonic bath (power effective 300 W) at 40 °C for 30 min, using 5 mL of analytical commercial HNO_3 (20% *v*/*v*) for the digestion, with the solid residue being separated by centrifugation. Then, 10 ppm of Ga solution was added as an internal standard, and aliquots of 5 μL were taken from this solution and dried on an acrylic support. Standard solutions with known concentrations of K, Ca, Cr, Mn, Fe, Co, Ni, Cu, Zn, Br, Sr, V, Ti, Pb, and Ba (with Ga as an internal standard) were prepared for the calibration of the system. Samples were measured for 100 s, using the total

Fig. 1 Location of the study site in the city of San Juan, Argentina

reflection setup mounted at the X-ray fluorescence beamline, and a polychromatic beam of approximately 5 mm wide and 0.1 mm high was used for excitation. For the X-ray detection, a Si (Li) detector was used with an energy resolution of 165 eV at 5.9 keV (Wannaz et al. 2011).

As a quality control, blanks and samples of the standard reference material CRM 281 were prepared in the same way and run after five determinations to calibrate the instrument, with the results found to be within ± 2% of the certified value. The coefficient of variation of the replicate analysis was calculated for different determinations, and the variations were found to be less than 10%.

Meteorological data

The mean temperature (T), relative humidity (RH), wind speed (W), and rainfall (R) were provided by the National Meteorological Service located in San Juan. Climatic data are recorded here at 1-h intervals, from which an average daily reading is calculated. This meteorological station is located approximately 5 km from the sampler.

PMF model

PMF 5.0 was adopted to source apportionment of heavy metals in $PM_{2.5}$. According to the EPA PMF 5.0 user guide:

$$X_{ij} = \sum_{k=1}^{p} g_{ik} f_{kj} + e_{ij}$$

where *xij* is a measurement matrix of the *j*th heavy metal element in *i* number of samples; *gik* is a contribution matrix of the *k*th source factor for *i* number of samples; *fkj* is a source profile of the *j*th heavy metal element for the *k*th source factor; and *eij* refers to the residual value for the *j*th metal element in *i* number of samples. The minimum value of the objective function *Q* can be computed by the following formula:

$$Q = \sum_{i=1}^{n} \sum_{j=1}^{m} \left(\frac{X_{ij} - \sum_{k=1}^{p} g_{ik} f_{kj}}{\mu_{ij}} \right)^2$$

where uij refers to the uncertainty in the *j* heavy metal element for sample *i*. The remarkable feature of PMF is using uncertainty to analyze the quality of every concentration data individually. If the concentration of heavy metal does not exceed the MDL (method detection limit) value, the uncertainty is calculated using the following formula:

$$Unc = \frac{5}{6} \times MDL$$

If the concentration of heavy metal exceeds its corresponding *MDL* value, the calculation is

$$Unc = \sqrt{(Error\ fraction \times concentration)^2 + (0.5 \times MDL)^2}$$

Statistical analysis

The Shapiro–Wilk test for normality was applied, with non-normal distributed elements being log-transformed before parametric statistics were performed. Subsequently, an analysis of variance (ANOVA) was used to compare different seasons. Whenever the ANOVA indicated significant effects ($p \leq 0.05$), a pairwise comparison of means was undertaken using the Tukey test.

Results and discussion

Concentration of $PM_{2.5}$

The mean concentrations found for the different seasons in the 2 years of the study area are shown in Fig. 2, where it can be observed that only in the winter of 2014 did the average values found exceed the daily guide value adopted by the National Ambient Air Quality Standard (NAAQS) of the United States for $PM_{2.5}$ (35 μg/m^3). Although the concentrations of $PM_{2.5}$ observed in 2014 were higher than those of 2015, ANOVA did not reveal significant differences between years. However, for different seasons, ANOVA gave significant differences, with the winter season having a significantly higher concentration of $PM_{2.5}$ compared to spring season ($p = 0.0483$).

The mean concentration for winter was 39.83 μg/m^3, which was similar to a value reported in Buenos Aires in summer–autumn (33 μg/m^3) (Bogo et al. 2003), but lower than that found in Santiago de Chile for the winter months (62 ± 15 μg/m^3) (Villalobos et al. 2015). According to Bogo et al. (2003), the topographical and meteorological differences can have a great influence on the presence of the particulate material. Thus, as San Juan is situated in a mountainous valley of the Andes, while Buenos Aires is located in a flat area, the latter is windier with the air being continuously renewed. On comparing our results for $PM_{2.5}$ with concentrations from some regions of the world, the values we found in San Juan were lower than those reported in Beijing (106 μg/m^3) (Shi et al. 2003) or in Agra in the north of India (90.16 μg/m^3) (Pipal et al. 2011), both cities that have serious problems of $PM_{2.5}$ contamination.

Another Argentina study carried out in the province of Córdoba reported an annual average of 71 ± 21 μg/m^3 of $PM_{2.5}$ (López et al. 2011), compared to values obtained in San Juan of 37.78 μg/m^3 in winter and 29.39 μg/m^3 in spring, but due to the increase in San Juan in population and industry, we consider it relevant to continue the measurements to determine if these results are maintained in future.

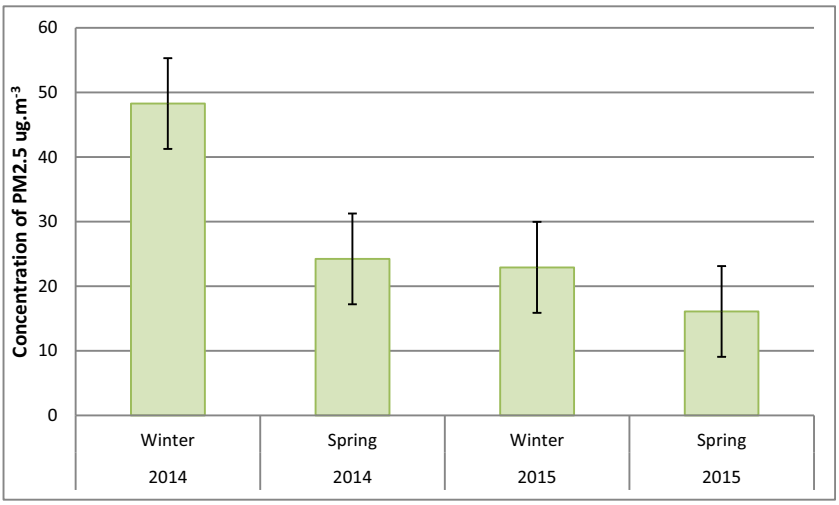

Fig. 2 Concentration of $PM_{2.5}$ during the sampling years and in the different seasons. Bars indicate standard error

Meteorological variable

Weather conditions strongly influence the concentration of particles, with wind, temperature, and solar radiation being able to drastically modify the dispersion and the type of pollutants that may exist at a given moment and alter the persistence of pollution in an area (Sanhueza et al., 1999). The values of temperature, wind, and relative humidity measured in the study area are presented in Table 1, where it can be observed that during the study period the wind speed and temperature presented significant differences between seasons. With respect to precipitation, the study area has a dry desert climate (BWk) with a marked water deficit according to the climatic classification of Koeppen, with precipitation mainly occurring in the summer and reaching a total of approximately 100 mm per year (Suvires 2014). Although during the course of this study only three samples were collected with precipitation occurrence, this had an impact on the concentration of particles in the atmosphere (Fig. 3), with that of $PM_{2.5}$ being significantly lower for samples with rain ($p = 0.020$). This may have been attributed to the fact that rain could reduce the concentration by means of wet scavenging as well as reduce the resuspension of dust, and in this way, rainy seasons are very effective for cleaning particle-laden air (Pandey et al. 2014).

Table 1 Mean concentrations (± stand ard error) of climatic variables in San Juan, Argentina, during the sampling periods 2014 and 2015. ANOVA between seasons

	Winter mean ± S.E.	Spring mean ± S.E.	ANOVA test p value
Wind (m/s)	12.58 ± 0.72	16.37 ± 0.59	*0.0013*
Temperature (°C)	18.4 ± 2.97	27.39 ± 2.42	*0.0355*
(%) relative humidity	40.01 ± 3.74	30.01 ± 3.05	ns

Italic numbers indicate significant differences between seasons

Elemental concentration in $PM_{2.5}$

Table 2 summarizes the concentrations of the elements measured in $PM_{2.5}$ in the present study, where it can be observed that the concentrations of the various elements varied considerably from a few micrograms per cubic meter for elements such as Ca to concentrations of less than nanograms per cubic meter for elements such as Ni, Sr, and V. Regarding the analysis of the variance between the different sampling seasons, only K and Cu revealed significant differences ($p \leq 0.005$), with winter being the season where the highest levels of these elements were found.

Other studies on the chemical composition of particles in Argentina have focused mainly on the fraction of PM_{10} in the city of Buenos Aires (Bogo et al. 2003; Bocca et al. 2006; Fujiwara et al. 2011). For $PM_{2.5}$, there is only one study published on elemental composition in Argentina, which was performed in Córdoba city (López et al. 2011), with reported concentrations being of the same order of magnitude as those found in the present investigation for the elements Cr, Mn, Fe, Co, Ni, and Cu. However, other elements such as V and Zn were an order of magnitude lower than those found in

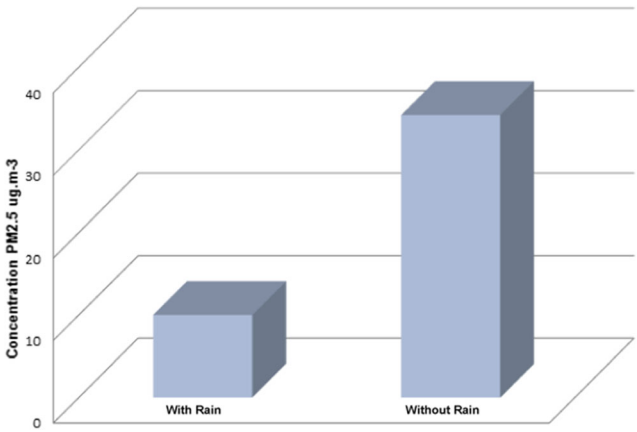

Fig. 3 Average concentration of $PM_{2.5}$ registered in the area according to samples with or without rain

Table 2 Mean concentrations (± standard error) of the elements quantified in $PM_{2.5}$ filters in San Juan, Argentina

	Spring		Winter		ANOVA
ng/m^3	Mean	SE	Mean	SE	*p* value
K	250	130	530	170	*0.004*
Ca	9070	3140	9590	6420	*0.837*
Ti	0.099	0.08	0.18	0.13	*0.162*
V	0.15	0.17	0.37	0.39	*0.140*
Cr	2.8	4.7	2.9	2	*0.948*
Mn	10	2.5	10	4.1	*0.431*
Fe	120	40	200	110	*0.060*
Co	0.15	0.084	0.19	0.076	*0.380*
Ni	0.72	0.53	1.9	3.5	*0.330*
Cu	10	3.2	20	10	*0.010*
Zn	3.6	4.4	4.2	3.3	*0.752*
Br	3.4	2.3	4	1.1	*0.592*
Sr	0.59	0.84	0.61	0.79	*0.962*
Ba	3.1	3.5	1.7	2.1	*0.398*
Pb	20	10	20	10	*0.787*

Italic numbers indicate significant differences between seasons

Córdoba city (López et al. 2011), whereas the concentrations of Ca (~ 9000 ng/m^3) $PM_{2.5}$ in San Juan were much higher than those found by Lopez et al. (2011) (~ 300 ng/m^3). Sylvestre et al. (2017) obtained different emission profiles of $PM_{2.5}$ in multiple industrial activities and observed that Ca was present in all these industrial activities, which suggests that the high levels of Ca found in this study could be due to the presence of a calcium carbide factory in the study area.

Multivariate analysis

EPA PMF 5.0 was used to determine which sources of elements (Pb, Ca, K, Cd, Ni, Cr, Mn, V, Cu, Ti, Ba, Co, Sr, and Fe) contribute to $PM_{2.5}$ emission in San Juan. It is important to mention that lack of major ions and carbon concentrations could limit the possibility to individuate all sources acting on the area. However, the source apportionment based on these elements could give important insights on some specific sources and allow the evaluation of their contribution to the species effectively measured in $PM_{2.5}$ as shown in previous studies (Clements et al. 2014; Gregoris et al. 2016).

The concentration values used in the package were the measured data and the values of analytical uncertainty. The XRF technique provides a spectrum, and the modeling of the integration of that spectrum produces the uncertainty associated with the measurement of each sample. The desired total uncertainty is composed from addition of the uncertainties of standards used for calibration, sample preparation, irradiation of patterns, irradiation of the sample, and processing of the collected x-ray spectrum (Wegrzynek et al. 2003). The most critical step in PMF analysis is to determine how many factors explain data variability, because there is no mathematical criteria to predict the optimal number of factors (Song et al. 2001).

PMF simulations for three, four, five, six, seven, and eight factors were conducted as an exploratory analysis to determine the number of factors. The *Q* (true) values (which measure the goodness of fit between the modeled solution and the input data) associated with the number of factors were evaluated and indicated an optimal goodness of fit for three factors. Therefore, these three factors were chosen for the $PM_{2.5}$ data set, with the contributions for the different factors being presented in Fig. 4. The results indicate that for $PM_{2.5}$ the most important factor accounted for 45% of the sum of the measured elements, which presented the highest values of K, Ti, NI, V, Mn, and Fe.

Because the soil contains characteristic elements such as Al, Si, K, Ca, Ti, and Fe (Watson et al. 2001), the first factor can be considered to come mainly from a natural source (soil), produced by the re-suspension of soil particles or construction activities, and could also be due to land use change due to the expansion of the city (Liang et al. 2017). However, high levels of Fe in the fine fraction have previously been associated with the abrasion of mechanical vehicle parts or from compounds that are added to lubricants and are associated with increased levels of Ni (Amato et al. 2009). This source is mixed with elements of the crust, such as Mn, Si, and Ti, suggesting that these particles are mixed with urban dust (Begum et al. 2007). Therefore, the first factor may be indicating mainly soil particles, which have been enriched with some elements such as Ni in the urban zone.

With respect to factor 2, which accounted for 30% of the sum of the measured elements, this emphasizes the presence of Ba, Zn, and Sr (elements originating from the traffic). Zn compounds are widely used as lubricants, antioxidants, and detergent builders, and oxidation of lubricating oils after exposure to high temperatures in the air results in the formation of metal oxide particles (Begum et al. 2007). There could also be a contribution from tire wear, braking, and abrasion. While the emitted products are considered to be mainly coarse particles, the braking temperatures at the coating/rotor interface may be high enough to vaporize many of these materials after condensation into fine-sized fractions (Salma and Maenhaut 2006).

The third factor, which accounted for 25% of the sum of the measured elements in $PM_{2.5}$, was attributed mainly to industrial processes in the study area, since the markers of this factor were Ca, Pb, Br, and Cr. The presence of a calcium carbide factory may have also been associated with the enrichment of this element. In addition, according to Pipal et al. (2011), processes carried out in certain industries, such as the exploitation of Ca in quarries, crushing, and grinding, can facilitate this element to be incorporated into fine particle size such as $PM_{2.5}$ (Pipal et al. 2011).

Although the objective of this analysis was to identify emission sources that contribute to the composition of $PM_{2.5}$, it is

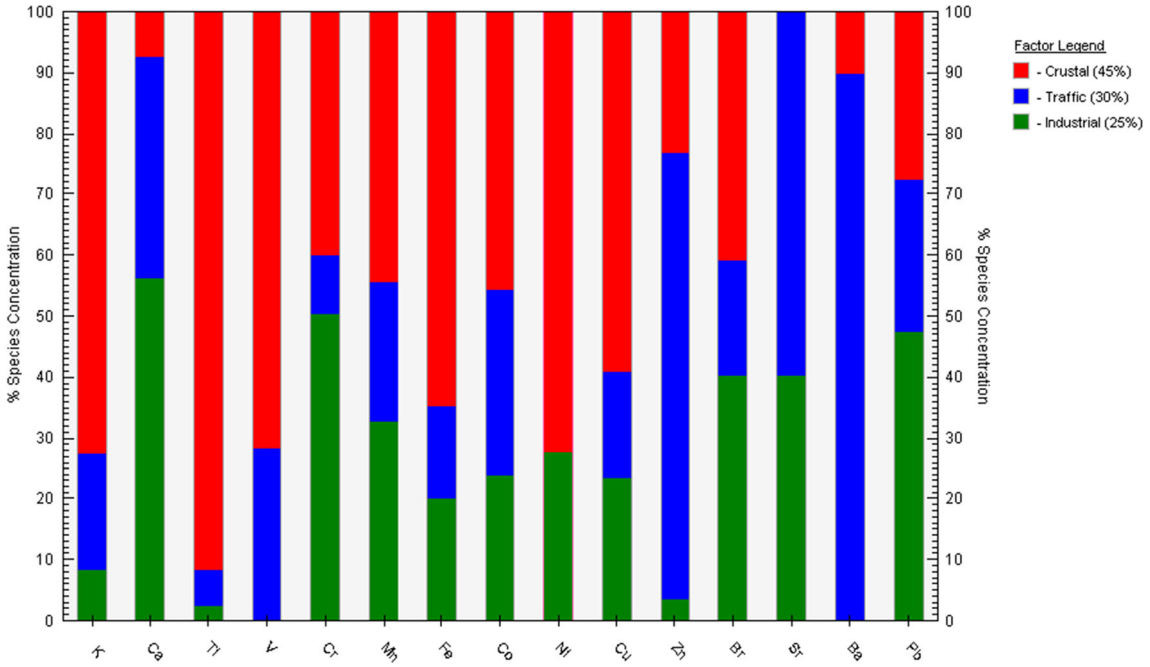

Fig. 4 Profiles of the contribution percentages of each element to the different factors found in $PM_{2.5}$

important to note that some authors such as Viana et al. (2008) have noted that aerosols are subject to mixtures with primary or secondary particles during transport, and that they share the same marker with anthropogenic emissions at the local scale. Therefore, clustering of secondary aerosols and local anthropogenic contributions into factors can be extremely complex (López et al. 2011). However, through the use of the multivariate analysis PMF, we have been able to identify the sources of well-defined emissions in the study area.

Conclusions

The data obtained in this work constitute the results of the first monitoring campaign of $PM_{2.5}$ concentration in the city of San Juan, which presented seasonal and annual variations, with winters presenting higher concentrations of $PM_{2.5}$. In addition, it was observed that variables such as wind and rain had effect on the concentration of $PM_{2.5}$.

The elements in $PM_{2.5}$ did not reveal any significant differences in their concentrations between spring and winter (except for K and Cu), which may indicate that the sources present in the study area remained constant during the different seasons of the year. In addition, the high concentration of Ca in $PM_{2.5}$ compared to other regions of Argentina might be related to the presence of an important calcium carbide factory.

The positive matrix factorization performed for the elements measured in $PM_{2.5}$ revealed three well-marked factors, which was the most important in contribution of the sum of the measured elements (45%), which was determined from soil particles and 30% of the quantified elements of vehicular traffic, with the remaining 25% being attributed to the industrial emissions present in the study area.

We hope that these preliminary studies will be considered as a reference for future research or environmental management activities aimed at predicting atmospheric pollution by particulate matter in the city of San Juan.

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