Morphological and chemical characterization of atmospheric particles PM10 in an urban site in Leon, Guanajuato, Mexico

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Abstract. Start PM10 concentrations and their trace metals content (Cd, Co, Cu, Fe and Zn) were determined in an urban site of Leon, Guanajuato during the rainy season 2018. PM10 exceeded the maximum permissible levels established by the Mexican Standards and WHO, constituting a potential risk to population health. Trace metals levels in PM10 were analyzed by Atomic Absorption Spectrometry, and in addition, morphology and elemental content were studied for some selected particles by Scanning Electronic Microscopy and Energy Dispersive Spectrometry. Chemical and morphological characterization revealed that Fe was the more abundant metal (0.986 µg m⁻³), followed in order of importance by Zn (0.243 µg m⁻³), due to thses metals are related to the crustal. Lower concentrations were found for Cu (0.140 µg m⁻³), Cd (0.119 µg m⁻³), Mn (0.070 µg m⁻³) and Co (0.043 µg m⁻³). Meteorological analysis showed that sources located at the SW and NE of the sampling site (industrial parks and facilities related to metals and mineral extraction) influenced the measured concentrations. Enrichment factors showed that all the measured metals were highly influenced by anthropogenic activity. Cancer Risk (CR) and Non-Cancer Risk Coefficients (cardiovascular and respiratory diseases) did not exceed the maximum permissible level established by EPA.

1. Introduction
The particulate matter present in the atmosphere is a set of substances in solid and liquid state, which is suspended in the air or, which are deposited on the surface very slowly when they reach a larger diameter. Some examples of particulate matter are soot, aerosols, sand, dust, etc. [1]. The increase in the concentration of atmospheric particles in large urban areas is related to negative effects on human
health [2], adding complexity to the fact that these particles contain a variable and complex mixture of different chemical elements and compounds that come from different sources [3]. Thus, PM10 atmospheric particles refer to particulate matter in size less than or equal to 10 micrometers, which may contain various elements, among which are trace metals, which play a very important role in human health problems because they are highly bio reactive and some of them are toxic for humans [4], and vegetation [5]. The metals present in the particles are found in the fine and ultrafine fractions (in the order of micrometers in diameter) [6], [7], [8], that facilitates their entry into the alveolar region of the lungs when they are breathed [9]. Despite its importance from a health point of view, the content of metals in PM10 particles is not regulated in Mexico, only the mass concentration of particles expressed in µg m⁻³ is regulated (Mexican standard: NOM-025-SSA1-2014) [10].

Atmospheric monitoring is one of the main indicators of air quality, in addition to representing a management tool to implement prevention and control actions for pollutants present in the atmosphere. In Mexico, automatic monitoring is carried out in the main cities of the country, usually measuring CO, O₃, NO₂, SO₂, PM10 and PM2.5; however, only the mass concentration of the particles is reported and no chemical speciation of their content is made. Some studies have been reported in Mexican cities on the levels of heavy metals in PM10, but most of these studies have focused on large metropolitan areas such as Monterrey and Mexico City [11], [12], [13], [14] and in the south of the Mexican Gulf [15]. In the center of the country there are some commercial and industrial areas with high demographic density, such is the case of Bajio region, where Leon is the most important city in this area. The city of León, has experienced an accelerated economic and industrial development that has resulted in increased levels of pollution and deterioration in air quality, reaching concentrations that may exceed the maximum permissible limits. Leon city is famous worldwide thanks to its shoe industry, and it is among the 5 cities with the highest number of inhabitants in the entire Mexican Republic (1 million 578 thousand 626 inhabitants). In this city, diverse economic vocations are consolidated, such as highly specialized health care and diagnosis institutions, higher education and research centers, as well as tourism and business activities. Therefore, the relative potential effects of the degradation of air quality on the health of population constitute one of the main concerns in this region. For this reason, this research project focuses on determining the concentrations in the atmosphere of particulate matter (PM10) of an urban site in the city of Leon, its content of heavy metals (Cd, Co, Cu, Fe and Zn) and morphology, to infer its possible sources and evaluate the risk to the health of the population in the study site. This study represents the first project focused on the speciation of some trace metals contained in PM10 particles in the city of Leon, Guanajuato in order to know in a preliminary way the health risk associated with the concentration levels found. This information will be useful to know not only the gravimetric concentration of the particles but also their metal content, since currently, none of the air quality monitoring stations in the city of Leon determine the content of metals in PM10. In addition, this information will serve as a basis for the future expansion of this monitoring network of trace metals in PM10 to other cities of the state of Guanajuato such as Celaya and Salamanca, since in the latter there are important sources of pollutant emissions to the atmosphere: an oil refinery and a thermoelectric plant. This will allow a comprehensive evaluation of health risks, since currently only the mass concentration of the particles is considered to evaluate the risk that this pollutant represents to human health.

2. Materials and Methods

2.1. Study Area

The municipality of Leon, is located in the state of Guanajuato. The territorial area of this municipality is 1,219.67 km², with an average height of 1,798 masl. This city limits to the north with the municipality of San Felipe, to the east with Guanajuato and Silao, to the south with the municipalities of Silao, Romita and San Francisco del Rincon and to the west with the municipalities of Purisima, Lagos de Moreno and the Unión de San Antonio (Municipal Government Program of Leon,
Guanajuato, 2018) [16]. The study site is located in an urban area in the center of the city of Leon, within the facilities of one of the 3 air quality monitoring stations belonging to the State Air Quality Information System (SEICA) at 21.133° N and 101.680° W (See Figure 1). In the surroundings near the study site is located the General Hospital of Leon, leather tanning factories and streets that connect with the main avenues of the city.

2.2. Sampling Methodology
The sample collection process was carried out using an air sampler, MiniVol TAS from Airmetrics, which is a portable sampler for particulate matter and non-reactive gases. The sampling technique used by the MiniVol is a modification of the PM10 reference method described in the United States Code of Federal Regulations (40 CFR part 50, Appendix J) [17]. The sampling was carried out during the rainy season 2018. The sampler was placed in the place of interest, then a Whatman® brand 47 mm quartz fiber filter was placed into the filter holder and the equipment was operated at a controlled flow of 5 L/min of ambient air, which passed through a particle size separator and then through the filter during a sampling period of 24 h. This process was made every day during the 8-sampling days from August 6 to August 13, 2018, obtaining a total of 8 samples during the study period.

2.3. Analysis Methodology
The filters were subjected to acid digestion with 10 mL of aqua regia (25mL HNO₃ + 75 mL HCl) and 1.065 mL of HClO₄ and heating at 60 °C for 70 minutes, until dryness. Then 20 mL of hot water were added, filtered and made up to 50 mL using deionized water, storing the extracts in polypropylene containers until later analysis [18]. Standard solutions of Cd, Co, Cu, Mn, Fe and Zn were prepared from 1000 ppm HYCEL brand standard solutions for atomic absorption. For each metal, from each stock solution, 7 dilutions were prepared at different concentrations and calibration curves were constructed. The calibration range adopted was 5–500 µg L⁻¹ (seven point calibration) for each metal; the correlation coefficients (R²) for the calibration curves were all greater than 0.998. A Thermo Scientific ™ iCE 3000 ™ Series AAS Atomic Absorption Spectrophotometer was used for samples analysis. The measurements were carried out according to the standard conditions recommended at the specific wavelengths for each metal according to standard analytical US EPA Methods IO.3.1 [17] and IO.3.2 [20]. A deuterium lamp was used as a background corrector in all measurements [19]. Limits of detection (LODs) of trace metals were in the range of 0.003–0.06 µg L⁻¹ and recovery percentages of trace metals from the spike method (n = 3) ranged from 97.4% to 104.56%, and expanded uncertainties ranged from 0.01262 to 0.05234. In order to verify reproducibility and low background
metal concentrations of reagents and filters, 5% of the total number of samples were taken as blanks and analyzed for the presence of trace metals.

2.4. Morphological characterization of PM10 using Scanning Electron Microscopy

The morphology of the particles and their elemental composition with respect to metal content was evaluated using a Hitachi FLEXSEM-SU1000 (Scanning Electron Microscope) scanning electron microscope equipped with an Energy Dispersive (EDS) X-ray detection system TM 40000 Quantax 75/80 of Bruker that works at 20 kV, located in the Corrosion Research Center of the Autonomous University of Campeche. The low vacuum scanning electron microscope was calibrated with a copper (Cu) grating, a filament current of 300 mA, and a working distance of 5 cm. A 1 mm x 0.5 mm rectangle was cut in each filter for analysis and these sections of the filters were analyzed by SEM-EDS. Ten particles from each sample were randomly selected and images were taken at magnifications of 500X, 1500X, 2500X, 5000X, 8000X, 10,000X, and 25,000X. Spectra for individual particles were obtained by EDS, with a measurement time of 100 s for each particle.

2.5. Morphological characterization of PM10 using Scanning Electron Microscopy

Pearson's correlation was used to identify bivariate relationships between measured metals, criteria pollutants, and meteorological variables. A variance analysis was applied to the data set using non-parametric Friedman test (α = 0.05) in order to determine if there were significant differences among the trace metals contained in PM10. Principal component analysis (PCA) was performed to explain multivariate variation and discover the structure of the data set. This method has been widely used in environmental studies to identify patterns in data [22]. ACP results generally are represented in bi-plots or factor loading tables, which reveal correlations between observations. The information disclosed by ACP is useful to identify whether a pollutant is secondary or primary, or to identify the specific source of the pollutants.

2.6. Meteorological Analysis

Wind roses were built for each of the sampling days, in order to know the distribution of the wind direction and speed at the study site, using the WRPLOT software (Wind Rose Plots for Meteorological Data developed by Lakes Environmental, available at: https://www.weblakes.com/products/wrplot/index.html) [23]. This analysis let know the dominant direction of wind, which was related to the levels of concentrations of measured air pollutants in order to infer the probable location of sources of this pollutants.

2.7. Health Risk Assessment

Exposure has been expressed in terms of lifetime average daily dose (LADD). This value makes it possible to calculate the corresponding level of cancer risk (CR) for each metal, considering two representative groups: adults and children. LADD is used to determine the amount in which a pollutant has adverse health effects when it is absorbed by the body over a long period of time, and is calculated with the following equations [24]:

\[
LADD = E \times C
\]  
\[
E = \frac{IR \times ET \times EF \times ED}{BW \times AT \times 365}
\]  

Where for equation (1), C (mg m⁻³) is the concentration of the metal in question in PM10, which is assumed to be the same at the point of exposure, while E (m³ Kg⁻¹ day⁻¹) is obtained from equation (2), where IR (m³ h⁻¹) is the air inhalation rate, ET (24 h day⁻¹) is the exposure time, EF (350 days year⁻¹) is the exposure frequency, ED (years) is the duration of exposure, BW (Kg) is the body weight and finally AT (days) is the average time, used as ATc for carcinogenic risk and as ATn for non-
carcinogenic risk. The parameters used for the estimation of cancer risk (CR) and non-cancer risk coefficients (HQ: hazard quotient) are shown in Table 1.

### Table 1. Parameters used for the estimation of cancer risk (CR) and non-cancer risk coefficients (HQ: hazard quotient)

| Parameters                  | Symbol | Unit of measurement | Values  |
|-----------------------------|--------|---------------------|---------|
| Inhalation rate             | IR     | m³/h                | 0.9     | 0.7    |
| Body weight                 | BW     | Kg                  | 70      | 15     |
| Exposure time               | ET     | h/day               | 24      | 24     |
| Exposure Frequency          | EF     | Day/year            | 350     | 350    |
| Exposure duration           | ED     | years               | 24      | 6      |
| Average Time                | *ATc   | days                | 25550*  | 25550* |
| Average time                | *ATn   | days                | 210240** | 52560*** |

* 25550 days corresponds to the age according to the typical life expectancy (70 years x 365 days / year); ** ED (24 years) multiplied by 365 days / year x 24 h / day; *** ED (6 years) multiplied by 365 days / year x 24 h / day

CR represents the increased probability of occurrence of diseases caused by tumors above the general average due to the impact of compounds of those that produce carcinogenic effects. The CR for carcinogenic substances is considered $10^{-6}$ (lifetime risk of developing cancer is 1 in 1,000,000). Values below $10^{-6}$ for individuals are considered negligible. For carcinogenic substances the Cancer Risk (CR) is determined with the following equation:

$$CR = LADD \times CSF$$

(3)

where CR is the probability of cancer occurrence in the exposed population during a life time of 70 years; It is determined by multiplying the LADD (mg / Kg day) and CSF (cancer slope factor) (mg / Kg day). Carcinogenic risk is defined as the increased probability of a person experiencing cancer during a lifetime as a result of exposure to a specific carcinogenic potential. The CSF is calculated with the following equation:

$$CSF = IUR \times \frac{BW}{(IR \times ET)} \times 1000$$

(4)

IUR (Inhalation Hazard Unit) is a reference value reported in the database of the Environmental Protection Agency [25]. Table 2 shows the values for IUR and RfC. Regarding the inhalation risk units (IUR) and the reference concentrations (RfC), only values for Cd, Co and Mn are reported. Regarding the non-carcinogenic risk, the HQ (risk coefficient) is calculated as reported in equation 5:

$$HQ = \frac{ADI}{RfD_i}$$

(5)

For HQ it is considered that there is an exposure level (RfDi) below which it is unlikely even for a sensitive population to experience adverse health effects. When the exposure level (ADI) exceeds the value indicated as 1, there may be concern about possible non-carcinogenic health effects; higher THQ values (> 1) suggest higher levels of concern. RfDi (mg / Kg day) represents the inhalation dose at which there are no adverse effects and is defined by equation 6:

$$RfDi = RfC \times \frac{20 m^3}{day} \times \frac{1}{70 Kg} \quad (Using \ 7.6 m^3 \ and \ 15 Kg \ for \ children)$$

(6)
Using 7.6 m³ and 15 Kg for children. ADI (mg/ kg day) is the estimated dose that the recipient receives from exposure to polluted air [24], and is calculated with the same variables used for cancer risk:

\[ ADI = E \times C \]  

(7)

Table 2. Inhalation Risk Unit (IUR) and Reference Concentration (RfC) (EPA) [22]

| Metal | CAS   | IUR(µg m⁻³⁻¹) | RfC (mg m⁻³) |
|-------|-------|---------------|--------------|
| Cd    | 7440-43-9 | 1.80 × 10⁻³ | 1.00 × 10⁻⁵ |
| Co    | 7440-48-4 | 9.00 × 10⁻³ | 6.00 × 10⁻⁶ |
| Mn    | 7439-96-5 | .............. | 5.00 × 10⁻⁵ |

* CAS unique number of the chemical element

3. Results and Discussion

3.1. Gravimetric concentrations of PM10

The morphology of the particles and their elemental composition with respect to metal content was evaluated using a Hitachi PM10 concentrations recorded during the study period were within the range of 17.48 (µg m⁻³) to 45.32 (µg m⁻³), with an average of 28.04 (µg m⁻³). The maximum permissible limit for PM10 established in the official Mexican standard (NOM-025-SSA1-2014) [10] is 75 (µg m⁻³), whereas, the guidelines of the World Health Organization (WHO, 2005) [24] establish a limit of 50 (µg m⁻³). During the study period these limits were not exceeded; the maximum value recorded was 45.32 (µg m⁻³).

3.2. Trace metals concentrations in PM10

Six metals were determined in this study: Cd, Cu, Co, Mn, Zn and Fe. The results can be observed in Figure 2. Fe and Zn were the metals with the highest concentrations 0.986 µg m⁻³ and 0.243 µg m⁻³, respectively. Major elements such as iron and zinc can come from the re-suspension of dust when passing vehicles and from its combustion in the case of diesel vehicles. Its higher proportion compared to other metals is explained by its geological origin, being part of the dust re-suspended in the ground by the wind and transport [27]. This agrees with the results observed in other studies as shown in Table 3, where these metals were also reported with higher concentrations. According to Table 3, the results observed for Copper (Cu) are higher than those reported in other studies carried out in Mexico (Hidalgo, Colima, Tamaulipas and Puebla), however, they reach similar concentrations with respect to studies carried out in Bogotá, Colombia and Agra, India. Cu is a metal that occurs naturally throughout the environment, in rocks, soil, water, and air. It is an essential element in plants and animals (including humans). Copper compounds are commonly used in agriculture to treat plant diseases such as mold, to treat water, and as preservatives for wood, leather, and fabrics.

Sources of exposure are smoke, copper smelting and related metallurgical operations, welding, and metal powders and copper salts [28]. In particular, copper emissions into the atmosphere can be related to the burning of fossil fuels (vehicle sources), confirming results with other studies [29]. The high concentrations of copper can be attributed to the presence of metallic copper particles, which is emitted by nearby bronze smelting processes [30]. The average concentration of copper present in the air is 0.05 µg m⁻³ but in polluted areas it can reach up to a thousand times that value. The cadmium (Cd) concentrations obtained in this study are higher than those observed in other cities in Mexico (Colima, Tamaulipas and Puebla) and in other cities around the world (Venice, Seoul and Bogotá). On the other hand, the results obtained are similar to those found in a city in China (Nanchang) and Iran (Agra) (Table 3). Cadmium is naturally present in the environment in the air, soils, sediments and even in uncontaminated seawater [31]. The main sources of pollution are non-ferrous metal mining...
metallurgy, iron and steel metallurgy, the manufacture of phosphate fertilizers, the incineration of waste wood, coal or plastics, the combustion of oil and gasoline and the applications industrial cadmium. Cd concentration in air in industrial areas ranges from 9.1 to 26.7 µg m⁻³, from 0.050 to 0.060 µg m⁻³ in urban areas and from 0.0001 to 0.006 µg m⁻³ in air in rural areas. Cd does not degrade and, once released into the environment, it remains in circulation [32]. The average value of Cd found in this study exceeds the maximum permissible levels established by the US EPA (11.8 times more), WHO (23.8 times more) and the European Community (23.8 times more) (See Table 4). Manganese (Mn) concentrations were higher than those found in other states of Mexico such as Hidalgo, Colima and Puebla and cities around the world such as Seoul and Venice, but lower than those found in Tampico, Bogotá and Agra (Table 3). Mn is used in the production of steel, carbon steel, stainless steel and high-temperature steel, cast iron, superalloys, dry cell batteries, matches, and fireworks [28]. It is a natural element that is found in low levels in water, air, soil and food. Manganese can be released into the air by industry or by burning fossil fuels. Elevated levels of manganese can be present in the air near iron and steel production plants, power plants and coke ovens and dust generated by uncontrolled mining operations. Manganese released from burning a gasoline additive also represents a source of manganese in the air. The most common route of exposure to high doses of manganese is through inhalation of contaminated air [33]. According to the Toxicological Profile of the ATSDR (Agency for toxic substances and disease registry) the average levels of manganese in the air are approximately 0.02 µg m⁻³. On the other hand, Chen and Lippmann (2009) [34] determined that the average concentration of manganese in fine particles (PM 2.5) in 13 cities of the USA (AIRS sites) was of 0.004 µg m⁻³. The average manganese concentrations found at the study site are 4.6 times higher than the maximum allowable level established by the World Health Organization (WHO) (Table 4). The Cobalt (Co) concentrations obtained in the present study are higher than those found in Puebla, the Metropolitan area of Mexico City and Venice. Lesser results are observed with respect to those found in Agra, India (Table 3). Cobalt is produced primarily as a by-product of mining and processing minerals such as copper and nickel. Various cobalt compounds are used as pigments in the glass and ceramic industries, as drying agents for paints, varnishes, printing inks, catalysts in the petroleum and chemical industries, and as trace metal additives for agricultural and medical uses. Traces of cobalt can be present in cement and various household products. Cobalt concentrations in ambient air are usually below 0.01 µg m⁻³ [35]. Higher cobalt levels can occur in air and water near industrial areas, particularly near industrial hard metal sites (0.61 µg m⁻³ has been reported in an industrial area) [28].

Table 3. Comparison of PM10 concentrations and trace metal content with other studies.

| Location                  | PM10 (µg m⁻³) | Cd  | Cu | Zn  | Fe | Mn | Co |
|---------------------------|---------------|-----|----|-----|----|----|----|
| This study                |               |     |    |     |    |    |    |
| Leon, Guanajuato (urban) | 28.04         | 0.12| 0.14| 0.24| 0.99| 0.07| 0.04|
| Tampico, Tamaulipas [11] | 23.44 - 60.67 | 0.0003| 0.03| 0.04| 0.24| 0.26| -  |
| Studies in Mexico         |               |     |    |     |    |    |    |
| Metropolitan Area         | 50 - 56       | 0.0010| 0.0750| 0.10| 1.00| 0.02| 0.0005|
| Puebla, Mexico City [36] | 55.9          | 0.005| 0.09| -   | 1.20| 0.03| 0.002|
Studies in other regions around the world

| Location          | Region   | Minimum (µg/m³) | Maximum (µg/m³) | Mean (µg/m³) | Standard deviation (n-1) |
|-------------------|----------|----------------|-----------------|--------------|--------------------------|
| Mexico            | [12]     | 0.0002         | 0.026           | 0.08         | 0.051                    |
| Tlaxcoapan,       | (industrial) |              |                 |              |                          |
| Hidalgo           | [13]     | 0.012          | 0.51            | 0.29         | 0.012                    |
| Colima,           | (industrial) |              |                 |              |                          |
| México            | [37]     | 0.0010         | 0.018           | 0.011        | 0.0010                   |
| (rural)           |          |                |                 |              |                          |
| Bogotá, Colombia  | [27]     | 0.026          | 3.15            | 0.91         | 0.026                    |
| (industrial)      |          |                |                 |              |                          |
| Agra, India       | [38]     | 0.01000        | 2.90            | 0.90         | 0.01000                  |
| (urban)           |          |                |                 |              |                          |
| Seoul, Korea      | [39]     | 0.0041         | 0.12            | 0.02         | 0.0041                   |
| (residential)     |          |                |                 |              |                          |
| Venice, Italy     | [40]     | 0.003          | 0.10            | 0.02         | 0.003                    |
| (urban)           |          |                |                 |              |                          |
| Nanchang, China   | [41]     | 0.11           | 0.24            | -            | 0.11                     |
| (industrial)      |          |                |                 |              |                          |
| Ahvaz City, Iran  | [42]     | 0.19           | 2.81            | -            | 0.19                     |
| (urban)           |          |                |                 |              |                          |

**Figure 2.** Boxplot and descriptive statistics for (a) Cd, Cu, Co and Mn concentrations; (b) Fe and Zn concentrations.
Zn concentrations; present in PM10 in the study site

**Table 4.** Maximum permissible limits in ambient air for some of the trace metals present in PM10 established in international standards

| Trace metal | Maximum permissible level (µg m⁻³) | Average Time | Regulation Organism             |
|-------------|-------------------------------------|--------------|----------------------------------|
| Cd          | 0.0100 Annual                       |              | USEPA [43]                        |
|             | 0.0050 Annual                       |              | WHO [44]                         |
|             | 0.0050 Annual                       |              | European Community [45]          |
| Mn          | 0.0150 Annual                       |              | WHO [46]                         |

3.3. **Influence of wind conditions on the trace metals concentrations**

From the wind roses analysis it was determined that the dominant wind during the study period blew from the SW. Figure 3 shows a representative wind-rose for the study site, as it can be observed the dominant wind came from SW. To the SW of the study site there are three tanneries (facilities focused on process that converts the skin of animals into leather), this process is associated with the release of copper into the atmosphere due to the additives that are used to preserve the leather [28]. In this direction is also Adolfo Lopez Mateos Boulevard, one of the main avenues of the city with high vehicular traffic. The burning of fossil fuels such as gasoline contributes significantly to the emissions of heavy metals into the atmosphere (Co, Cu, Mn, Zn and Cd). To the NW of the study area there is another important avenue: Paseo de los Insurgentes, which connects to the Lagos de Moreno highway where around thirteen thousand five hundred cars circulate daily. In this direction there are also foundry facilities for metal smelting such as iron, bronze and aluminum.

![Representative wind rose for the study site during the sampling period.](image)

Figure 3. Representative wind rose for the study site during the sampling period.

Figure 4 shows the behavior of the concentrations of the metals measured in PM10 with respect to the prevailing winds during the study period. Fe showed higher concentration values when the wind blew from the West. Cd and Zn, showed higher levels when the winds blew from the South. Cu and Co concentrations were higher when the winds blew from the Southwest. Mn concentrations were higher
when the wind came from the East. Sources located in these directions could contribute to the trace metals measured in PM10 in the study site.

It is important to mention that the study site is located within the facilities of the Faculty of Medicine and next to the General Hospital of the city, which is why it is considered that vehicular activity is important in that area. Being an urban area, many gasoline service stations are located near the sampling site. All these sources could contribute to the levels of trace metals in PM10 particles during the study period.

**Figure 4.** Relation between the prevailing winds and the trace metals concentrations for (a) Fe, (b) Cd and Cu and (c) Zn, Co and Mn

### 3.4 Statistical Analysis

The results of the non-parametric Friedman test at a level of significance of $\alpha = 0.05$ showed that there were significant differences between Fe with Cu ($p$-value $= 0.047$) and Fe with Mn ($p$-value $= 0.009$) and between Co with Fe ($p$-value $<0.0001$) and Co with Zn ($p$-value $=0.039$), indicating that these metals had their origin in different sources. Pearson's analysis represented in Table 5 shows that there is a positive correlation between Cd, O$_3$, and relative humidity (RH), as well as negative correlations between Cd-Mn, and temperature (TEMP) -wind speed (WS). A negative correlation with wind speed...
may indicate that, at higher speeds, the concentrations of pollutants decrease due to the phenomenon of dispersion by the action of the wind. With respect to Cu, it can be observed that it had an important positive correlation with Co, which indicates that they could have sources in common. Cu was also significantly correlated with PM10, indicating that these pollutants could have originated from the same source. Table 5 shows that Mn positively correlates with Fe, suggesting that they were originated in common sources, since both are tracers of the cortex. Mn negatively correlated with RH indicating that manganese is a soluble element, therefore it can be inferred that a washout occurs in the column of the atmosphere, which causes Mn to decrease with increasing relative humidity. Fe positively correlated with SO2 and TEMP, which indicates that Fe may also have had anthropogenic sources of enrichment. Zn negatively correlated with SO2, CO, NO2 and PM10, indicating that these pollutants could be precursors of the Zn present in PM10 particles. SO2 correlated positively with NO2, PM10 and TEMP, indicating that these pollutants could have a strong influence from sources of combustion at high temperatures and sources of combustion that use highly sulfurized fuels such as diesel and fuel oil. CO significantly correlated positively with NO2, HR and WD, indicating that these pollutants had their origin in vehicular sources and could be transported from nearby regions. NO2 showed significant positive correlations with PM10 and WD, indicating that they had sources in common at the study site. From the analysis of principal components, it can be observed that two factors F1 and F2 were necessary to explain the 65.49% of the total variability of the data. Figure 5 shows the biplot including the two principal components F1 and F2. Factor F1 includes Cd, Mn, CO, HR, WS and WD (Table 6), which correlated with each other, indicating that they could have originated from common sources. CO is a tracer of vehicular combustion sources, for which Cd and Mn could be originated from vehicular emissions. The correlation with WS, WD, HR indicates that these pollutants were influenced by the transport of air masses from other areas and by the high moisture content. Factor F2 includes Fe, Zn, SO2, NO2, PM10 and TEMP (Table 6), which correlated with each other, indicating that they could have originated from common sources. NO2 is a tracer of vehicular combustion sources and sources of combustion at high temperatures, which indicates that Zn, Fe and PM10 were enriched due to the vehicular activity that exists around the area. SO2 is a tracer of industrial emissions from combustion sources that use highly sulphur fuels, so Zn and Fe could also have their origin in industrial sources. Factor F3 includes Co and O3, showing a significant correlation with each other, indicating that these pollutants were influenced by photochemical activity. Factor F4 (Table 6) included only Cu, indicating that this metal had its origin in a different source, possibly from the metal-mechanical industry and the assembly of metal parts.

|          | Cd    | Cu    | Mn    | Co    | Fe    | Zn    | O3    | SO2   | CO    | NO2   | PM10  | TEMP  | HR    | WS    | WD    |
|----------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| Cd       | 1.00  | -0.53 | -0.08 | -0.48 | 0.12  | 0.82  | -0.34 | 0.08  | -0.03 | -0.09 | -0.59 | 0.58  | -0.55 | 0.419 |
| Cu       | 0.11  | 1.00  | -0.37 | 0.82  | -0.09 | -0.43 | -0.14 | 0.42  | 0.25  | 0.42  | 0.53  | 0.07  | -0.01 | -0.17 | 0.436 |
| Mn       | -0.53 | -0.37 | 1.00  | -0.30 | 0.54  | 0.28  | -0.06 | 0.23  | -0.60 | -0.49 | -0.07 | 0.72  | -0.75 | 0.46  | -0.283 |
| Co       | -0.08 | 0.82  | -0.30 | 1.00  | -0.16 | -0.18 | -0.32 | 0.02  | 0.05  | 0.12  | 0.21  | -0.03 | -0.10 | -0.24 | 0.085 |
| Fe       | -0.48 | -0.09 | 0.54  | -0.16 | 1.00  | -0.49 | -0.12 | 0.58  | -0.17 | 0.07  | 0.14  | 0.59  | -0.19 | 0.43  | 0.219 |
| Zn       | 0.12  | -0.43 | 0.28  | -0.18 | -0.49 | 1.00  | 0.32  | -0.70 | -0.51 | -0.79 | -0.54 | -0.29 | -0.18 | -0.39 | -0.490 |
| O3       | 0.82  | -0.14 | -0.06 | -0.32 | -0.12 | 0.32  | 1.00  | -0.27 | -0.17 | -0.32 | -0.15 | -0.43 | 0.45  | -0.58 | 0.459 |
| SO2      | -0.34 | 0.42  | 0.23  | 0.02  | 0.58  | -0.70 | -0.27 | 1.00  | 0.34  | 0.63  | 0.78  | 0.66  | -0.24 | 0.48  | 0.491 |
| CO       | 0.08  | 0.25  | -0.60 | 0.05  | -0.17 | -0.51 | -0.17 | 0.34  | 1.00  | 0.90  | 0.70  | -0.33 | 0.56  | -0.17 | 0.572 |
| NO2      | -0.03 | 0.42  | -0.49 | 0.12  | 0.07  | -0.79 | -0.32 | 0.63  | 0.90  | 1.00  | 0.78  | 0.004 | 0.36  | 0.13  | 0.583 |

Table 5. Pearson Correlation Matrix for Cd, Cu, Mn, Zn, Fe, Co, criteria air pollutants (O3, SO2, CO, NO2 and PM10) and meteorological parameters (Temperature, Relative Humidity, Wind speed and Wind direction).
PM10  -0.09  0.53 -0.07  0.21  0.14  -0.54 -0.15  0.78  0.70  0.78  1  0.24  0.02  0.06  0.707
TEMP  -0.59  0.07  0.72 -0.03  0.59 -0.29 -0.43  0.66 -0.33  0.004  0.24  1  -0.83  0.84 -0.187
HR     0.58 -0.01 -0.75 -0.10 -0.19 -0.18  0.45 -0.24  0.56  0.36  0.02 -0.83  1 -0.65  0.545
WS    -0.55 -0.17  0.46 -0.24  0.43 -0.39 -0.58  0.48 -0.17  0.13  0.06  0.84 -0.65  1 -0.397
WD     0.41  0.43 -0.28  0.08  0.21 -0.49  0.45  0.49  0.57  0.58  0.70 -0.18  0.54 -0.39  1

Values in bold are different from zero at a significant level of $\alpha=0.05$

**Figura 5.** Biplot resulting of the multivariate statistical analysis applied to the measured variables, showing the two principal components: F1 and F2.

**Table 6.** Principal Component Analysis for Cd, Cu, Mn, Co, Fe, Zn, criteria pollutants and meteorological parameters.

|       | F1   | F2   | F3   | F4   |
|-------|------|------|------|------|
| Cd    | 0.364| 0.280| 0.056| 0.070|
| Cu    | 0.216| 0.190| 0.265| 0.294|
| Mn    | 0.692| 0.029| 0.106| 0.077|
| Co    | 0.061| 0.038| 0.632| 0.191|
| Fe    | 0.116| 0.313| 0.243| 0.024|
| Zn    | 0.126| 0.625| 0.008| 0.014|
| O₃    | 0.105| 0.322| 0.345| 0.210|
| SO₂   | 0.003| 0.857| 0.059| 0.029|
| CO    | 0.580| 0.157| 0.000| 0.186|
| NO₂   | 0.402| 0.487| 0.000| 0.102|
| PM10  | 0.214| 0.559| 0.005| 0.022|
| TEMP  | 0.443| 0.464| 0.006| 0.043|
| HR    | 0.683| 0.096| 0.067| 0.059|
| WS    | 0.386| 0.354| 0.003| 0.084|
| WD    | 0.555| 0.107| 0.185| 0.130|

Values in bold correspond for each variable to the factor for which the cosine squared is the highest
3.5. Morphological characterization of PM10 and SEM-EDS Analysis

The properties of the particles such as size, density and surface can have an important influence on their behavior in the air and their effects on the environment and health [47]. On the other hand, the properties of the particles (shape, size and texture) are also indicative to determine the origin of the particle, that is, if it is the result of natural or anthropogenic emissions. The particles that come from a natural source tend to have a different structure and composition than those emitted by anthropogenic sources [48]. The individual particles analyzed in this work present different morphologies, which include irregular, amorphous, spherical and groups of small particles that form larger particles (clusters / agglomerates). For this study, around 75 particles were analyzed, for practical purposes only the 3 most representative ones are shown. Figure 6 shows the images of the SEM-EDS analysis of said particles and Table 7 shows the elemental content of these selected particles. Figure 6a shows a point particle with an irregular shape with a shiny appearance identified as 1705 which has a high content of Pb, Si and O, in a lesser proportion it has C, Na, Cd, Al, Ca and Mg. The mass percentages found are shown in Table 7. The particle with a regular spherical shape with a shiny appearance identified as 1736 shown in figure 6b, is a particle rich in Pb and O, to a lesser extent it is composed of C, Si, Na, Al and Ca. In figure 6c, two particles identified as 1745 and 1746 are observed, both with a regular spherical shape with a shiny appearance, which appear to be adhered one on top of the other. Both particles are rich in Cu and O, and to a lesser extent are composed of Si, C, S and Al. According to Aragon Piña (2011) [47] anthropogenic particles are those that contain heavy elements in their chemical composition, such as Pb, Cu, Cd, Mn, Zn, Ni and As; among others. They commonly present irregular shapes (Figure 6a), spherical (Figure 6b), and also in complexes of agglomerates of fine particles that can be globular or acicular (Figure 6c) and above all, they generally present a highly variable chemical composition. This type of particles is associated as a result of activities such as the burning of fuels, industrial processes, agriculture, mining, etc. It should be noted that most of the material that constitutes the earth's crust are silicates, and contain elements such as Si, Al, Ca, Mg, Fe and O, so it is not surprising that these elements are part of the composition of most of the particles presented in this study. Several particles containing lead were found from the SEM-EDS analysis, as shown by the morphology of particles 1745 and 1746 shown in Figure 6. The presence in the air of these lead-rich particles is related to emissions generated from the metallurgical industry (non-ferrous materials) and dense vehicular traffic [47]. Short-term exposure to lead can cause brain damage, kidney damage, and gastrointestinal problems [28]. In Mexico, the air quality standard NOM-026-SSA1-1993 [48] indicates that lead levels in ambient air must not exceed the exposure limit of 1.5 µg m⁻³ within an arithmetic average of three months.

Table 7. Elemental analysis results: mass percentage of metals contained in the selected PM10 particles

| PM10 Particle identification Code | 1705 | 1736 | 1745 | 1746 |
|----------------------------------|------|------|------|------|
| Al                               | 0.64 | 0.29 | 0.11 | 0.13 |
| C                                | 1.24 | 6.52 | 9.75 | 8.64 |
| Ca                               | 0.27 | 0.25 |      |      |
| Cd                               | 0.66 |      |      |      |
| Cu                               |      |      | 30.3 | 31.8 |
| Mg                               | 0.22 |      |      |      |
| Na                               | 0.74 | 0.48 |      |      |
| O                                | 22.52| 25.7 | 33.3 | 33.2 |
| Pb                               | 47.65| 60.9 |      |      |

Metals (mass percentage)
|  Element | Value 1 | Value 2 | Value 3 | Value 4 |
|----------|---------|---------|---------|---------|
| S        | 8.68    | 8.3     |         |         |
| Si       | 26.07   | 5.8     | 17.7    | 17.8    |
| **Total**| 100     | 100     | 100     | 100     |

**Figure 6.** SEM-EDS Analysis of PM10 selected particles identified as 1705 (a), 1736 (b), 1745 and 1746 (c)
3.6. Health Risk Analysis

For the health risk analysis, two sectors of the exposed population were considered: adults and children. The carcinogenic risk (CR) was determined for the metals Cadmium and Cobalt, which have been reported to have carcinogenic effects. Table 8 shows the results of carcinogenic risk obtained in this study and their comparison with that reported by other authors, it can be observed that the limit value established by the EPA of $10^{-6}$ was not exceeded, indicating that the risks of cancer by inhalation of Cd and Co in the study is low. Cancer risk coefficients (CR) in adults are higher than those obtained in children, this because the extramural exposure of adults is greater than in the child population, due to mobility related to their work and occupations. According to Table 8, the CR value calculated for Cd in adults and children in the city of Torino, Italy [49] exceeds the established permissible limits. Comparing the CR values found in this study with other studies around the world (Table 8), it can be observed that CR values for Cd were lower than those reported for Torino, Italy [49], Gijón, Spain [50] and Acerra, Italy [52]. The CR values found for Co in the study site were lower than those reported for Torino [49] and Acerra [52] cities in Italy. Table 9 shows the results obtained for non-carcinogenic risk (HQ) for Cd, Co and Mn. These values must be <1 in order not to represent a health risk. During the present study, no value above that established was observed, which suggests that the risk of contracting diseases other than cancer (respiratory and cardiovascular diseases) in the exposed population is low. According to Table 9, the values observed in the city of Torino, Italy for the case of Cd in exposure to the child population exceed 6 times the legal limit and 1.69 times in exposure for the adult population. Comparing these results with those obtained in the present study, the value calculated for the city in Italy is 12.93 times higher than the value registered in children in the present study and 3.56 times higher in adults. It is worth mentioning that the results obtained for exposure to children are higher than those obtained for exposure to adults, this because the child population may be more sensitive to non-carcinogenic substances than adults [49].

HQ values for Cd in the study site were higher than those reported in Acerra, Italy [52], and lower than those reported for Torino, Italy [49], Gijón, Spain [50], Baoutou, China [51] and Kuopio, Finland [53]. Co showed HQ values in this study higher than those reported in Acerra, Italy [52], Athens, Greece [53] and Kuopio, Finland [53] and lower than those reported for Torino, Italy [49], Gijón, Spain [50], Baoutou, China [51] and Lisbon, Portugal [53]. The HQ values for manganese in this study were similar than those reported for Torino, Italy [49] and Kuopio, Finland [53], but lower than those reported in Acerra, Italy [52], Gijón, Spain [50], Baoutou, China [51] and Athens, Greece [53]. The integrated HQ values (the sum of the three metals considered) in the present study were less than 1 for adults and children. This suggests that exposure to these metals means a low risk of suffering or aggravating respiratory and cardiovascular diseases in the study site.

Table 8. Carcinogenic risk evaluated in the study area and its comparison with two cities in Europe.

| City              | Trace Metal | Adult  | Children |
|-------------------|-------------|--------|----------|
| Gijon, Spain (Urban) [50] | Cd          | 5.44E-07 | 1.36E-07 |
| Torino, Italy (Urban) [49] | Cd          | 1.00E-05 | 9.46E-06 |
| Acerra, Italy (Urban) [52] | Co          | 8.00E-08 | 7.00E-08 |
| Leon              | Cd          | 2.54E-06 | 6.35E-07 |
| Guanajuato, Mexico (This Study) | Co         | 3.47E-07 | 8.67E-08 |
Table 9. Non-cancer risk coefficients evaluated in the study area, and its comparison with two cities in Europe and one city in Asia.

| City                               | Metal | Adult | Children |
|------------------------------------|-------|-------|----------|
| Baoutou, China (Urban) [51]        | Cd    | 0.2900| 0.7800   |
|                                    | Co    | 0.0500| 0.1800   |
|                                    | Mn    | 0.9000| 0.9600   |
| Gijon, Spain (Urban) [50]          | Cd    | 0.0881| 0.0881   |
|                                    | Co    | 0.0272| 0.0272   |
|                                    | Mn    | 0.5060| 0.5060   |
| Torino, Italy (Urban) [49]         | Cd    | 1.6900| 6.1300   |
|                                    | Co    | 0.0050| 0.0100   |
|                                    | Mn    | 0.0300| 0.1000   |
| Acerra, Italy (Urban) [52]         | Cd    | 0.1400| 0.4100   |
|                                    | Co    | 0.1400| 0.400    |
|                                    | Mn    | 0.2900| 0.8400   |
| Athens, Greece (Urban) [53]        | Cd    | -     | -        |
|                                    | Co    | 0.4510| 0.4510   |
|                                    | Mn    | 0.2300| 0.2300   |
| Kuopio, Finland (Urban) [53]       | Cd    | 0.0050| 0.0050   |
|                                    | Co    | 0.0118| 0.0118   |
|                                    | Mn    | 0.0643| 0.0643   |
| Lisbon, Portugal (Urban) [53]      | Cd    | -     | -        |
|                                    | Co    | 0.0855| 0.0855   |
|                                    | Mn    | -     | -        |
| Leon, Guanajuato, Mexico (This study) (Urban) | Cd | 0.4740 | 0.4740 |
|                                    | Co    | 0.2850| 0.2840   |
|                                    | Mn    | 0.0560| 0.0550   |

4. Conclusion
The concentrations of PM10 particles did not exceed the maximum permissible limits established by the Mexican standard (75 µg m\(^{-3}\)) or those established by the WHO (50 µg m\(^{-3}\)). The studies carried out on the collected samples showed that of the six metals that were analyzed, Fe and Zn were the ones with the highest concentrations, which was to be expected due to their higher proportion compared to the other metals due to their geological origin. It should be noted that the concentrations found for Cu in the present study were higher than in other studies carried out in Mexico, although the values were similar to those found in cities such as Bogotá, Colombia and Agra, India. The results obtained for Cd showed that the concentrations were higher in the study area than in other studies carried out in Mexico and similar in studies carried out abroad. Regarding Mn, it was found that the results were lower than those reported in other studies carried out in Tampico, Tamaulipas and in the same city of León, higher than those found in Hidalgo, Colima and Puebla and cities outside the country such as Seoul, Korea and Venice Italy. The concentrations of Co that were obtained were higher than those registered in other studies carried out in Mexico such as Puebla and the Metropolitan Area of Mexico City, and Venice, Italy, however, they were lower than those obtained in Agra, India and in the simultaneous study carried out in the same city (León, Guanajuato). The enrichment of the particles with heavy metals could be attributed to emission sources located to the Southwest of the study site, in this direction there are two main avenues, on which a significant percentage of vehicles travel daily, in addition there are leather tanneries and gas stations, which may have acted as sources
contributing to the levels of the metals measured in PM10. It is worth mentioning that around the city of Leon there are important mining and industrial parks, therefore its contribution to the enrichment of the particles with heavy metals is not ruled out. In the statistical analyses carried out, significant correlations were found with the speed and direction of the wind, indicating the influence of the transport of pollutants from nearby local areas. Cu and Co showed a significant positive correlation, which indicates that both could have sources in common, this is corroborated with the wind roses and pollutants carried out for the present study, which shows that both metals had a higher concentration when the wind blew from the SW. Mn negatively correlated with RH, this could be due to the fact that manganese is a soluble element, therefore it can be inferred that a washing of the column of the atmosphere occurs at higher humidity values. In the analysis of principal components, the correlation of CO with Mn and Cd stands out, since CO is a tracer of vehicular combustion sources, it is inferred that Cd and Mn could be originated in vehicular emissions and combustion sources at high temperatures. In the morphological analyses, the presence of metals such as Al, Cd, Pb, Cu, among others, could be observed, finding particles with irregular shapes and regular shiny spherical associated with anthropogenic sources. Regarding the estimation of the risks to the health of the population, the cancer risk index in the lifetime (CR) did not exceed the value of $10^{-6}$ established by the US-EPA nor the value of $10^{-5}$ established by the WHO, so the concentrations of heavy metals in PM10 measured at the study site do not represent a carcinogenic risk to the health of the exposed population. Regarding the non-carcinogenic risk during the present study, no value above that established was observed, indicating that the risk of contracting diseases other than cancer (respiratory and cardiovascular diseases) by inhalation of PM10, containing metals, at the study site is low.

As future work, it is recommended to consider the concept of bio accessibility of metals to carry out a comprehensive assessment of health risks, since the total values of CR and HQ may overestimate the dangers they pose to humans. It is also necessary to design a bio monitoring plan in the future in the city of Leon, also considering other nearby cities such as Celaya and Salamanca (in the latter there is an oil refinery and a thermoelectric plant). In this study, only some metals in PM10 particles and the associated risk due to the exposure of the population to the concentration levels found were considered, however, it is necessary to include in the future in the evaluation of health risk, other toxic metals such as Pb, Cr, Al, Si, as well as organic pollutants (dioxins, PCBs, among others) to evaluate a broader matrix of pollutants that may represent an inhalation risk in the study population.

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