



Chemical Characterization and Source Determination of Trace Elements in PM_{2.5} and PM₁₀ from an Urban Area, Northern Jordan

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Abstract: PM_{2.5} and PM₁₀ atmospheric particulate material samples were collected from Irbid city, Northern Jordan using a dichotomous air sampler. Collected samples were digested and analyzed for 17 elements (Al, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, P, Pb, S, V and Zn) using ICP-OES. Levels of measured elements were compared to concentrations in many other locations worldwide. Concentrations of most elements are in the lower limits of the reported values for urban and sub-urban sites. Elements of natural sources; Al, Ca, Fe, and P have higher concentrations compared to other cities worldwide. Results indicated that, elements of crustal origin are associated with the coarse particles, while elements of anthropogenic origins are more associated with fine particles. Crustal enrichment factor calculations showed that concentrations of Pb, Zn, Cd, Sb, and Ag are highly enriched and As, Cu, Co, Ca, and Ni are moderately enriched. Factor analysis calculations permitted the identification of three sources affecting the levels of elements in the fine fraction, namely re-suspended road dust, automobile emissions, and industrial emissions.

Keywords: PM₁₀, PM_{2.5}, ICP-OES, Trace Elements, Source Identification, Jordan

1. Introduction

Industrial expansion took place without much awareness of the environment and brought with it a rapid rise in pollution, chiefly particulate matter (PM). Therefore, an intensive effort has been spent to study the trace metals composition of the PM in the atmosphere. Previous studies have pointed toward the presence of a strong connection between the levels of fine particles in the ambient atmosphere and many health problems [1, 2]. Fine particles are easily inhaled deep into the lungs where they readily trapped for long periods of time [3]. Long-term epidemiological studies have revealed consistent associations between ambient concentrations of inhalable (PM₁₀) and respirable (PM_{2.5}) particle and increased risk of various adverse health outcomes, including lung diseases, premature death and cardiopulmonary mortality [1, 4]. High lead levels cause decreased vitamin D and haemoglobin synthesis,

anemia and acute central nervous system disorders. A study of blood lead levels of 389 children in Northern Jordan indicated that 30% of the children had lead levels above 10 µg/dL in their blood [5].

Elevated levels of heavy metals in the atmosphere can be caused by various local sources [6, 7] or by “long-range transported aerosols from other regions [8-10]”. Due to their small size and long residential time of several days to weeks in atmosphere, fine particles can travel for long distances. Analysis of PM in rural areas in conjunction with air mass back trajectory calculations indicated that, when the dust air mass passes over heavily industrialized zones, it can pick up anthropogenic air pollutants [11, 12]. For instant, Saharan dust that originate from North Africa desert areas are frequently transported to Jordan with high levels of crustal elements [10, 11]. Therefore, understanding of the size distribution of trace metals in atmospheric particles is important because it affects the toxicity of a metal when

inhaled and can be utilized in estimating the aerosol sources.

In Jordan, most of the environmental studies are related to soil pollution [13-17] with little attention to air pollution. The aim of this study was to quantify the levels of toxic metals in PM₁₀ and PM_{2.5} in Irbid, the second largest city in Jordan. Also, this study aimed to identify possible sources of heavy metals associated with the atmospheric aerosols using enrichment factors (EFs) and principal component analysis (PCA).

2. Experimental

2.1. Sampling Site

Irbid is the second largest city in Jordan with a population of about 1.4 million. The city is located about 80 km north of Amman. It is the highest population density in the kingdom. The location of Irbid is shown in Figure 1. Samples were taken at the roof of one of the buildings of Yarmouk University, which is located in the center of the city. The building height is about 20 meters above the surface of the earth. The sampler was installed in an open area and far away from the direct influence of any pollution source.

2.2. Sampling Procedure

PM₁₀ and PM_{2.5} air samples were collected daily (24-hour) using the Andersen dichotomous sampler (model-240). This sampler is a low-flow rate (16.7 L/min) sampler that divides the air stream passing the 10 μ m inlet into two portions that are filtered separately. The sampler cuts the 0 - to 10- μ m total sampler into 0 - to 2.5- μ m (fine) and 2.5 - to 10- μ m (coarse) fractions that are collected on separate 37-mm diameter Teflon filters.

2.3. Sample Digestion and Preparation

The PM₁₀ and PM_{2.5} aerosol samples and the NIST standard reference materials were digested in Teflon beakers with a 15 ml mixture of concentrated redistilled nitric acid, and hydrofluoric acid (10:5 v/v, respectively). The beakers were kept covered with a Teflon cover over night. Next day, samples were heated in a sand bath on a hot plate with a temperature at about 120 - 160°C for four hours with the covers on beakers, then the cover was taken off allowing for the solvent to start evaporation. Before complete dryness was reached, additional three ml of concentrated nitric acid was added and this was allowed to evaporate until the total volume of the solution was about one ml. The mixture was then diluted and brought into a final volume of 25 ml in a volumetric flask with deionized distilled water.

2.4. Analysis of Samples

Concentrations of 17 element including Al, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, P, Pb, S, V and Zn were measured in all samples using Varian VISTA-MPX instrument with CCD inductively coupled plasma- optical emission spectrometry (ICP-OES). The procedure

recommended by the manufacturer was followed during the analysis. Reagent and filter blanks were treated and analyzed using the same procedure as for the actual samples. Calibration curves were constructed by using commercial multi-element standard solution (100 ppm) (Sigma-Aldrich).

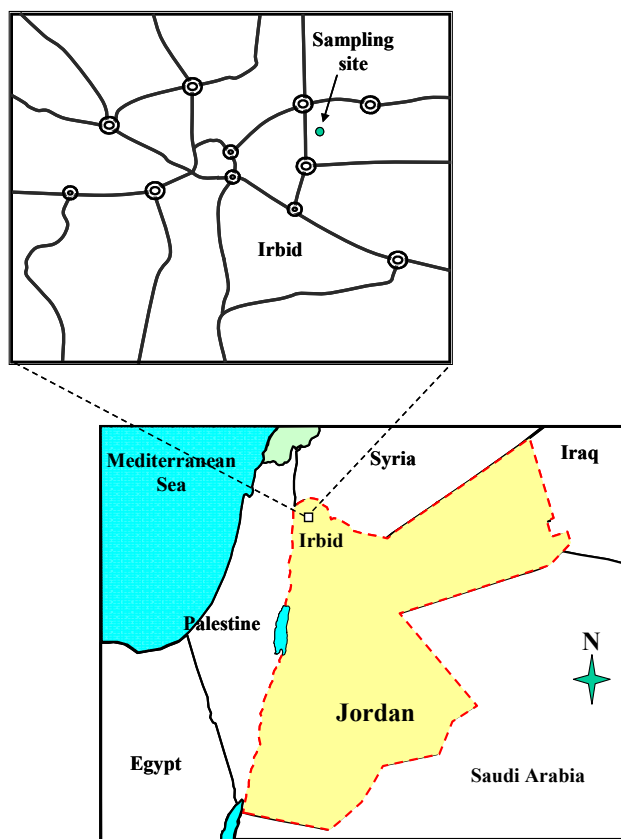


Figure 1. The location of the study site, Irbid.

2.5. Quality Assurance of Measurements

The accuracy of the analytical results were checked by periodic analysis of Standard Reference Materials (SRMs) obtained from the National Institute of Standards and Technology (NIST). Three SRMs were used; namely: SRM-1646A (Estuarine Sediments), SRM-1633B (Trace Elements in Coal Fly Ash), and SRM-2702 (Inorganics in Marine Sediments), and were treated in the same procedure as the samples and were analyzed along with the samples. Results were always in good agreement with the certified concentrations within 5% for most measured elements.

3. Results and Discussion

3.1. Ambient Concentrations of Elements

Statistical summaries for all measured species in both fine and coarse fractions of the collected aerosol samples are presented in Table 1. Concentrations of 17 elements; Al, Fe, Mn, Mg, Ca, Na, K, Cd, Co, Cr, Cu, Ni, Pb, P, S, V and Zn were higher than the detection limits in most samples. Average concentrations, geometric means, and standard deviations are given in this Table. As shown, the highest

average concentration in both fine and coarse fractions is for Ca, and is 3.3 and 6.3 $\mu\text{g}/\text{m}^3$, respectively. The average total concentration for Ca is 9.6 $\mu\text{g}/\text{m}^3$. However, the lowest concentration measured is for Cd, which was 2.0 and 1.4 ng/g for the fine and coarse fractions, respectively.

Table 1. Statistical summary of the metal concentrations in the fine and coarse fractions (ng/m^3).

Element	PM _{2.5}			PM ₁₀		
	Avg	SD	GM	Avg	SD	GM
Al	1475.0	1815.4	509.1	2267.9	2961.2	881.9
Ca	3309.4	3029.6	1951.3	6266.2	6605.4	3508.7
Cd	2.0	2.0	0.8	1.4	1.9	0.3
Co	3.5	3.8	2.1	2.6	3.3	0.5
Cr	16.0	19.8	4.8	15.1	27.0	3.0
Cu	21.6	20.6	9.7	19.2	24.8	2.8
Fe	1390.1	1887.2	665.8	2190.2	3325.8	926.8
K	675.7	668.3	424.3	1060.5	1541.9	494.7
Mg	726.0	831.3	386.7	1094.8	1164.5	651.2
Mn	41.4	41.7	24.9	49.3	64.0	28.4
Na	713.8	756.4	411.6	1190.7	1197.6	706.0
Ni	12.0	12.8	6.7	12.9	17.5	4.8
P	2749.2	2662.5	1757.4	6126.0	5949.7	3849.7
Pb	28.3	29.5	12.8	11.1	17.8	1.4
S	591.5	478.8	337.4	451.6	496.5	196.2
V	11.4	15.3	5.7	8.7	12.9	3.7
Zn	74.4	96.2	35.7	57.9	68.7	27.9

Comparing the data with data obtained from resembling

Table 2. Comparison of observed concentrations of elements in the PM_{2.5} and PM₁₀ fractions with literature values (ng/m^3).

	This Study		Kolkata, India ^[18]		Beren, Switzerland ^[19]		Algeria ^[20]		Hashimya, Jordan ^[6]	
	PM _{2.5}	PM ₁₀	PM _{2.5}	PM ₁₀	PM _{2.5}	PM ₁₀	PM _{2.5}	PM ₁₀	PM _{2.5}	PM ₁₀
Cd	0.8	0.3	5	8.6	0.2	0.26	18.7	21.2	2.83	1.6
Co	2.1	0.5	2.1				27.9	377	0.86	10.1
Cr	4.8	3	84	101					1.42	5.5
V	5.7	3.7	9.5		1.6	1.4			0.65	11.2
Ni	6.7	4.8	40	48	1.3	3	33.7	42.4	4.01	7.2
Cu	9.7	2.8	58	107	8.7	74	67.9	102.8	6.36	7.7
Pb	12.8	1.4	368	394	30	49	302.5	299.3	9.71	55
Mn	24.9	28.4	132	249	4.4	25	38	57.8	2	46
Zn	35.7	28	542	761					253.3	142.7
Mg	386.7	651	715		17	85	13		583	1060
Na	411.6	706	2047		96	665	84		233	725
K	424.3	495	2656		223	255	186		84	405
Al	509.1	882	2240		48	152	26		343	1211
Fe	665.8	927	3150	11242	124	2048	204	639.8	187	46
Ca	1951.3	3509	3351		54	1199	106		3514	15554

3.2. Metal Distribution in Different Size Fractions

Particulate air pollution is a mixture of solid, liquid or solid and liquid particles suspended in the air. These suspended particles vary in size, composition and origin. Anthropogenic sources involving high temperature processes, such as coal combustion and vehicle exhaust. Therefore, the smaller particles contain the secondarily formed aerosols (gas-to-particle conversion), combustion particles and recondensed organic and metal vapors [21]. Natural and anthropogenic mechanical processes, such as grinding, mining, construction, wear and tear of materials, and dust resuspension, can produce coarse particles [21]. Therefore, the larger particles usually contain earth crust materials and

areas may help in finding out the unusual results, which could be due to particular analytical problem. Observed concentrations of measured elements in the fine and coarse fractions are compared with those found by other researchers in urban areas and presented in Table 2. As shown, except for those reported for Kolkata (India) and Al-Hashimya (Jordan), levels of measured elements of crustal origin (Al, Ca, Mg, Na and K) in both fine and coarse fractions of Irbid atmosphere are higher in this study than those reported in Table 2. These high levels for crustal elements are generally attributed to the arid nature of the region, which is characterized by hot and dry weather most of the time. In addition, desert areas surround the region, therefore, the influence of Saharan dust, which is highly loaded by these elements must not be excluded. Moreover, the lack of significant plant cover around the city results in enhanced resuspension of soil and hence fairly high concentrations of elements associated with crustal material.

Levels of elements of anthropogenic origin varied in their levels with respect to those reported in Table 2. For instance, levels of Pb, Cd, Cu and Zn were close to those observed in Al-Hashimya (central Jordan) and Beren (Switzerland) and much lower than those observed for Algeria and Calcutta. Levels of the remaining elements (Cr, V, Ni, Mn, Co, ... etc.) fall between lower and upper limits of other sites.

fugitive dust from roads and industries.

Metals concentration ratios between the fine and the coarse sample fraction are calculated and presented in Figure 2. The figure reveals that, the fine-to-coarse ratios for Al, Ca, Fe, K, Mg, Mn, Na, Ni and P are less than unity. This means that concentrations of these elements in the fine fraction are quit lower than in the coarse fraction. Careful inspection of these elements indicates that, except for Ni, all of the remaining are of crustal origin. Theses results are in good agreement with the literature data, in which, natural sources derived elements are primarily associated with coarse aerosol particles [21, 22]. This observation is obvious in our results, for instant, the weight percent of Al, Ca, K, Na and P in the coarse fraction were 61%, 65%,

61%, 63% and 70%, respectively.

A morphological and compositional study of ambient aerosol in an industrial area of Italy [23] have found that compounds such as carbonates, chlorides and phosphates belong almost completely to the coarse fraction, while silicates and sulphates belong almost equally to both the fractions. Same conclusion have been reached by Rodriguez et al., [24], in which, phosphate emissions that are linked to crude oil refineries, phosphate-based fertilizer industry and power plants are associated with the coarse fraction. In this study, the weight percents of P and S in the coarse fraction are 70% and 43%, respectively.

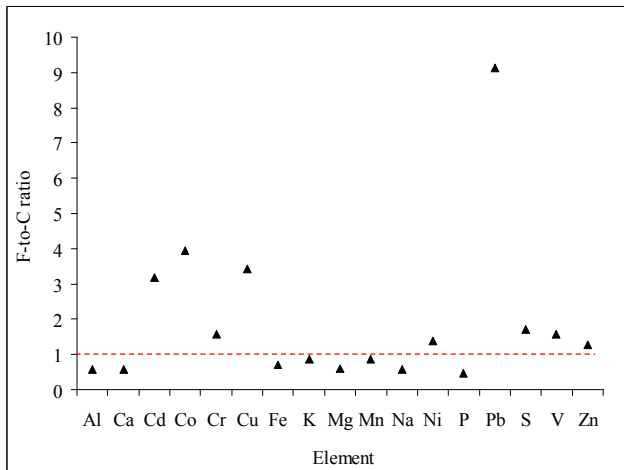


Figure 2. Fine-To-Coarse concentration ratio of elements.

On the other hand, anthropogenic elements (Cd, Co, Cr, Cu, Pb, S, V and Zn) were mainly associated with the fine size fraction, with a fine-to-coarse ratio of larger than nine for Pb and about one for Cr. Accordingly, the results reveal that, metals from anthropogenic sources are accumulated in the fine particles, while elements from natural sources are primarily associated with coarse particles. Furthermore, the weight percent calculations were in agreement with these findings. The anthropogenic elements such as Pb, Cd, Co, S, V and Zn are more concentrated in the fine fraction with a mass percent of (55 -75%).

3.3. Enrichment Factors of the Elements

The calculation of trace metal enrichment factors (EF) in airborne particles relative to crustal abundances has been used to evaluate anthropogenic versus natural sources. Calculations are generally based on the average upper continental crustal composition given by National Physical Laboratory. Each EF was calculated using equation (1), in which Al is used as a reference element [10, 18, 21]:

$$EF_X = \frac{(C_X / C_{Al})_{Air}}{(C_X / C_{Al})_{Crust}} \quad (1)$$

Where $(C_X/C_{Al})_{Air}$ and $(C_X/C_{Al})_{Crust}$ are the concentration ratios of element X to Al in airborne particles and the upper

continental crust, respectively. Generally, if EF is close to unity for any element X, this element may have a crustal source. Whereas, if the EF value is over 10, the element would have a significant contribution from non-crustal sources [22].

Figure (3) shows the crustal enrichment factors of Irbid aerosol samples (EFs), based on average concentrations of the elements identified in PM_{2.5} and PM₁₀. According to this Figure, measured elements can be classified into three different classes. The first class of elements includes the non-enriched elements (crustal elements; Al, Fe, Mg, Mn, K, V and Na). The EFs values for these elements are ranging from unity to 10. The second class of elements has enrichment factors between 10 and 100. These elements include; Co, Ni, Cr, Cu, and Zn. These elements are considered to be moderately enriched with respect to crustal composition, indicating that a large fraction of the element can be attributed to non-crustal or anthropogenic sources [6]. The last class of elements contains the highly enriched elements (S, P, Pb and Cd), with EF values greater than 100 indicating the presence of anthropogenic sources. These sources could be the high-temperature processes such as metal smelting, oil combustion and vehicular emissions.

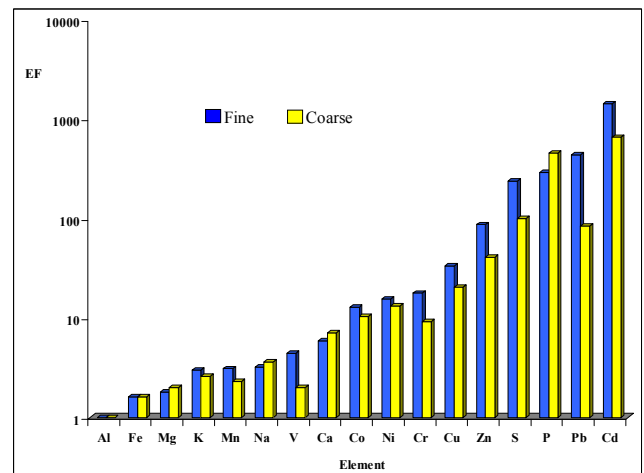


Figure 3. Average enrichment Factors of the elements in the PM_{2.5} (fine) and PM₁₀ (coarse) fractions.

Except for Na, Ca, Mg and P, the fine-to-coarse EF ratios of elements were higher than unity for all samples, indicating that most of the elements are more enriched in the fine fraction. These findings are coincided with literature results regarding the association of non crustal fraction of these elements with the fine particles in both urban and rural areas [21, 22]. The elements; Na, Ca, Mg and P are of crustal origin, and their association with the coarse fraction is expected. When the fine-to-coarse EF ratios were calculated on a seasonal base, different trend between winter and summer are observed. The fine-to-coarse EF ratio in winter for the elements K, Mg, Mn, Na, V and Zn are less than unity, indicating that they have higher enrichment factors in the PM₁₀ during the cold period than in the warm period. The increased enrichment of Na, K and Mg in the coarse

fraction during the winter time is attributed to the influence of the sea-salt spray [6, 10].

3.4. Source Apportionment

Principal component analysis (PCA) was performed to identify common sources of heavy metals in daily PM_{2.5} fraction. PCA was executed by the varimax rotation to improve the orthogonality of resolved factors using a statistical package Mintab-15. Principal components extractions were made by considering eigen values that are ≥ 1 . Three components were extracted accounting for 82% of the overall variance. PCA results of trace elements in PM_{2.5} are given in Table 3.

The first factor is highly loaded by Fe, V, Mn, Al, K, Zn and moderately loaded by Mg, Ni, Na, Ca and P. This factor includes some anthropogenic elements, such as Ni, V, Mn and Zn, indicating a mixture of crustal and anthropogenic sources. This Factor represents re-suspended soil dust. This factor explains 36% of the total system variance indicating the importance of the emissions of re-suspended road dust in fine fraction of the dust particles.

Table 3. Varimax rotated matrix for the fine fraction.

Variable	Factor1	Factor2	Factor3	Communality
Fe	0.92	0.1	0.33	0.97
V	0.89	0.26	0.13	0.87
Mn	0.88	0.35	0.19	0.93
Al	0.81	0.32	0.32	0.86
K	0.75	0.52	0.16	0.87
Zn	0.74	0.3	0.37	0.81
Mg	0.6	0.56	0.31	0.8
Na	0.59	0.56	-0.12	0.72
S	0.38	0.84	0.1	0.86
Co	0.04	0.72	0.34	0.63
Ca	0.5	0.71	0.2	0.81
Cu	0.39	0.65	0.43	0.83
P	0.51	0.55	0.3	0.65
Cd	0.07	0.2	0.8	0.68
Cr	0.44	0.14	0.76	0.79
Ni	0.51	0.36	0.69	0.86
Pb	0.1	0.97	0.06	0.97
Varian	6.15	5.08	2.68	13.91
% Var	36	30	16	82

The second factor is highly loaded by Pb, S, Ca, Co and moderately loaded by Cu and P. This factor explains 30% of the total variance representing emissions from the transport sector. Automobile emissions are heavy loaded with Pb, while diesel-powered vehicles emit sulfur. Jordan's Petroleum Refinery Company (JPRC) produces diesel with sulfur content of 9,000 ppm [25]. This level of sulfur is much higher than that produced by most other Middle Eastern countries. Emissions from high sulfur diesel fuel contribute to the observed high levels of S.

The third factor is loaded by Cd, Cr, Ni and moderately loaded by Zn and Cu. This factor explains 16% of the total system variance. This is possibly the industrial emissions component. These elements are usually emitted from different sources. Zinc is known to be originated from different sources, these include, coal combustion, smelting

operation and the wearing down of vehicle tires.

4. Conclusions

Based on the analysis of PM_{2.5} and PM₁₀ aerosol samples collected from an urban area in Jordan, concentrations of elements of crustal origins were predominantly contained in the coarse fraction (PM₁₀). However, the more elements (Cd, Co, Cr, Cu, Pb, S, V and Zn) which are usually emitted by anthropogenic sources are concentrated more in the breathable PM_{2.5} fraction. Enrichment factor calculations indicated that the elements Co, Ni, Cr, Cu, and Zn are moderately enriched with respect to crustal composition, while the elements S, P, Pb and Cd are highly enriched due to the contribution of various anthropogenic sources. The fine-to-coarse EF ratios of elements indicated that most of the elements are more enriched in the fine fraction. Three major sources affecting the observed levels of elements in the fine aerosol of the city were identified by principal component analysis, namely re-suspended road dust, automobile emissions, and industrial emissions.

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