

# Characterization of airborne particles collected in Duhok city (in Iraq), by using various techniques

B H Mahdi<sup>1</sup>, K M Yousif<sup>2</sup> and L M Salih Dosky<sup>3</sup>

<sup>1</sup>Branch of Basic Sciences, College of Agriculture, Duhok University, Duhok, Iraq.

<sup>2</sup>Departement of Environmental Sciences, College of Sciences, Zakho University, Zakho, Iraq.

<sup>3</sup>Gihan University, Duhok, Iraq.

E-mail: Berivan.hadi@uod.ac, kamy22002@yahoo.co.uk, luqman.kader@uod.ac

**Abstract.** The contamination level of metals in the dust of Duhok (DK) city in Northern Iraq (an urban area) was investigated. Particulate matter (PM) accumulated on Funnel container was examined in order to investigate the air that people are exposed to. The morphology, microstructure, and chemical composition of a variety of particles were studied using Reflected light microscope (RLM), Atomic Absorption spectrometer (AAS), Scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX). The particles exhibited different morphologies and composition, indicating mostly a soil origin. The concentration of the following heavy metals (Fe), (Mn), (Cu), (Pb), (Zn), (Cd), (Ni) and (Co) are investigated by (AAS). The average values of concentration of most of the heavy metals in summer season were higher than other seasons. Amongst individual particles of tested dust by EDX were Si, Fe, Ca, Al, Mg, Ti, and K. And the predominant elements are Si (16.79 Wt%), Fe (10.68 Wt%), Ca (10.67 Wt%) and Al (5.99 Wt%).

**Keywords:** Dust, Particle, Atomic Absorption, Scanning electron microscopy, energy-dispersive X-ray

## 1. Introduction

The composition of air, which is a mixture of solid particles, liquids, and gases, is important in determining life quality. Air pollution and its impacts have become one of the most important challenges for public health and environmental quality [1]. Air pollution is a growing problem generated mainly by industrial and vehicular emissions. Several chemicals are emitted into the air from natural sources, forest fires, and anthropogenic (or man-made sources, such as vehicle emissions, industrial discharges, residential fossil fuel burning, waste incineration, power generation facilities, construction and demolition activities, etc.) [2,3]. Particulate matter (PM) is one of the major atmospheric pollutants. PM in the atmosphere often contain high levels of toxic metals and organic contaminants [3]. These can be affecting the atmospheric environmental quality [4] as well as the weather. Particles with diameters between PM<sub>10</sub> and PM<sub>2.5</sub> are defined as the coarse fraction particles [5]. PM<sub>2.5</sub> includes particles with diameters less than 2.5 $\mu$ m, also known as fine particles. These fine particles are able to penetrate deep into the human respiratory system and then absorbed into the blood. Also these PM can scatter light and therefore play a major role in visibility impairment [6]. Road vehicles emissions are considered one of the main sources of heavy metal (HVM) contamination in DK city environment [7]. HVM produced from vehicular exhaust, tire and brake abrasion can be deposited as road dust by dry or wet atmospheric deposition [8, 9]. HVM pollution is



potentially a persistent problem all over the world [10-13]. Emissions from traffic contain many toxic HVM such as Pb, Cd and Zn [14]. Exhaust gases and fossil fuel combustion have been claimed as sources of Ni and Zn [15-17], while Fe and Mn have been traced to steel industrial processes and the Earth's crust [16,17]. Therefore, numerous studies were carried for determination of concentrations (CONC) of HVM in the environment, because it threatens human health and the environment [18, 19].

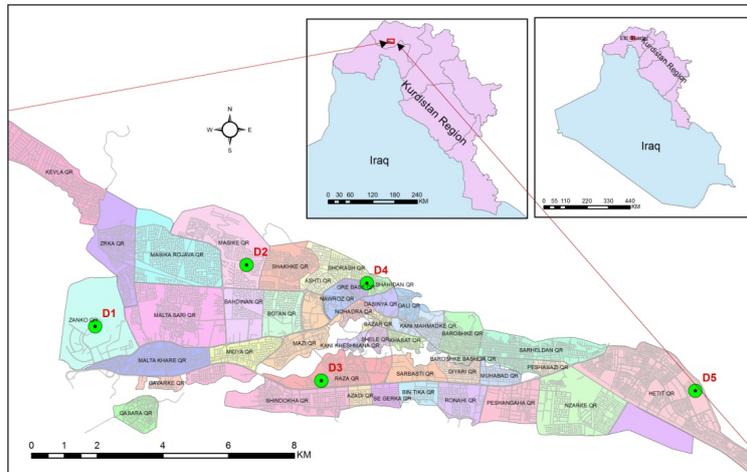
To the best of our knowledge, only a few studies were conducted to detect the level of HVM in dust for Kurdistan region, Iraq, [7, 20, 21].

The main aims and objectives of this study are to determine the concentration of heavy metals (Fe, Mn, Cu, Pb, Zn, Cd, Ni and Co) in dust in Duhok (DK) city, Iraq, in various locations and some seasons of 2017-2018. Also to study the morphology, microstructure, and chemical composition of particles present in the dust samples in DK city, by using Reflected light microscope (RLM), Atomic Absorption spectrometer (AAS), Scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX), along with the Statistical analysis of results obtained.

## 2. Materials and methods

### 2.1. Study area

The study was conducted in Duhok (DK) city located in the northwest part, Iraq, at latitude ( $36^{\circ}53'20'' - 36^{\circ}50'N$ ) and longitude ( $42^{\circ}54'10'' - 43^{\circ}5'$ ) [see figure 1], it surrounded from three direction by mountains, the white (Bekhair) mountain in the north and northeast, Shindokha (Zawa) mountain in the south and southeast and Mamseen mountain in the east. The seasonal average temperatures for 2017 were  $32.9^{\circ}C$  for summer,  $21.6^{\circ}C$  for autumn, and  $10.1^{\circ}C$  for winter. The area is characterized by semiarid climate with dry, hot summers and cold winters.



**Figure 1.** Duhok city map showing location of the study area.

### 2.2. Atmospheric dust sampling

The sampling was carried out during the period ranging from June 2017 to February 2018, for three seasons, Summer (SU), Autumn (AU) and Winter (Wi) seasons. A total of 15 atmospheric dust samples from five locations were processed and analyzed in the urban area of DK city, Iraq (figure 1). The sampling sites were selected based on the following criteria. They were not shaded by any obstacles such as buildings or trees. The dust collectors [Funnel container of neck-nozzle] were installed on the roof of buildings about (7–8 m) above the ground. The funnel of top squared area ( $50 \times 50 \text{ cm}^2$ ), covered with mesh on top to form a rough area for trapping to avoid animal stand. Dust samples were collected carefully and then transported to the laboratory. The samples were then placed

in sterilized plastic bags and transport to the laboratory. Dust samples were dried by oven for 24 hours at temperature (T)= 105 °C and then their weights were recorded.

### 2.3. Chemical analysis by atomic absorption spectrometer (AAS)

An accurately weighed (0.5 g) of dust after dried it and sieved it through a (0.5mm) mesh was placed in a test tube to digested it the test tube content 10 ml of a 3:1 concentrated HCl/HNO<sub>3</sub> the mixture was added to each test tube, and at room temperature the mixture was left overnight. Each test tube was covered with an air condenser and refluxed gently at (80 °C) for two hour. After cooling, the digested sample was filtered through a moistened Whatman filter paper and diluted to (50 ml) volume with distilled water [22-24]. The final solutions were analyzed for their Fe, Mn, Cu, Pb, Zn, Cd, Co and Ni CONC using an Atomic Absorption spectrometer (AA240FS) (VARIAN model).

### 2.4. SEM and EDX microanalysis

Particle size and elemental composition of PM were obtained through scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX) analysis, respectively. The SEM of the related samples was conducted, using SEM system model Nova Nanosem -450). The images of the samples were taken at a magnification of range 500×-100,000×. Spectra of particles were obtained after scanning an electron beam with an accelerating voltage of 10 kV for determination of elemental chemical composition of the particles. The Working Distance was 4mm. The SEM system was equipped with energy dispersive X-ray spectroscopy (EDX). The EDX microanalysis was carried out for elemental analysis using the line scan analysis technique, and the present elements were both qualitative and quantitatively measured for dust sample. The principle of EDX measurements is based on the detection of the generated X-ray.

### 2.5. Statistical analysis

Descriptive statistics including minimum (Min), maximum (Max), mean, median, standard deviation (SD), and coefficient of variation (COV) were performed using SPSS package version (16.0).

## 3. Results and Discussion

### 3.1 light microscope and Atomic Absorption

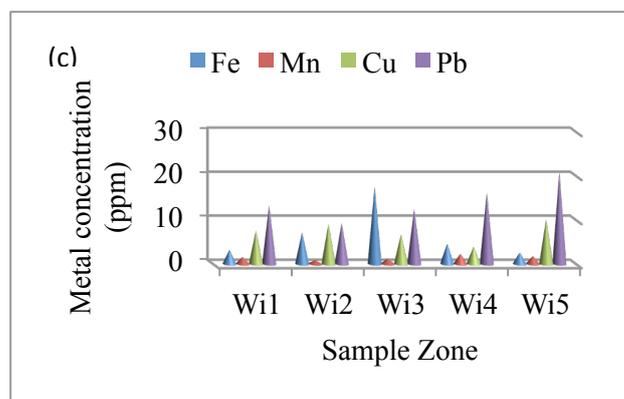
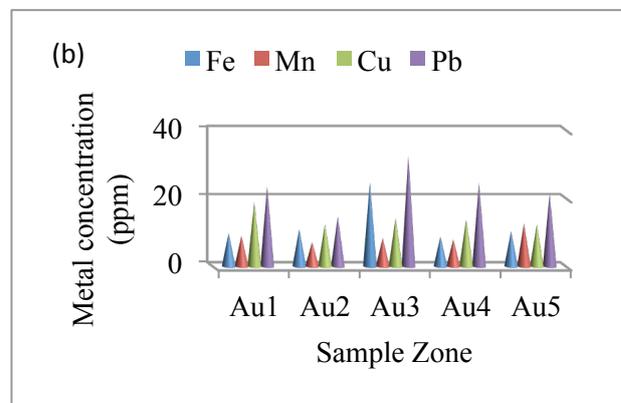
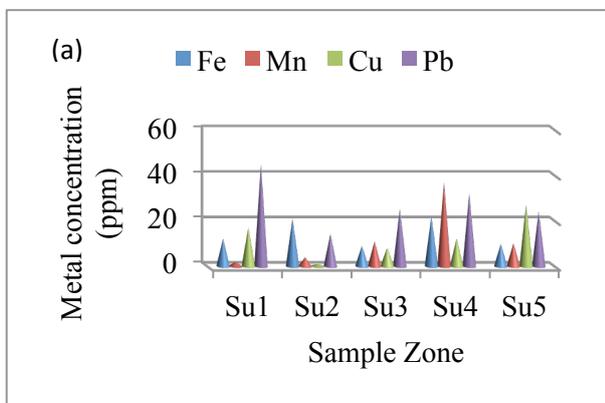
The dust retained has a heterogeneous composition due to its varied origin, transport, and emissions. The dust particles varied greatly with respect to size, morphologies, and chemical composition. Particles trapped in the 200 µm mesh were represented mainly by large biological material that was visible to the naked eye. Small insects, such as, wasps, legs, flies, small ant, and wings, as well as plant debris, fragments of wood, leaves and seeds was observed by light microscope. Inorganic particles and synthetic fibers were also found. We believe that the source of fibers may be from washed-carpets which normally dried over roofs of houses in DK city.

The concentrations (CONC) of HVM (Fe, Mn, Cu, Pb, Zn, Cd, Ni and Co) obtained by using AAS are given in table 1. The Zn and Cd detected only in samples (Su1 and Su4) and not detect (N.D) in other samples, also Ni and Co not detect, which indicate that there is a very low CONC of these elements, so they cannot be detect. The variation of (Fe, Mn, Cu and Pb) CONC for three seasons for all location is shown in figure 2.

Table 1 and figure 2 indicated that the CONC of Pb approximately high in all location, The main source of the Pb pollution in the dust of the study area may be related to the motor vehicles burning leaded gasoline (that contains tetraethyl lead as an anti-knock agent) [12, 25]. Mn CONC is lower than other HVM CONC in autumn and winter. Previous studies indicated that road traffic can be contribute to airborne metals through different pathways, e.g. combustion products from fuel, wear products from tires, brake linings and road construction materials, and re-suspension of road dust [7, 26, 27]. As the number of vehicles in DK city is on the increase in the past decade [e.g. No. of vehicles in Duhok (DK) in 2002 was ~20000, and it was ~ 130277 vehicles, on 2010] [7, 28].

**Table 1.** Seasonally concentration (ppm) in atmospheric dust samples in Duhok city.

| Summer |      |      |      |      |     |     |     |     |                |
|--------|------|------|------|------|-----|-----|-----|-----|----------------|
| Sample | Fe   | Mn   | Cu   | Pb   | Zn  | Cd  | Ni  | Co  |                |
| Su1    | 12.2 | 2.1  | 17   | 45   | 0.9 | N.D | N.D | N.D | Pb>Cu>Fe>Mn>Zn |
| Su2    | 21   | 4    | 1.1  | 14.2 | N.D | N.D | N.D | N.D | Fe>Pb> Mn> Cu  |
| Su3    | 9    | 11   | 8.2  | 25.3 | N.D | N.D | N.D | N.D | Pb>Mn>Fe>Cu    |
| Su4    | 21.9 | 37   | 12.2 | 32   | N.D | 1.1 | N.D | N.D | Mn>Pb>Fe>Cu>Cd |
| Su5    | 9.9  | 10.2 | 27.1 | 24.2 | N.D | N.D | N.D | N.D | Cu>Pb>Mn>Fe    |
| Autumn |      |      |      |      |     |     |     |     |                |
| Au1    | 9.8  | 9    | 18.9 | 23.5 | N.D | N.D | N.D | N.D | Pb>Cu>Fe>Mn    |
| Au2    | 10.9 | 7.1  | 12.3 | 14.6 | N.D | N.D | N.D | N.D | Pb>Cu>Fe>Mn    |
| Au3    | 24.6 | 8.3  | 14.2 | 32.5 | N.D | N.D | N.D | N.D | Pb>Fe>Cu>Mn    |
| Au4    | 8.8  | 7.9  | 13.7 | 24.5 | N.D | N.D | N.D | N.D | Pb>Cu>Fe>Mn    |
| Au5    | 10.3 | 12.6 | 12.2 | 21.4 | N.D | N.D | N.D | N.D | Pb>Mn>Cu>Fe    |
| Winter |      |      |      |      |     |     |     |     |                |
| Wi1    | 3.2  | 1.6  | 7.6  | 13.4 | N.D | N.D | N.D | N.D | Pb>Cu>Fe>Mn    |
| Wi2    | 7.2  | 0.9  | 9.1  | 9.3  | N.D | N.D | N.D | N.D | Pb>Cu>Fe>Mn    |
| Wi3    | 17.6 | 1.3  | 6.7  | 12.5 | N.D | N.D | N.D | N.D | Fe>Pb>Cu> Mn   |
| Wi4    | 4.6  | 2.3  | 3.9  | 16.2 | N.D | N.D | N.D | N.D | Pb>Fe>Cu>Mn    |
| Wi5    | 2.6  | 1.8  | 10.2 | 21   | N.D | N.D | N.D | N.D | Pb>Cu>Fe>Mn    |



**Figure 2.** Variation of CONC for some HVM in all locations during three seasons (a) summer season, (b) autumn season and (c) winter season.

### 3.2. SEM and EDX

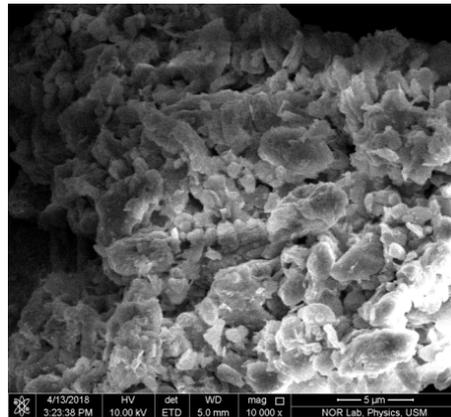
Morphological features of particles were observed. The size distribution analysis of particles is shown in figure 3. These particles were mostly of a large size with diameters ranging from 0.4-7.5  $\mu\text{m}$ . The average value of diameters was 2.7  $\mu\text{m}$ . Also, the particles display variable morphology. The shapes vary from rectangular to irregular, from spherical to oval-like aggregates. We believe that increasing in wind velocity can alter the erosion of soil structures and cause great changes in metal CONC in dust particles. Also rising of air temperature can cause dryness of clay, and an increase in breaking and modification of the van der Waals bonds exist in clay mineral structures and enhance the production of dust particles and their HVM contents [29, 30].

Figure 4 shows the qualitative EDX analysis for the dust collected from DK city. It reveals the existence of C, O, Si, Fe, Ca, Al, Mg, Ti, and K. Silicon and Iron were the most abundant elements found in large particles. Quantitative results of typical EDX analysis for the dust collected from DK city are listed in table 2. Since the soil in DK city is classified as a semi-desert soil mainly composed of silicates and clay minerals. So the dust collected has some silicates and clay minerals. However, the dust collected displayed a more complex composition. Most of the mineral particles observed had a diameter between (1-2.8  $\mu\text{m}$ ). SEM-EDX microphotograph data revealed different kinds of Aluminosilicates (e.g. Si+Al, clay or kaolinite), with irregular morphology, which were comprised of Al, Si, and O, along with other minerals of minor CONC such as Ca, Fe, Mg, K and Ti. The origin of these elements is mainly crustal, but they can also come from erosion of building products and road dust, etc. Other elements are present in dust samples of DK city, in minor concentration such as Pb, Mn and Cu, and were founded by using AAS, and not detect or found by using EDX, for unknown reasons. By detailed analysis of the particles it was noticed that some of them suffered from a nucleation or condensation, especially among the smaller size (2.5  $\mu\text{m}$ ), which shows as aggregates particles. (figures 5 and 6). These figures depict the typical microphotography of an aluminosilicate particles, with a diameters <8  $\mu\text{m}$ . SEM also show a large metallic particles found in dust. The corresponding EDX spectrum of the particles revealed that these particles contain iron (Fe), and iron oxides. We think that fine particles (PM < 1  $\mu\text{m}$ ) are linked to anthropogenic sources, or contributions from mechanical material abrasion. Sgrigna et al [31] reported that coarse particles (PM > 1  $\mu\text{m}$ ) are mostly generated by mechanical actions such as material abrasion and/or dust re-suspension, and are largely associated with anthropogenic activities. Based on the morphology and elemental composition study of the particles in dust of DK city, we can sorted these particles into 3 categories: 1-mineral particles (derived from soil sediments and weathered rock surfaces, e.g. silica particle, and aluminosilicate particle, i.e. clay or kaolinite), 2-metallic particles (derived from industrial activities, e.g. Fe), and, 3-biogenic particles (e.g. pollen derived from plants, which considered as potentially allergenic).

### 3.3. Statistical analysis

Descriptive statistics of heavy metal CONC in the DK city during three seasons are summarized in table 3. The average CONC of HVM during three seasons is shown in figure 7. The Pb CONC is higher than other heavy metals, and varied from (9.3-45) ppm with a higher mean value (28.14 ppm) observed in summer season (table 3 and figure 7). However, the mean CONC of lead for atmosphere dust samples was lower than that for dust samples reported by other literature, e.g in Erbil [20] and in Duhok [7]. The main source of the Pb pollution in the soils of the study area may be related to the motor vehicles burning leaded gasoline (that contains tetraethyl lead as an anti-knock agent) [25, 26]. The Cu CONC varied from (1.1-27.1) ppm with a higher mean value (14.26) ppm observed in autumn season. It can be released to the urban environment as a result of wear and corrosion of metallic parts of the automobiles [32,33]. Fe CONC ranged from (2.6-24.6)ppm with a higher mean value (14.8)ppm observed in summer season. The mean CONC of Fe for dust samples was lower than for dust samples reported by other literature [20]. The mean CONC of Fe for atmosphere dust samples was higher than for dust samples reported by Al-Khashman in Amman for street dust [32]. Also the mean CONC of

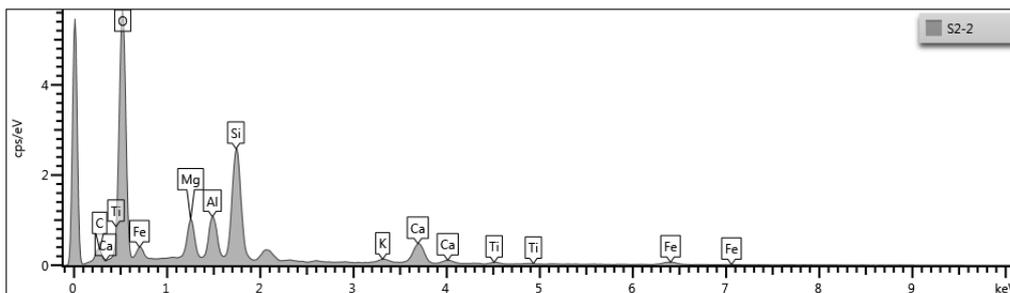
Mn for dust samples was lower than for dust samples reported by other literature [34]. Figure 7 shows that the CONC of all HVM in summer season is higher than other season, these higher CONC may be due to high speed of wind and dryness of soil due to low level of rainfall. In addition moderate degree of coefficient of variation (COV) for some HVM indicates no homogeneous levels [35, 36], while a large COV for HVM indication that the CONC of HVM differed greatly in the study area. In HVM the larger value of SD indicated the wide variation of CONC in atmosphere dust collected in DK city.



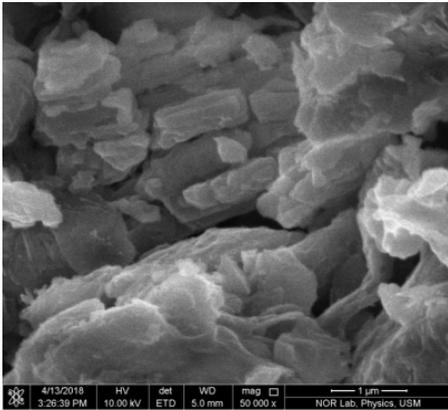
**Figure 3.** Photomicrograph of the scanning electron microscope (SEM) image of the dust particles.

**Table 2.** Quantitative EDX analysis results.

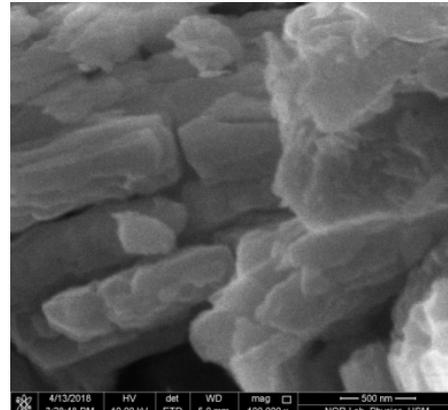
| Element | C    | O     | Mg   | Al   | Si    | K    | Ca    | Ti   | Fe    | Total |
|---------|------|-------|------|------|-------|------|-------|------|-------|-------|
| Wt %    | 4.08 | 43.68 | 4.42 | 5.99 | 16.79 | 1.34 | 10.67 | 2.34 | 10.68 | 100   |



**Figure 4.** Typical EDX Analysis for the dust collected from DK city.



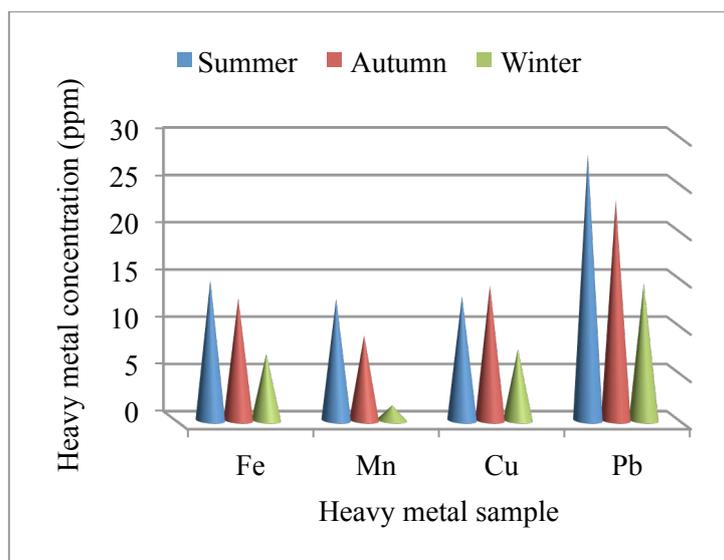
**Figure 5.** Photomicrograph of the scanning electron microscope (SEM) image of the alumino-silicate particle.(Lower Mag. X50k).



**Figure 6.** Photomicrograph of the scanning electron microscope (SEM) image of the alumino-silicate particle. (higher Mag. X100k)

**Table 3.** Descriptive statistics of HVM concentration (ppm) in the study areas during summer, autumn and winter seasons, in DK city.

| <b>Summer</b>                   |            |            |             |               |            |            |
|---------------------------------|------------|------------|-------------|---------------|------------|------------|
| <b>Metal</b>                    | <b>Min</b> | <b>Max</b> | <b>Mean</b> | <b>Median</b> | <b>S.D</b> | <b>COV</b> |
| <b>Fe</b>                       | 9          | 21.9       | 14.8        | 12.2          | 6.189911   | 0.418237   |
| <b>Mn</b>                       | 2.1        | 37         | 12.86       | 10.2          | 14.03168   | 1.09111    |
| <b>Cu</b>                       | 1.1        | 27.1       | 13.12       | 12.2          | 9.747153   | 0.742923   |
| <b>Pb</b>                       | 14.2       | 45         | 28.14       | 25.3          | 11.36961   | 0.404037   |
| <b>Pb&gt;Fe&gt;Cu&gt;Mn</b>     |            |            |             |               |            |            |
| <b>Autumn</b>                   |            |            |             |               |            |            |
| <b>Fe</b>                       | 8.8        | 24.6       | 12.88       | 10.3          | 6.596742   | 0.512169   |
| <b>Mn</b>                       | 7.1        | 12.6       | 8.98        | 8.3           | 2.137054   | 0.237979   |
| <b>Cu</b>                       | 12.2       | 18.9       | 14.26       | 13.7          | 2.735507   | 0.191831   |
| <b>Pb</b>                       | 14.6       | 32.5       | 23.3        | 23.5          | 6.430785   | 0.275999   |
| <b>Pb&gt;Cu&gt;Fe&gt;Mn</b>     |            |            |             |               |            |            |
| <b>Winter</b>                   |            |            |             |               |            |            |
| <b>Fe</b>                       | 2.6        | 17.6       | 7.04        | 4.6           | 6.163441   | 0.875489   |
| <b>Mn</b>                       | 0.9        | 2.3        | 1.58        | 1.6           | 0.526308   | 0.333106   |
| <b>Cu</b>                       | 3.9        | 10.2       | 7.5         | 7.6           | 2.421776   | 0.322903   |
| <b>Pb</b>                       | 9.3        | 21         | 14.48       | 13.4          | 4.398522   | 0.303765   |
| <b>Pb&gt; Cu &gt; Fe &gt;Mn</b> |            |            |             |               |            |            |



**Figure 7.** Average CONC of Fe, Mn, Cu and Pb during three seasons.

#### 4. Conclusions

This study demonstrates the potential of urban characterization of PM pollutants in distinct urban environments. The combination of AAS, SEM, and EDX techniques, together with the statistical analysis are valuable tools for the characterization of particles of dust tested at Duhok (DK) city, Iraq. The identification of the morphology and chemical composition of the dust particles provides valuable information for the determination and tracing of their origin. The results obtained show the presence of a wide diversity of particles from natural (e.g. crustal) and anthropogenic (fuel-oil combustion, agricultural fires, or industrial) origin.

The major sources of air pollution in DK city, are gaseous wastes in the form of vehicles exhaust, emissions from diesel generators, agricultural fires, and electric power plant (Kuashi) located in west side of DK city, as well as dust input from south and west of Iraq. In addition to the rapid increase in building density and high population density. The analysis of heavy metal (HVM) contamination, such as (Fe, Cu, Pb, and Mn) in dust of DK city, in various locations and some seasons of 2017-2018, by using AAS, along with the statistical analysis, indicated that heavy metal (HVM) contamination in DK city, increases according to the following trends:  $Pb > Fe > Cu > Mn$  during Summer season,  $Pb > Cu > Fe > Mn$  during Autumn season, and  $Pb > Cu > Fe > Mn$  during Winter season.

Amongst individual particles of tested dust by EDX were Si, Fe, Ca, Al, Mg, Ti, and K. And the predominant elements are Si (16.79 Wt%), Fe (10.68 Wt%), Ca (10.67 Wt%), and Al (5.99 Wt%).

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