Elemental chemical characterization and morphology of PM10 using SEM/EDS in air quality monitoring station filters in Poza Rica, Veracruz, Mexico

Caracterização química elementar e morfologia de PM10 por meio de SEM/EDS em filtros da estação de monitoramento da qualidade do ar na cidade de Poza Rica, Veracruz, México

DOI:10.34115/basrv7n1-001

Recebimento dos originais: 04/12/2022
Aceitação para publicação: 05/01/2023

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ABSTRACT
Air quality has been a global problem for several decades and has had several approaches for its mitigation, analysis and quantification. There are several ways and methods to quantify primary and secondary air pollutants. Therefore, the objective of this research was to determine, through SEM-EDS analysis, the elemental chemical characterization and morphology of the compounds present in the tapes of PM10 BAM (Beta Attenuation Mass Monitor) equipment from air quality monitoring campaigns in the city of Poza. Rica, Veracruz in Mexico. The methodology consisted of collecting filter tapes from the air quality monitoring stations used in the evaluation period, sampling and analysis was performed by the SEM/EDS scanning electron microscopy technique. The results allowed identifying in the samples the elemental chemical characterization and morphology of the particles retained in them.

Keywords: PM10, PST, particle morphology, chemical characterization, BAM, SEM/EDS.

RESUMO
A qualidade do ar é um problema global há várias décadas e tem tido várias abordagens para a sua mitigação, análise e quantificação. Existem várias formas e métodos para quantificar os poluentes atmosféricos primários e secundários. O objetivo dessa pesquisa, portanto, foi determinar, por meio da análise SEM-EDS, a caracterização química elementar e a morfologia de compostos presentes nas fitas do equipamento PM10 BAM (Beta Attenuation Mass Monitor) a partir das campanhas de monitoramento da qualidade do ar em Poza. Rica, Veracruz no México. A metodologia consistia na recolha de fitas filtrantes das estações de monitoramento da qualidade do ar utilizadas no período de avaliação, na sua amostragem e na análise realizada pela microscopia eletrônica de varredura SEM/EDS. Os resultados permitiram identificar nas amostras a caracterização química elementar e morfologia das partículas retidas nelas.

Palavras-chave: PM10, PST, morfologia de partículas, caracterização química, BAM, SEM/EDS.

1 INTRODUCTION

Air pollution is a global public health problem. According to the World Health Organization (OMS, 2018), the exposure to poor air quality is responsible for around 7 million deaths per year worldwide, having different repercussions on environment and human health. Among these pollutants, particles stand out, also known as suspended particles, aero particles, particulate matter, and aerosols, which are some of the terms used to name the mixture of microscopic compounds in the form of aerosols and fine solids suspended in the air. All particles with a diameter ≤ 10 μm are called PM10, as well as all particles with a diameter ≤ 2.5 μm called PM2.5 which are generated by a wide variety of anthropogenic and natural sources.

Studies from the STATE OF GLOBAL AIR (2020) report have described the increase in suspended particles in the world over 19 years (2000-2019). For the year 2019, 36,600 deaths were registered because of PM2.5 in Mexico caused by Atmospheric Pollution.

According to the National Report on Air Quality 2017 (INECC, 2018) it is observed two serious debts with people in this matter: first, the generation and access to reliable and trustworthy information of air quality, since there is no updated data over what we are breathing to develop effective pollution mitigation plans. On the other hand, the other great debt is the improvement in the air quality that we breathe day by day, since the data offered reveal very low percentages of compliance with the maximum limits allowed for the presence of contaminants established in the official standards, which are usually measured on equipment with a little or null quality control and maintenance.

Atmospheric monitoring makes it possible to determine the air pollution levels and meteorological conditions that favor the transport and dispersion of pollutants, and its contributions are essential to establish pollution control measures and policies to protect the health of the population. For this reason atmospheric monitoring stations are essential to generate reliable information, which will help us to formulate air quality standards, as well as verify compliance. The information collected will be valuable in the academic, scientific, informative spheres and civil society.

Therefore, the objective of this work was to determine through SEM-EDS analysis, the elemental characterization and morphology of the compounds present in filtering tapes of the PM10 BAM equipment (Beta Attenuation Mass Monitor) coming from air quality campaigns and air quality monitoring station in the city of Poza Rica, Veracruz state in Mexico.

2 THEORETICAL FRAMEWORKS

2.1 BACKGROUND

Mexico as many countries have developed their economy under the impulse and development of activities immersed in the oil industry, commerce, construction of cities and roads, among many activities where different pollutants are emitted into the atmosphere. From this perspective, urban growth promoted by anthropogenic activities emits various pollutants into the atmosphere, and in synergy those emitted and generated in nature are added. PST, PM10, and PM2.5 are some of them, which according to the WHO, cause several respiratory diseases and are also associated with nervous or cardiovascular diseases (OMS, 2018).

Nature transports a lot of elements such as Saharan dust (Prospero and Mayol-Bracero, 2013), transported to Barbados and Miami, where elements such as Iron (Fe), Phosphorus (P) and compounds acidify rain and soils in the region. The impact on health implied by the estimated concentrations and regulatory measured in summer season where the highest incidence of this transport occurs slowly. Dimitriu and Kassomenos (2018) found elements such as Calcium (Ca), Silicon (Si), Titanium (Ti), Magnesium (Mg) and Fe, and some health and mortality relationships because of PMCOARSE material (PM10+PM2.5) associated with cardiovascular and respiratory diseases, mainly in Barcelona, Italy, among others. Ramírez-Romero et al 2021, carried out a study on the arrival of Saharan dust in Yucatán and its effects on air quality, they Observed several compounds such as Fe, Aluminum (Al), Si, Ca, Sodium (Na), P, Mg, Manganese (Mn), Ti, Chlorine (Cl), Zinc (Zn), Potassium (K), Sulfur (S), Copper (Cu) and Nickel (Ni).

Slezakova *et al.* (2009) in Porto in Portugal found in their analysis of traffic emissions through morphological characterization and elemental composition SEM-EDS, the influence of this type emissions has on the air quality (Quintas *et al.*, 2022) associated with PM10 and PM2.5 for this work they made use of polytetrafluoroethylene (PTFE) filters, where the SEM-EDS analysis found 25 elements such as Na, Mg, Al, Si, P, S, Cl, K, Ca, Mn, Fe, Cu, Zn, lead (Pb), lanthanum (La), cerium (Ce), Ti, barium (Ba), molybdenum (Mo), chromium (Cr), cobalt (Co), vanadium (Va), Ni, tin (Sn), and antimony (Sb).

Angulo *et al.* (2011) in Colombia, used the SEM-EDS method to determine the elemental composition of particles emitted by the mining industry, determining the morphology. They found quartz, feldsphates, altered grains, bioxites, muscovites, chlorites, calcites, among other organic materials. Franco Pineda (2020) in his degree work applied the SEM-EDS to analyze and characterize particulate matter in the city of Manizales in Colombia found ions, sulfates, and phosphates in the elemental characterization in 18 filters of PM10 and PM2.5 and the morphology associated with sources related to the steel and welding industry, vehicular traffic, Diesel combustion, volcanic ash and mineral materials.

2.2 PM2.5, PM10 AND PM COARSE

Particles atmospheric pollutants that exist in the atmosphere due to natural and anthropogenic causes. The cycles in the atmosphere generate compounds called aerosols, which are small liquid or solid particles that are transported with other compounds such as pollen, mineral soil particles, among others that can come from fixed or mobile human sources. These particles, according to the WHO (2014), are the cause of many human diseases associated with both the respiratory and nervous systems or the cancer. Airborne particles in suspension can have different sizes such that, the diameter can vary between nanometers (nm) and tens of microns (µm). The different ranges of particle sizes are called "modes" and are mostly related to the formation mechanism; the main modes are Nucleation, Aitken, Accumulation and Thick Mode (Warneck, 1988; U.S.EPA, 1996; Seinfeld and Pandis, 1998).

The denomination of the particles included in certain granulometric ranges varies depending on the field of study. In atmospheric sciences, particles with a diameter less than 0.1µm (PM0.1) are called “ultrafine particles” and particles with a diameter <1µm (PM1) are called “fine particles”, while in epidemiology this last definition covers even particles diameter <2.5µm. Consequently, in epidemiological studies reference is made to coarse particles from 2.5 µm in diameter, while in atmospheric sciences particles with a diameter greater than 1 µm are considered coarse. The levels of atmospheric particulate matter are expressed in units of concentration of mass, volume, surface, or number of particles per unit volume of air (Bengochea, 2007).

2.3 PARTICLE COMPOSITION

Particulate material has a very different nature and chemical composition, since there are several types of material that can reach the atmosphere from different sources. Thus, a sample of material does not unequivocally indicate the source of origin, but it does reduce all possible to a limited number (Bengochea, 2007). As can be seen, there are mineral particles such as calcite ($CaCO_3$), quartz (SiO_2), dolomite [$CaMg(CO_3)_2$], clays [especially kaolinite, $Al_2Si_2O_5(OH)_4$, and illite, $K(AlMg)_3SiAl_{10}(OH)$], feldspars [$KAlSi_3O_8$ and $(Na,Ca)(AlSi)_4O_8$] and lower amounts of calcium sulfate ($CaSO_4.{}_2H_2O$) and iron oxides (Fe_2O_3), among others (Glaccum and Prospero, 1980).

Colloidal aerosols of solid or liquid particles are emitted by the seas and oceans and those of the natural sources of sulfate precursor gases from biogenic and volcanic emissions (Andreae, 1997). The second natural source of carbon dioxide (SO2) being volcanic emissions, which inject large quantities at high levels into the atmosphere causing sulfate aerosol precursors (Keppler, 1999).

Carbon present in the atmosphere (total carbon, TC) can be found as elemental carbon (EC), organic carbon (OC) and carbonates ($CaCO_3$ and $MgCO_3$). There is also the term “soot”, which encompasses fine EC and OC particles of primary origin, the result of incomplete combustion processes (automobiles, fuel-oil, coal, biomass burning), common in urban areas (Husain, 2007). EC particles are usually primary and anthropogenic, originating from incomplete combustion processes of fossil fuels, showing a granulometry of approximately 0.1 µm.

2.4 MORPHOLOGY AND AIRBORNE PARTICLES

Particles from several natural or anthropogenic sources, due to their origin, are classified as natural and artificial solid particles. Table 1 shows their origin.

Table 1.- Classification of natural particles and origin

Classification	Origin
Minerals	Ground dust, imported dust, marine minerals, extractive activities if they are like that of the surface soil, if they are not, they are considered artificial.
Fossil	Fossil plankton, algae, fungi, pollen, seeds, and other fossilized micro-structures
Biomass	Vegetable or animal remains, fungi, bacteria, pollen, seeds, if the carbon-silicon relationships make us suspect structures in an organic state.

Adapted from: http://www.juntadeandalucia.es/medioambiente/consolidado/publicacionesdigitales/60-149_CARACTERIZACION_DE_PARTICULAS/60-149/5_CARACTERIZACION_Y_SELECCION_DE_PARTICULAS.PDF

Artificial particles are considered this way because they are the product of human action as part of their construction, transportation, and production processes, among others. In this group it can be classified as shown in Table 2.

Table 2- Classification of artificial particles and sources.

Type of particle	Origin or source
Industrial combustion	Emitted from chimneys, burners and industrial equipment.
Industrial activities	Dust emitted from industries such as mining, steel, cement, construction and public works.
Domestic combustión	Domestic heating that uses coal, fuel oil, LP gas, butane, diesel oil, among others.
Road transport (Traffic)	Fuels used such as gasoline, diesel, LP gas, among others.
Melting	Proveniente de industria metalmecánica y siderurgia.
Other	Difícil clasificación y especificidad

Adapted from: http://www.juntadeandalucia.es/medioambiente/consolidado/publicacionesdigitales/60-149_CARACTERIZACION_DE_PARTICULAS/60-149/5_CARACTERIZACION_Y_SELECCION_DE_PARTICULAS.PDF

These particles present several shapes, sizes, textures, and aspects ranging from organic, amorphous, to crystalline or spherical forms with different textures and sizes as mentioned in previous sections. Based on their morphology, these particles can be classified as shown in Table 3.

2.5 EFFECTS OF PARTICULATE MATERIAL

Air pollution represents a significant environmental risk to health. Diminishing air pollution levels, countries can reduce the burden of disease from stroke, lung cancer, chronic and acute lung diseases, including asthma (Zimmermann, 2011).

The new estimates are based on the latest WHO mortality data and evidence that exposure to air pollution poses a health risk. Estimates of human exposure to air pollution in different parts of the world were made using new global data mapping that included data satellite, measurements from ground-based monitoring stations, and data on pollutant emissions from air sources fundamentals, as well as models of air pollution movement patterns (OMS, 2014).

Table 3.- Morphology of artificial and natural solid particles

Type	Origen	imagen
Cenosphere	Coming from combustion, normally fuel oil, with carbonaceous matrices and high sulfur content. Contains trace metal.	

Smooth spherical	Coming from the combustion of mineral coal, with a silica or aluminum-silica matrix	
Rough spherical	Coming from metal smelting furnaces, composed of metal oxides.	
Irregular	It come from land or soil, but also from deforestation processes due to works or constructions; Their matrices can be calcium, siliceous, aluminous and their variants.	
Amorphous	It is formed by aggregates of smaller ones, of variable composition due to their origin.	
Crystalline	Natural or artificial from dissolved salts that crystallize by evaporation; usually composed of chlorides or sulfates.	
Biological	Plant or animal origin, fossilized or not, easily distinguishable; their structures are normally based on carbon and if they are fossils on silicon.	

Source: adapted from http://www.juntadeandalucia.es/medioambiente/consolidado/publicacionesdigitales/60-149_CARACTERIZACION_DE_PARTICULAS/60-149/5_CARACTERIZACION_Y_SELECCION_DE_PARTICULAS.PDF

Particles are classified in different ways: by their effect on human health, as a product of a natural or anthropogenic process, and by their physical characteristics, as shown in Table 4.

Table 4.- Particles and effects on human being.

Particle size	Effects
Settleable particles (> 10 µm).	In general, they do not represent significant risks to human health.
Particles smaller than 10 micrometers, PM10 (<= 10 µm).	They are considered harmful to health because they are not retained by the natural cleaning system of the respiratory tract.
Particles smaller than 2.5 micrometers PM2.5 (<= 2.5 µm).	They represent a greater risk to human health, may be a factor of premature death in the population

Source: (Lopez, 2011)

2.6 BETA ATTENUATION MASS MONITOR (BAM)

The Met One Instruments BAM 1020 Beta Attenuation Mass Monitor automatically measures and records ambient particle mass concentration levels using the beta attenuated principle. This method provides a simple determination of the ambient concentration of particulate matter in mg/m3 or µg/m3. A small 14C (carbon 14) element within BAM 1020 provides a constant source of beta rays (PERSIS, 2018).

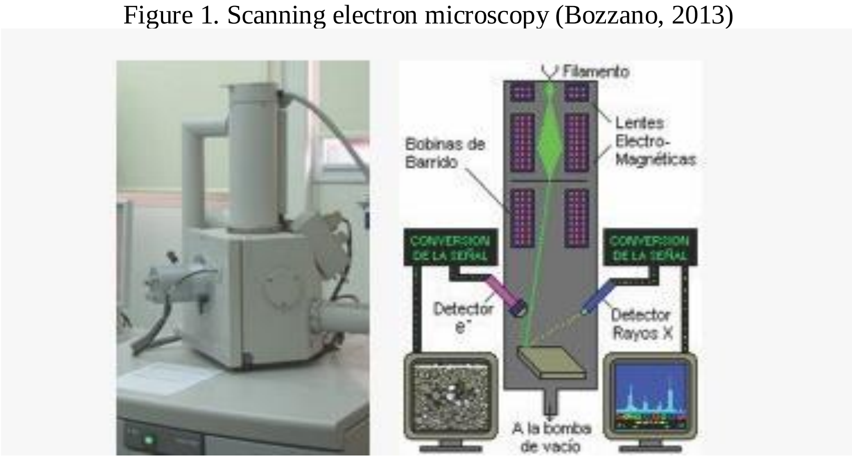
ISSN: 2595-3621

2.7 SEM-EDS ANALYSIS

The analytical technique provides quantitative and qualitative information on the surface of a sample, identifying the weight of the elements. This technique is combined with the detection of secondary radiation emitted (X-rays) when a high intensity electron beam collides with the material in the path, releasing in this process an energetic charge of at least 20 kV electron and this allows the analysis of the element and represent its composition in an EDS spectrum. The technique makes use of flat materials and covers that allow optimal reading of the analyzed material.

The scanning electron microscope (SEM, Scanning Electron Microscope for its acronym in English), allows the observation and superficial characterization of organic and inorganic materials, giving a fast, efficient, and simultaneous morphological information and chemical composition of the analyzed material (INECOL, 2021). The images of an electron microscope are obtained by detecting, processing, and visualizing the signals resulting from the interactions between a beam of high energy electrons with matter. (JEOL, s.f.). These interactions can provide information about topography, composition, and structure. According to the three xyz axes and vary its orientation according to two axes of rotation. The detectors are placed in this chamber to record the different signals emitted by the sample Figure 1 shows a SEM equipment (Bozzano, 2013).

Figure 1. Scanning electron microscopy (Bozzano, 2013)



3 METHODOLOGY

3.1 DESCRIPTION AND LOCATION OF THE STUDY AREA

Figure 2 Location of the city of Poza Rica Within the state of Veracruz (CEIEG, 2019)

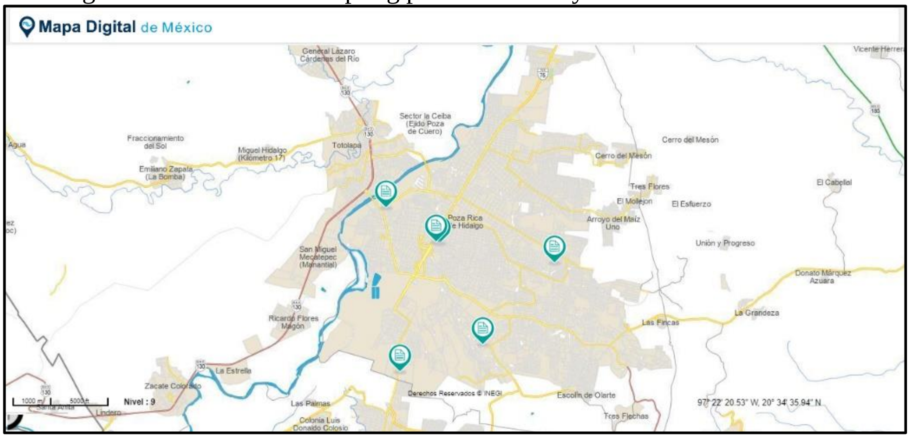
The work considered the monitoring equipment used in the municipality of Poza Rica de Hidalgo, Veracruz in Mexico shown in figure 2. The city is located between parallels 20° 29' and 20° 36' north latitude, and the meridians 97° 24' and 97° 29' west longitude, the altitude oscillates between 40 and 200 m asl (CEIEG, 2019). Poza Rica has a very high degree of human development due to the oil zone in which it is located, and an average annual rate of population increase of 0.73%. Poza Rica is a municipality in the northern zone of the state of Veracruz and borders to the north with the municipalities of Tihuatlán and Papantla, to the east with the municipality of Papantla, to the south with the municipalities of Papantla and Coatzintla, to the west with the municipalities of Coatzintla and Tihuatlán (CEIEG, 2019), being for this reason the center of the Poza Rica Metropolitan Area. The urban area of Poza Rica and surrounding areas is growing on Paleogene sedimentary rocks and Quaternary soil, in hills with plains, low sierra and typical valley; on areas where originally abundant soils called Regosol (24%), Phaeozem (6%) and Vertisol (4%) (INEGI, 2009).

3.2 MUESTREO DE PARTÍCULAS

The sampling of particles was carried out in two historical moments, the first consisted of a monitoring campaign carried out in 2010, with an air quality monitoring equipment from the Universidad Veracruzana. This monitoring was carried out at 5 points distributed throughout the city for two weeks. From this process, the tapes of the PM10 BAM equipment were kept in sealed packages for later analysis, since at the time they did not have the means to carry out the tests. Two 20 mm long random samples were taken from this tape.

The rest of the samples were taken from the air quality equipment installed in the city of Poza Rica by the Veracruz State Secretary of the Environment (SEDEMA Veracruz Spanish acronym). The tapes were requested and stored in sealed packages, from this packages were taken the eight remaining samples for the analysis with the same length as the 2010 samples. This station is in the center of the city in the facilities of the Library Services Unit (USBI Spanish acronym) of the Universidad Veracruzana. Figure 3 shows the monitoring points in the city of Poza Rica.

Figure 3.- Location of sampling points in the city of Poza Rica – Veracruz



Source: Author. INEGI Digital Map, 2022.

3.3 SEM-EDS ANALYSIS

The analyzed tapes selected from the air quality monitoring equipment in Universidad Veracruzana and SEDEMA Veracruz of 2010, 2015 to 2018 were transported in sealed packages to avoid contamination during transport to the Mario Molina research center at UNAM in Mexico City (CDMX Spanish acronym). From a total of ten tapes obtained, ten pieces of 20 mm tape were randomly cut for analysis in the JSM-5900 scanning electron microscope. The samples were placed on a stage inside the JSM-5900 scanning electron microscope shown in figure 4.

Figure 4. High vacuum scanning electron microscope model: JEOL JSM-5910

In this investigation, the SEM was used in Resolution and Depth modes with a voltage acceleration of 20 kV, a Beam intensity between 6.00 and 10.00, and a working distance of between 15.00 mm (PM10) to obtain clear and sharp images.

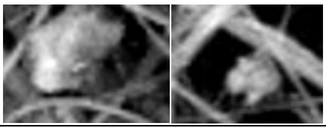
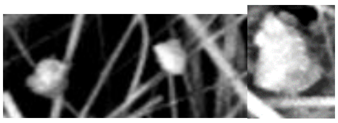
Subsequently, the x-ray signal that identifies the elements that make up the sample was analyzed, this analysis is known as energy dispersive x-ray spectroscopy (EDS, also abbreviated EDX) that validated the components found in each sample.

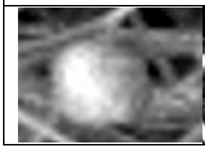
4 RESULTS AND DISCUSSIONS

4.1 MORPHOLOGY

The particle morphology as described in previous sections, presents various forms associated with the compounds that predominate in the location of the study area. Table 5 shows the shapes of particles found in the filter tapes of the BAM equipment.

Table 5.- Particles found in the BAM equipment samples

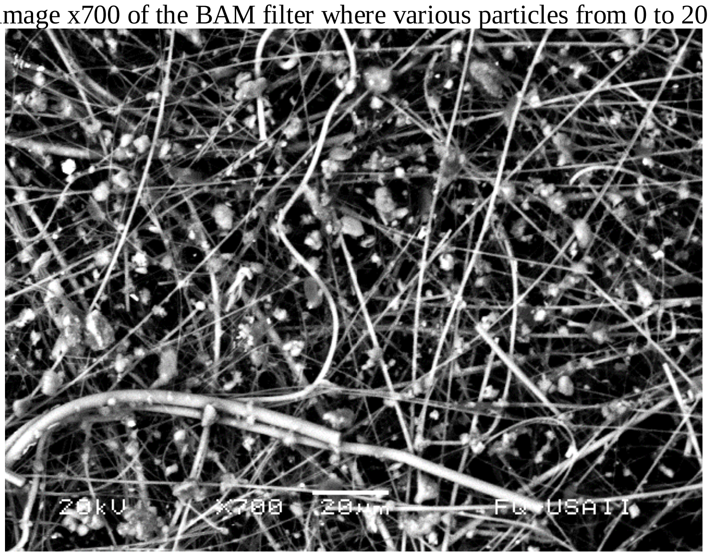
SEM Image	Particle	Description
	Biological	Particles of animal or vegetable origin fossilized or not. With structures normally based on carbon and if they are fossils in silicon with some exceptions, for example diatomaceous earth.
	Amorphous	Formed by groups of smaller ones. Variable composition according to its origin.
	Irregular	It usually comes from the ground or sometimes from emission sources. Its matrices can be calcic, siliceous, aluminous and their variants.

	Smooth spheric	Particles from combustion, normally with a silica or aluminum-silicon matrix
	Crystalline	Natural or artificial from dissolved salts in aqueous medium that conform crystals usually chlorides or sulfates, by evaporation of the liquid phase.
 	Cenosphere	Combustion particle, with carbonaceous matrix and high sulfur content. Contains characteristic trace metals based on size.

Source: Author

As observed in the previous table, the particles found according to their aerodynamic size ranged from 0 to 20 mm as shown in figure 5, the characteristic morphology coincides with those cited in the document of the Junta de Andalucía in Spain (2015), where the forms of particulate material and some probable associated sources are described, such as biological sources normally based on carbon or silicon, amorphous particles of several sources, irregular that can come from the ground; smooth spherical and cenospheres from combustion sources; and crystalline from aerosol sources of salts such as chlorides and sulfates.

Figure 5.- SEM image x700 of the BAM filter where various particles from 0 to 20 mm can be seen



4.2 SEM/EDS ELEMENTAL CHARACTERIZATION

The chemical characterization of the elements found in the filters and analyzed by means of the SEM/EDS technique carried out using a JEOL JSM-5910 model scanning electron microscope, is described from the series of 31 samples selected in the BAM equipment filters, which are summarized in Table 6.

As can be observed in figure 6, the elements found in the percentage elemental analysis in greater proportion were O, C, Si, Na, Al, Ba, Zn, Ca, K, Fe, Mg, S, Cl and only one shows traces of Ti. In general, all these compounds are associated with vehicle emissions, soot, and emissions from the metal-mechanic industry. Other associated emissions are linked to soil material, the construction and cement industry, the paint industry, combustion of petroleum gases, aerosols, and biological elements.

Figure 6.- Average percentage composition by weight of the elements found in the samples of the BAM particle equipment of the air quality station of Poza Rica, Veracruz.

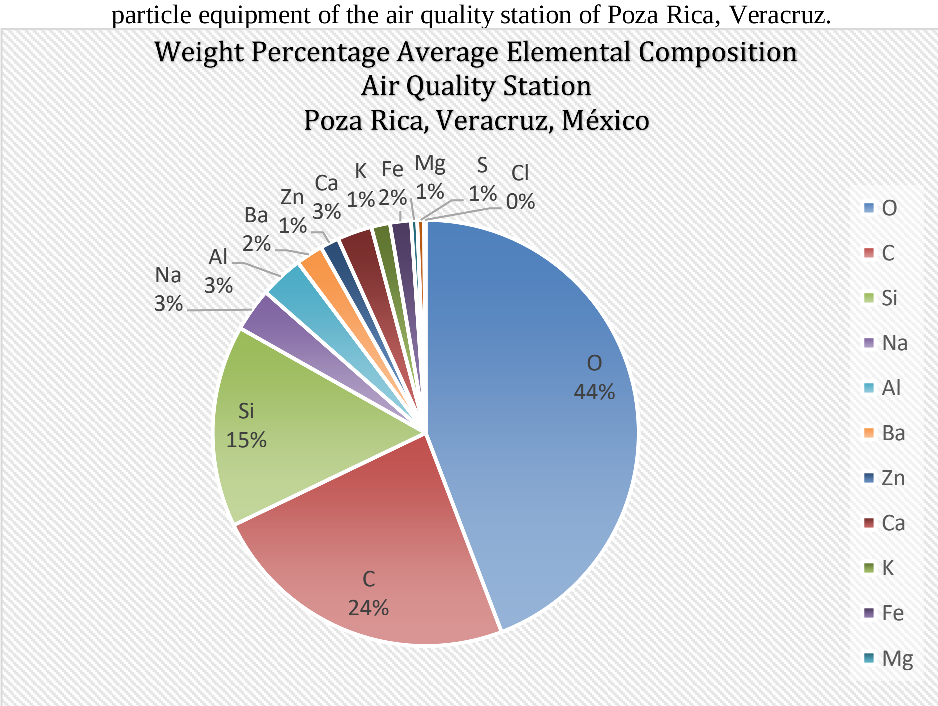


Table 6.- Summary of the percentage by weight of the elements found in 31 samples of the BAM particle equipment.

Compound	Min	Average	Std. Deviation	Max
O	39.00	44.25	2.64	50.40
C	16.90	23.70	2.94	28.90
Si	10.60	15.32	2.61	20.30
Na	2.20	3.30	0.52	4.40
Al	1.70	3.28	1.53	7.20
Ba	1.40	2.06	0.37	3.00

Zn	1.00	1.43	0.30	2.30
Ca	0.80	2.64	2.32	9.10
K	0.90	1.42	0.41	3.10
Fe	0.20	1.59	2.72	14.90
Mg	0.20	0.47	0.34	1.90
S	0.20	0.52	0.77	4.10
Cl	0.10	0.11	0.04	0.20
Ti*				

Note: * Ti only got a value of 0.01 in one sample

5 CONCLUSIONS

It is observed in the results that tapes from the BAM equipment allow for a reliable elemental chemical quantification by means of the SEM-EDS technique, and at the same time allows a rapid analysis of the particle morphology. This pair of techniques together give a first approximation to the elements that may be breathing in the city and municipality of Poza Rica de Hidalgo in Veracruz, which made it possible to determine for the first time what is being breathed in that metropolitan region with a high industrial development around of the oil.

Derived from the sampling points and in the years evaluated 2010, 2015 - 2018, it was observed that the sources of origin of the pollutants recovered from the references correspond in many cases to the sources that are listed as fixed or mobile, in general industrial sources, road traffic, construction works and other anthropic activities in the area, and also by natural sources such as those associated with the land and land use change operations.

The associated morphologies described in works of the Junta de Andalucía, correspond to a great extent to those found in the samples from the metropolitan area in Poza Rica. Size and shapes describe forms of particulate matter and some probable sources associated with biological sources usually based on carbon or silicon, amorphous particles from various sources, irregular that can come from the ground, smooth spherical and cenospheres from combustion sources, and crystalline from aerosol sources of salts such as chlorides and sulfates.

The elemental characterization found in the percentage elemental analysis in greater proportion O, C, Si, Na, Al, Ba, Zn, Ca, K, Fe, Mg, S, Cl and, traces of Ti in one sample. In general, all these compounds are associated with vehicle emissions, soot, and emissions from the metal-mechanic industry. Other associated emissions are linked to soil material, the construction and cement industry, the paint industry, combustion of petroleum gases, aerosols, and biological elements.

These results allow us to conclude that the elemental and morphological analysis of PM10 particles from samples from silica tapes from BAM equipment is feasible to preliminarily determine which elements may exist in the study area and their probable sources, thus fulfilling the objective of the study. worked.

Future work will consist of updating through current samples and from other air quality monitoring stations in the state to continue with these characterizations and carry out a more detailed chemical characterization with some other technique that identifies ions and cations in addition to the characterization elemental using the SEM-EDS.

ACKNOWLEDGMENTS

We thank the Universidad Nacional Autonoma de México (UNAM) for the support provided by the SEM/EDS analysis and, also to the environmental engineer Frida Cobos for her support in this project.

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