Characterization of ambient air dust particulates deposited on *Ficus* leaves in the Metropolitan Area of Guadalajara, México

Caracterización de partículas de polvo ambiental depositados sobre hojas de *Ficus* en la zona metropolitana de Guadalajara, México

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Abstract

Leaves of Ficus were collected in 11 points. The particles was mainly under the age of 10 µm with a spherical shape and oval, and agglomerates of particles. The elemental analysis showed mainly C, Yes, OR, Ca and K with different concentrations of elements such as Zn, Pb, Cd and Ni in addition to W, Fe and Ti. *Objectives, methodology*: Identify morphology and elemental composition of particles present. The sampling sites were established according to the urban trace, land use and types of roadways. MEB was applied x-ray spectroscopy Using atomic absorption measured the concentrations of trace metals (Cu, Zn, Co, Ni, Cd, Cr and Pb) the ambient dust deposited on the surface of the sheet. *Contribution*: On the site with industrial, commercial, services and housing identified greater presence of elements analyzed with high levels of heavy metals. We stress the importance of conducting further analysis that allows us to control cyclical fine particles in the air and the evolution of the same.

Quality of air, Ultra fine particles, Heavy metals, Geophysical observations

Resumen

Se colectaron hojas de *Ficus* en 11 puntos. Las partículas fue principalmente menores de 10 µm con forma esférica y ovalada, y aglomerados de partículas. El análisis elemental mostro principalmente C, Si, O, Ca y K con diferentes concentraciones de elementos como Zn, Pb, Cd y Ni además de W, Fe y Ti. *Objetivos, metodología:* Identificar morfología y composición elemental de partículas presentes. Los sitios de muestreo se establecieron según la traza urbana, uso de suelo y tipos de vialidades. Se aplicó MEB espectroscopia rayos X. Mediante absorción atómica se midieron las concentraciones de metales traza (Cu, Zn, Co, Ni, Cd, Cr y Pb) del polvo ambiental depositado en la superficie de la hoja. *Contribución*: En el sitio con actividades industriales, comerciales, de servicios y de vivienda se identificó mayor presencia de elementos analizados con elevados niveles de metales pesados. Subrayamos la importancia de realizar posteriores análisis cíclicos que nos permita controlar las partículas finas en el aire y la evolución del mismo.

Calidad del aire, Partículas ultra finas, Metales pesados, Observaciones geofísicas

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Introducción

Urban areas represent sources of continuous emission of polluting particles mainly due to traffic and industrial activities (Aguilar et al., 2011). The World Health Organization (WHO) has made several publications, which state that air pollution by metal particles is a major cause of mortality and morbidity worldwide, and in the future will be the leading cause of premature mortality. Therefore, it is the most important environmental risk is for health; affecting mainly the vulnerable groups (younger than five and older than 65 years, population in poverty and / or with health problems). At the Latin America level, Mexico is in the third place of countries with highest number of deaths attributable to air pollution, with the particles of an aerodynamic diameter less than 10 (PM_{10}) and 2.5 μm microns ($PM_{2.5}$) as the main air pollutants. Small particles are particularly harmful, because of their ability to penetrate deep into lungs, causing inflammation and aggravating heart or lung problems (Chen, Shah, Huggins, Huffman, & Dozier, 2005; OMS, 2014).

The particles are diverse in shape, they can be observed as spheres, ellipses, cubes, or aggregates. The chemical composition of particles is also very diverse and depends mainly on both the source station and the formation mechanism of the particles. In the case of coastal areas, their composition is predominantly sodium chloride; those with geological origin shall be composed of oxides of iron, calcium, silicon and aluminum.

For the particulate matter (PM) produced by combustion, they will be based on carbon and inorganic materials whose origin will be the mineral present in the fuel. Regarding most industrial processes, such as cement factories, where the main compounds are inorganic, they will frequently have a chemical composition similar to that of raw materials or products generated. As for PM produced by gasoline powered mobile vehicles, they will have a mixture of organic carbon, trace of metals and sulfate (Glynis C. Lough et al., 2004).

The PM can travel long distances (it depends on the size, smaller particles will travel longer distances) and subsequently is deposited in soil, water or vegetation. Therefore, it generates health effects, even at very low concentrations.

So, it is essential to reduce the levels of air pollution by particles, because this can reduce the burden of disease resulting from stroke, lung cancer, chronic and acute pulmonary diseases, including asthma.

In Mexico, cities considered as critical, due to the levels of environmental pollution from PM are Mexico City, Guadalajara Metropolitan Area (GMA) and Monterrey. According to the environmental monitoring network of Guadalajara, in the period 2000-2009 the main problems with the air quality in the GMA are related to high concentrations of ozone (O_3) and PM10 (Secretaría de Medio Ambiente y Recursos Naturales, 2011). Guadalajara is considered one of the most important industrial and economic cities in the country, with 11 industrial parks and one technological park; the latter do not support manufacturing or real estate activities (Instituto Tecnológico y de Estudios Superiores de Monterrey, 2011).

Economic activities taking place in the municipalities of the area under study are shown in Fig. 1, being the city of Guadalajara the one where the highest percentage of these are located, followed by Zapopan, etc., according to the National Statistics Directory of Economic Units (INEGI, 2014).

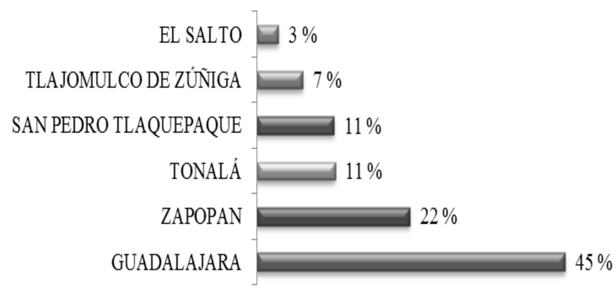

Figure 1 Distribution by municipality of economic activities

There are approximately 31,265 businesses, 45 % of which are engaged in trade, 27 % in manufacturing of various products, and the remaining 27 % are engaged in diverse activities. As for manufacturing activities, the main corresponds to blacksmithing products (20 %), furniture (13 %), bricks (9 %), wood products (4 %), pottery (4 %), footwear with cutting skin (3 %), stone-based products (2 %), plastic footwear (2 %), integrated kitchens (2 %), and office furniture and shelving (2 %); the rest (39 %) are engaged in the manufacture of various products.

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As for the distribution of these factories by city, Guadalajara, Zapopan and Tonala are the main (Fig. 2).

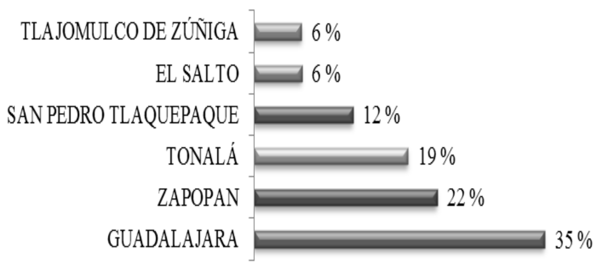

Figure 2 Manufacturing distribution by municipality (INEGI, 2014).

Moreover, previous studies have showed the metal trapping capacity of tree species for monitoring air in different parts of Mexico and the world (Aguilar et al., 2013; Padgett, Meadows, Eubanks, & Ryan, 2008). The technique of scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDS) can be used to observe morphological details and the chemical composition of elements deposited in the cell structure of the leaves of the tree species (Aguilar et al., 2012). Additionally, the atomic absorption spectroscopy method has been used for the analysis the concentration of trace elements including heavy metals coming from air pollution (Bhattacharya, Chakraborty, Tuteja, & Patel, 2013).

The objective of this research was to use scanning electron microscopy equipped with an energy dispersive X-ray spectrometer and atomic absorption spectroscopy to characterize heavy metals deposited on leaves of *Ficus* collected in the metropolitan area of Guadalajara and thus assess air quality.

Experimental

Study area

Jalisco is one of the 32 states that comprise Mexico. It is located in the west of the country, between the mountain areas of the Sierra Madre Occidental, the Sierra Madre del Sur, the Mexican Volcanic Belt and the Central Plateau. Jalisco has an area of 78,588 km^2 (accounting for 4.0 % of the national total) (Gobierno del estado de Jalisco, 2015). The GMA is the country's second largest city, after the Mexico City. It is located in the center of the State of Jalisco.

Surrounding the GMA, at the NE is Sierra de San Esteban; At SE, Serranía de San Nicolas and mountain sets Cerro Escondido, San Martin and El Tapatío, La Reyna; At the S, Cerro del Cuatro, Gachupín and Santa Maria; At W, the Sierra La Primavera. These mountains are natural physical barriers to the movement of the winds. The slopes vary with an average of 3 % (Instituto Nacional de Ecología, 2007).

Regarding winds in the GMA, the prevailing winds are weak with the presence of thermal inversion phenomena about 300 days a year. Plus anticyclone systems generated in the Gulf of Mexico and the Pacific Ocean influence the area, causing atmospheric stability and preventing vertical air mixing (García et al., 2014).

The GMA consists of eight municipalities, San Pedro Tlaquepaque, Tonala, Zapopan, Tlajomulco de Zuniga, El Salto, Juanacatlán, Ixtlahuacán de los Membrillos and Guadalajara. The area covered is 2,734 km^2. According to the projections of the population of the municipalities that make up the metropolitan areas, 2010-2030, the total population of the eight municipalities of the GMA in 2014 is 4, 737,096 inhabitants (CONAPO, 2014), of which six (Guadalajara, Zapopan, Tlaquepaque, Tonala, Salto and Tlajomulco de Zuniga) represents our study area.

According to the INEGI in 2013, the vehicle fleet for these six municipalities was 1,878,270, Automobiles (67 %), cargo trucks and vans (25 %), motorcycles (7 %) and passenger buses (0.4 %). In 2009 55 % of the vehicle fleet was over 10 years old (Secretaría de Medio Ambiente y Recursos Naturales, 2011). This trend increased by 4 % to 2012 (59 %) (CONAPO, 2014).

Features of *Ficus* (Moraceae)

The *Ficus* are tree (up to 50 m in height and 1 m in diameter) and bushes (0.3 to 1 m high and diameter of 3-4 cm) species, more commonly from 3-15 m, with an appearance of lianas or vines. It is a representative species of tropical and subtropical forests for their evergreen foliage. The leaves are usually alternate, with a symmetrical or asymmetrical base, varied, simple, spiral, complete, toothed or lobed shapes. Young individuals have leaves larger than those from adults.

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These plants have latex in its leaves, which usually shows as milky white and rarely aqueous, yellow or even light green. In the GMA, *Ficus benjamina* L. (Moraceae) species are widely distributed. In the city, the individuals belonging to this gender can be observed in gardens, sidewalks, streets, medians, parks, roundabouts, among others (Cazimir, Sylvestre, & Carvajal, 2001). The *Ficus* tree leaves have been used for trapping heavy metal particles from atmospheric dust in urban areas due to the latex secretion and the morphological characteristics (stomata) of the leaves (Aguilar et al., 2012).

The stomata are groups of two or more specialized epidermal cells whose function is to regulate the gas exchange and transpiration. Each stoma is formed by two occlusive cells and between them there is an opening called ostiole (pore) (Botánica Morfológica, 2013). The stomata are found in the aerial parts of the plants, particularly in the leaves.

Sampling design

Cartographic and land use information of the Government of the State of Jalisco (Secretaría de Medio Ambiente y Desarrollo Territorial, 2016) were consulted to obtain the urban layout, land use and the roads. This was used to design the sampling in the GMA, making classification and grouping in seven land uses: 1. Low density housing (LDH), 2. High density housing (HDH), 3. Intraurban not developable (IND), 4. Urban corridor (UC), 5. Urban infrastructure (UI), 6. Industrial (I) and 7. Mixed (M). Similarly, they were classified by type of road, making an estimate of the traffic flow (high, medium, low and rural), and the dimensions of the road.

Obtaining four types of roads 1) primary, 2) secondary, 3) tertiary and 4) rural. A stratified sampling was designed relying on the satellite photo of the GMA (Fig. 3). For the cartographic design, we worked with free software for Digital Map of Mexico QGIS desktop version 6.0.1 and version 2.6.

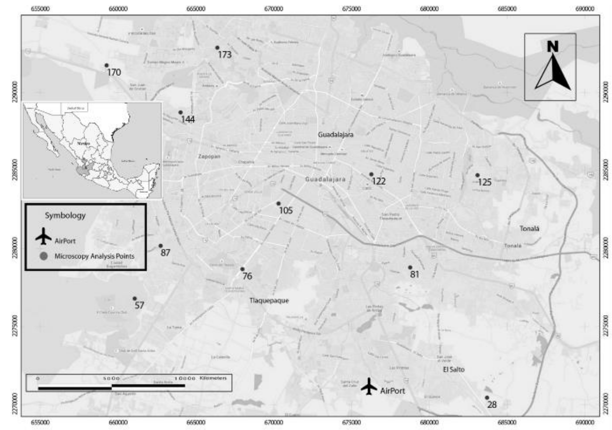

Figure 3 Location of the sampling sites in the Metropolitan Area of Guadalajara

Sampling

Sampling was performed on Saturday March 23, 2013. Eleven samples of *Ficus* leaves were collected. The sites were georeferenced in coordinates of Universal Transverse Mercator (UTM) and Datum WGS84. For the sampling, safety equipment (goggles, gloves and face masks) was used. The leaves were collected from a height of 1.5 to 2 meters. 30 large mature brown leaves were cut with pruning shears. The leaves were collected in re-sealable bags and a colored label was added with the sample number, time, name of the collector, and the coordinates of the site according to previously developed methodology (Aguilar et al., 2013; Bautista et al., 2011).

Preparation of sampled leaves *Ficus benjamina* (Moraceae)

Sampled leaves were taken to the drying room of the Institute of Botany, where they were placed individually on brown paper. They were accommodated in a press and put on a stove with convection heat system.

This process lasted 48 hours, during which they were checked periodically for re-accommodate them to ensure a homogeneous drying. Once dry, they were placed in re-sealable bags with the respective labels.

SEM and heavy metals analysis

The SEM measurements were performed with a microscope JEOL JSM 6610LV, operating at 10kV, with an EDS Oxford Xmax detector. The use of this technique allowed us to identify the size, morphology and elemental composition of the particles that are present in the leaves.

SEM analysis was principally applied as a complementary tool due to its great specificity that bulk analytical methods. This technique has been used to obtain images of the surfaces of the leaves, as it enables dry analysis or with a thin conductive film that covers the sample and facilitates analysis (Barkay, Teller, Ganor, Levin, & Shapira, 2005; Walker, Allen, Bell, & Roberts, 2015). In this case, for SEM analysis, eleven samples were taken of those leaves with the most dust observable at the naked eye and that belonged to different areas of the GMA.

Using a scalpel blade, small fragments of leaves were cut and with a curved forceps, they were placed on the aluminum sample holder, which was previously prepared with a double sided conductive carbon tape to hold the samples still during the analysis process within the equipment. Because of the leaves were dry, it was not necessary to add any substance to stabilize them (Echlin, 2009). Each sample was taken through a general scanning at a small scale (250 or 100 µm), to know the chemical composition of the particles present in the leaves and, subsequently, in the areas of interest (for example, increased accumulation of particles). These particles were observed in greater detail (10, 2.5 µm and smaller). This procedure allowed making EDS elemental mapping and measurements of particles size and shape for comparisons of contamination between the different sampling areas.

On the other hand, trace metals (Cd, Co, Cr, Cu, Ni, Pb and Zn) were measured with a flame atomic absorption spectrometer model Varian AA240 FS equipped with cathode lamps as radiation source, a Czerny-Turner monochromador and an adjustable nebulizer system. The absorbance measurement was converted to concentrations using standard calibration curves. All samples were treated in duplicate.

Results and discussion

In general, particles of several shapes ranging from 0.5 to 10 µm not homogeneously distributed within the ostioles and surrounding areas were observed from SEM images of the leaves. For example, individual ultra fine particles of different sizes (< 1 µm) as well as individual big particles (from 5 to 10 µm) and aggregates deposited around the stoma openings, were observed in the sample 170 (Fig. 4a).

The sample 76 is mainly characterized by ultra fine and fine particles (~0.5-2.5 µm), ranging from sub-round to round shapes as well as some aggregates located outside stoma openings. Particles larger than 5 µm with sharp borders within the ostioles, typical of mineral soil, were also observed in this sample (Fig. 4 b). Small individual particles (< 2 µm) emitted by anthropogenic sources from combustion processes are characterized by their spherical shape and smooth surfaces, although agglomerates of these particles can also be observed on the leaf surfaces (Tomašević, Vukmirović, Rajšić, Tasić, & Stevanović, 2005). The presence of these particles around and over the stoma openings causes physiological disturbance (decreasing of the stomatal conductance and gas exchange), which may further influence the water regime and photosynthesis (Tomašević et al., 2005).

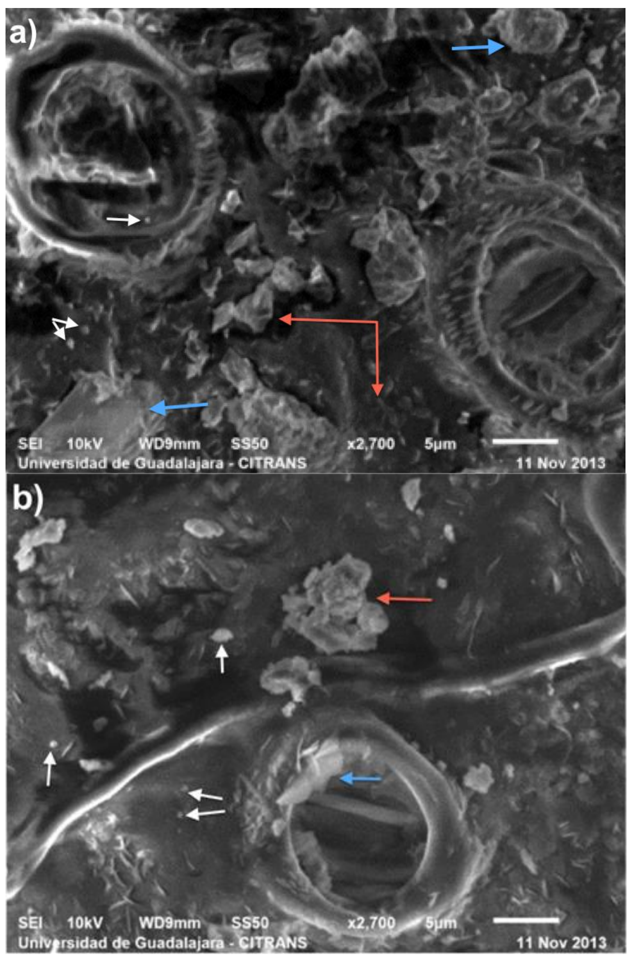

Figure 4 Abaxial face *Ficus* leaf SEM images for the samples (a) 170 and (b) 76. The variety of particles is indicated with arrows. Individual small particles (white), individual big particles (blue) and aggregates (red)

By using elemental mapping with EDS, it was possible to identify the presence and prevalence of elements present on the surface of the leaves.

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In almost all the samples appearing the main elements O, Ca, K, Si, and C. Some these elements are most likely to be of natural origin (coarse particles) but fine coal ash emitted from anthropogenic sources from combustion processes should not be ruled out due to the rounded shape of the particles and smaller size. The presence of heavy metals such as Pb, Cd, Co, Ni, Cu, Zn originated by industrial processes (Tomašević et al., 2005) was also abundant in many sites.

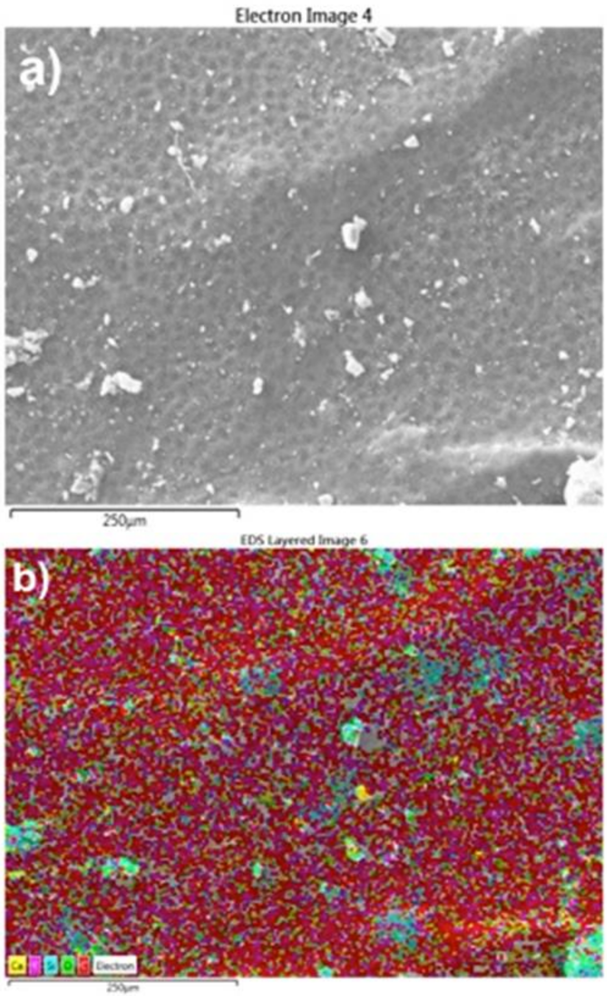
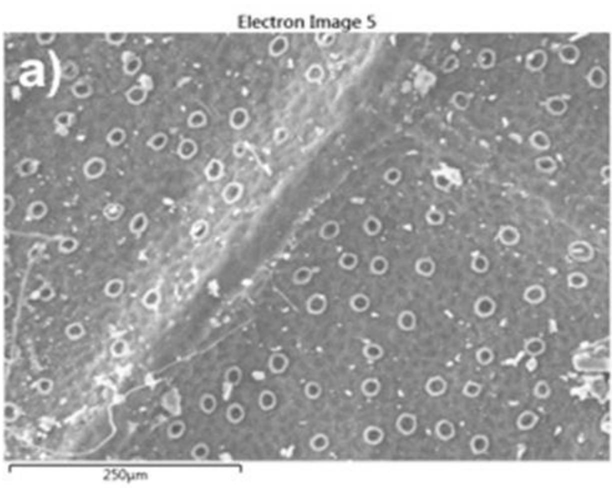


Figure 5 SEM image of a selected area (a) and EDS mapping (b) of the sample 170



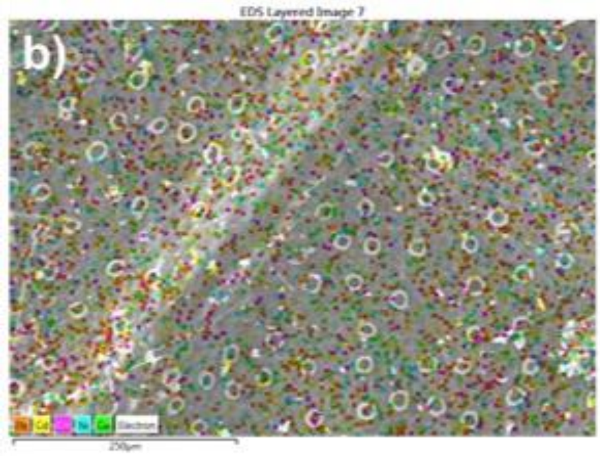
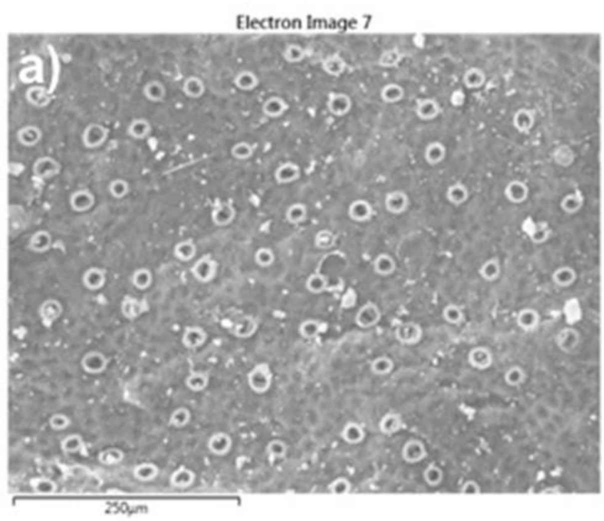


Figure 6 Abaxial face *Ficus* leaf SEM image (a) and EDS mapping (b) of the sample 170

For example, the SEM images and EDS elemental mapping of the sample 170 are shown in Figs. 5-6. The elements found were Ca, K, Si, O and C (Fig. 5) with varying concentrations of elements such as Pb, Cd, Co, Ni and Cu (Fig. 6). Similar results were found for single particles.

In another example, according to the EDS mapping on the abaxial side of the sample 125 among the main elements identified in the environment were Cr, Pb, Cd, Co and Ni (Fig. 7). The presence of similar heavy metals in many sites suggests a common anthropogenic source. For example, Ni has been identified in rural and urban areas and its principal source in street dust coming from the combustion of diesel fuel (Bhattacharya et al., 2013).



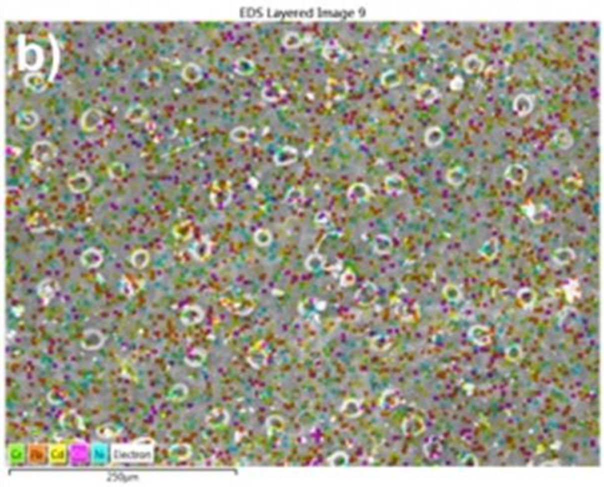


Figure 7 Abaxial face Ficus leaf SEM image (a) and EDS mapping (b) of the sample 125

In essence, it was feasible to determine by using the SEM technique the size and shape of the particles of dust deposited in both the abaxial side and adaxial faces of Ficus leaves, consistently observed in all samples, so it can be inferred that a significant amount of particulate matter floating in the atmosphere is retained in the tree leaves. The most representative elements of all sites were Si, Ca, K, O and C. However, it is important not to lose sight of the fact that the heavy metals Cd, Co, Cr, Cu, Ni, Zn, Pb in dust are predominant. Besides trace elements as W, Ti and Fe were also identified in some sites. In all cases, fine and ultrafine particles with spherical and oval shapes were abundant. These data correlate with those reported by Aguilar (Aguilar et al., 2011) for GMA, which refers to a magnetic carrier (Aguilar et al., 2011).

On the other hand, the atomic absorption spectroscopy method was used to measure the trace element concentrations such as Cu, Zn, Co, Ni, Cd, Cr and Pb in the *Ficus* leaves for the eleven monitoring sites (Fig. 8). The concentrations of Cu, Pb and Zn were substantially greater at sites 125, 105 and 122, with 38.7, 35.8 and 30.3 ppm, respectively. Specifically, the concentration of Cu was 3 times higher at site 125 (industrial area, tertiary road) than at site 170 (mixed area, rural road). Similarly, the concentration of Pb is 5 times higher at site 105 (mixed area, secondary road) than at site 87 (high density housing area, secondary road) and the concentration of Zn is 19 times higher at site 122 (mixed area, tertiary road) than at site 76 (mixed area, rural road).

The mixed area with secondary road (site 105) was the most contaminated with Cu (38.7 ppm), Co (8.7 ppm), Cd (8.4), Ni (11.4 ppm) and Pb (35.8 ppm). The most abundant element found in a mixed area with tertiary road (sample 122) was Zn with 30.3 ppm. Finally, the concentration greater of Cr (13.8 ppm) was found at site 173 (low density housing area, tertiary road). Consequently, at site 105 (industrial activities and relating to trade, services and housing) was the most contaminated with seven elements found, five of these have the highest concentration of all monitoring sites.

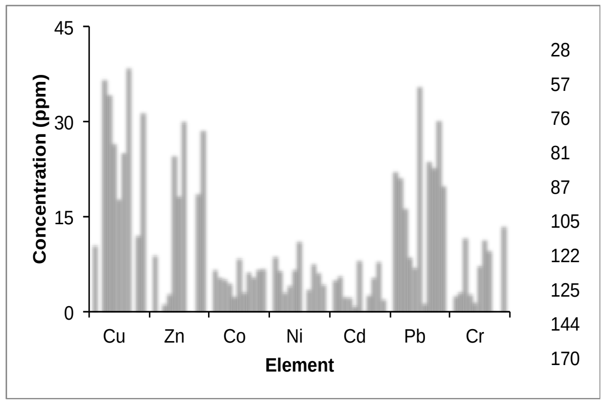


Figure 8 Distribution of heavy metals at eleven monitoring sites indicated with numbers to the right

In general, the presence of Cu, Zn, Co, Ni, Cd, Pb and Cr was identified for all the analyzed samples (except at sites 122, 170, 144 and 57). The characteristics of the sites and number of elements found are broken down in Table 1. As noted above, it can be seen that the sample 105 corresponding to a type of secondary road, and land use is Mixed is the one that presented a greater presence of the analyzed elements. In the following three sites 76 (mixed area, rural road), 87 (HDH, secondary road) and 81 (NUI, primary road), the presence of all heavy metals is a principal characteristic.

Land uses and types of roads are diverse in these sites. With respect to the sample 81, it is interesting to observe that it corresponds to a type of primary road and has strong presence of heavy elements. Similarly, at sites 28 (UI, tertiary road), 125 (industrial area, tertiary road) and 173 (LDH, tertiary road) were found all the analyzed heavy metals suggesting the strong impact of the urban infrastructure, industrial activities and housing on the air pollution.

For the samples 57 (UC area), 122 (mixed area) and 144 (industrial area) were found less than seven heavy metals, is the type of tertiary roads, i.e. the vehicular movement is relatively low; however, land uses are urban equipment (facilities to accommodate the required functions to satisfy community needs), type I (comprising a wide range of manufacturing activities), and mixed. For the sample 170 land usage is mixed and the type of road is rural were found all heavy metals except Cr.

Sample	Elements	Land use	Type of road
105	7	M	Secondary
28	7	UI	Tertiary
125	7	I	Tertiary
122	6	M	Tertiary
170	6	M	Rural
173	7	LDH	Tertiary
57	5	UC	Tertiary
76	7	M	Rural
144	5	I	Tertiary
87	7	HDH	Secondary
81	7	NUI	Primary

Table 1 Land use for analyzed samples

Conclusions

Collecting leaves of *Ficus* located in several areas of the GMA allows knowing the specific air pollution of a short period of time (since these are renewed constantly) by using SEM/EDS and atomic absorption spectroscopy. All types of particles adhere to the leaves (not selective) by the latex they have on their surface. Dust samples collected in this way represent the air quality at the zone level that is breathed by people, so the type of suspended particles can be analyzed quantitatively and qualitatively. *Ficus* leaves are stored as historical material for further verification or interpretation with another methodology.

The costs of this study are low compared with the installation and operation of the few existing monitoring stations in the GMA. High concentration of metals found shows that environmental quality in the GMA is bad and it follows that there must be a correlation with the health of the population. It is important to stress that, since the diameter of the particles present in the leaves of Ficus is mainly less than 2.5 μm ($PM_{2.5}$), the health problems of the population of the GMA associated with fine and ultra fine particulate matter are expected to be significant. Similar studies are needed periodically to compare the evolution of the environmental air quality in the GMA.

Acknowledgements

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