

## APPLICATION OF NUCLEAR ANALYTICAL TECHNIQUES (XRF AND NAA) TO THE EVALUATION OF AIR QUALITY IN MOROCCAN CITIES - CASE OF MEKNES CITY

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**Abstract:** Meknes City is located in northern of central Morocco at the medium Atlas and it contains about 171 industrial units. The studied site is characterized by important road traffic and it is situated in the center of Meknes city, near a taxis station and not far from railway traffic. The sampling was carried out from March 2007 to November 2008. During this period, particulate matters (Fine and Coarse) were collected using, Gent, Partisol and Dichotomous samplers. The collected filter samples were analyzed using Total Reflection X-Ray Fluorescence and Neutron Activation Analysis. Besides, the sources of emissions of fine and coarse fractions were identified by means of enrichment factor analysis. The distribution of element contents between fine and coarse fractions showed that most of the analyzed elements are equally distributed on both fractions; except for some elements like Ca that was more distributed in coarse fractions and Ni that was more concentrated in fine fractions. These results were supported by the calculation of the ratios (Fine/Coarse) of each element for the four seasons. Enrichment factor analysis revealed that Fe and K came mainly from natural sources; Ca, Mn and Ni came mainly from natural sources but we cannot rule out the possibility of contamination from anthropogenic sources; Cr, Cu, Zn and Pb were attributed mainly to anthropogenic sources. The Principal Component Analysis identified crustal, traffic and industries as emission sources for coarse and fine particles.

**Keywords:** PM2.5-10; PM2.5; XRF, NAA, EF Analysis, PCA.

### 1. INTRODUCTION

Air pollution has become a matter of global concern and attracts more and more interest and attention (Mrkajic et al, 2010; Gümrukçüoğlu, 2011). The term of air pollution is used to describe any unwanted chemicals or others materials that contaminate the air resulting in the degradation of air quality.

A major component in air urban air pollution is particulate matter (PM), which can be coarse or fine. Coarse particulates can be regarded as those with a diameter greater than 2.5  $\mu\text{m}$ , and fine particles less than 2.5  $\mu\text{m}$ . Coarse particles usually contains materials from the earth's crust and dust from vehicles and industrial plants; while fine particles contain the

secondary formed aerosols, combustion particles and re-condensed organic and metallic vapors.

In Morocco, the major built-up areas, like most international metropolitan areas, have experienced great urban and industrial development over the last twenty years. Such development has of course resulted in the development of the productive fabric, major employment opportunities for hundreds of thousands of families, but to the detriment of the mobility and quality of the environment.

Air pollution in Morocco, particularly in the big and industrial cities (Casablanca, Rabat, El Jadida, Safi, Kenitra) (Bounakhla et al., 2003; El Khoukhi et al., 2004; Houssaini et al, 2007; Zghaid et al., 2009), is a major concern for both the local public authorities and citizens. In 2003, the air

law was promulgated in order to prevent, eliminate and reduce the release of pollutants from fixed or mobile sources.

For appropriate air quality management, the sources of air particles pollution must be known and identified. For this reason, the nuclear analytical techniques such as instrumental neutron activation analysis (INAA) and X-Ray Fluorescence (XRF) is widely used to analysis the filter samples of airborne particulate matters. Nuclear techniques are used to the characterization of particulate matter from air pollution around the world (Cohen et al., 2004; Kim Oanh et al., 2006; Santoso et al., 2008; Biswas et al., 2003; Chueinta et al., 2000; Begum et al., 2005; Chung et al., 2005; Hien et al., 2005; Trompetter et al., 2002). These techniques are generally multi-elemental techniques and have distinct advantages for particles characterization. Their simultaneous measurement, multi-elemental capabilities, high sensitivity, short analysis time and non destructive properties make them ideal for this type of work. Furthermore, the large numbers of samples obtained from air quality monitoring can be most efficiency characterized by multi-elemental techniques such as INAA and XRF. These techniques can produce large data sets for statistically characterizing pollution sources as well as estimating different source contributions which is critical to understanding air pollution and air quality management. Therefore, the characterization of airborne particulate matter becomes increasingly important.

In CNESTEN (National Center for Energy, Sciences and Nuclear Techniques), the available nuclear analytical techniques are X-Ray Fluorescence (XRF) and Instrumental Neutron Activation Analysis (INAA). For XRF technique, two laboratories are installed: the first is the Wavelength Dispersive X Ray Fluorescence (WDXRF) and the second is the Total Reflection X-Ray Fluorescence (TXRF). For NAA technique, the CNESTEN reactor, designed by General Atomics, is a 2 MW TRIGA Mark II nuclear research reactor; it is equipped with 3 lateral channels and 1 tangential channel.

The present paper discusses the application of INAA and XRF in characterization the airborne particulate matter in Meknes City, Morocco. The work is focused on particles with aerodynamic diameter less than 2.5  $\mu\text{m}$  (fine particles) and between 2.5 and 10  $\mu\text{m}$  (coarse particles).

## 2. METHODS AND MATERIALS

### 2.1. Sampling

Sampling of air particulate matter was carried

out in Meknes City during the period from March 2007 to April 2008. Meknes City is located in northern of central Morocco at the medium Atlas. It contains about 171 industrial units (MICEM, 2000). From March to October 2007, the sampling was carried out in the vicinity of a travel agency (Moroccan Transport Company 'CTM': FAR Avenue) situated in the FAR Avenue. This site is situated in the center of Meknes city, near a taxis station and not far from railway traffic. Since November 2007 to April 2008, and because of logistic reasons, the site was changed to another similar site (it's the Allal Ben Abdallah Avenue) which is not so far from the first site. The second site is located on the roof of the Allal Ben Abdallah College situated in the Allal Ben Abdallah Boulevard that is less narrow than the FAR Avenue. This site is characterized by dense road traffic, mainly during the rush hours, the presence of bus and taxis stops and traffic with one-way street. The particulate matter was collected using the following samplers:

- Dichotomous sampler (Graseby Anderson) (Clyde, 1993; Compodium Method, 1999) which allow sampling separately the fraction  $\text{PM}_{2.5}$  (Fine) and the fraction  $\text{PM}_{2.5-10}$  (Coarse). The used filters were in membrane or Teflon with a diameter of 35-37 mm and a porosity of 2  $\mu\text{m}$ .

- Gent sampler (Hopke et al., 1997), composed from two filters of polycarbonate placed in series. The filter porosity (0.4  $\mu\text{m}$  for the first and 8  $\mu\text{m}$  for the second), coupled with a flow rate of approximately 17 l/mn, allows to obtain a fine fraction (roughly equivalent to  $\text{PM}_{2.5}$ ) and coarse fraction (type TSP). This type of sampler, recommended by the IAEA in Africa, has the advantage of being simple to use and very cheap, but with no flow control. The used filters were in polycarbonate (Nuclepore) with a diameter of 47 mm and a porosity of 0.4  $\mu\text{m}$  for fine and 8  $\mu\text{m}$  for coarse particles.

- Partisol 2000 (Thermo) equipped with PM10 head. The used filters were in Teflon with a porosity of 2  $\mu\text{m}$  (Zefluor de Pall Gellman).

### 2.2. Determination of elemental concentration

Airborne particulate matter retained on the filter may be analyzed chemically by a variety of methods. In this paper, only nuclear analytical techniques (NATs) are considered.

So, the used NATs in the present work were: Neutron activation analysis (NAA) and Total reflection X-Ray Fluorescence (TXRF).

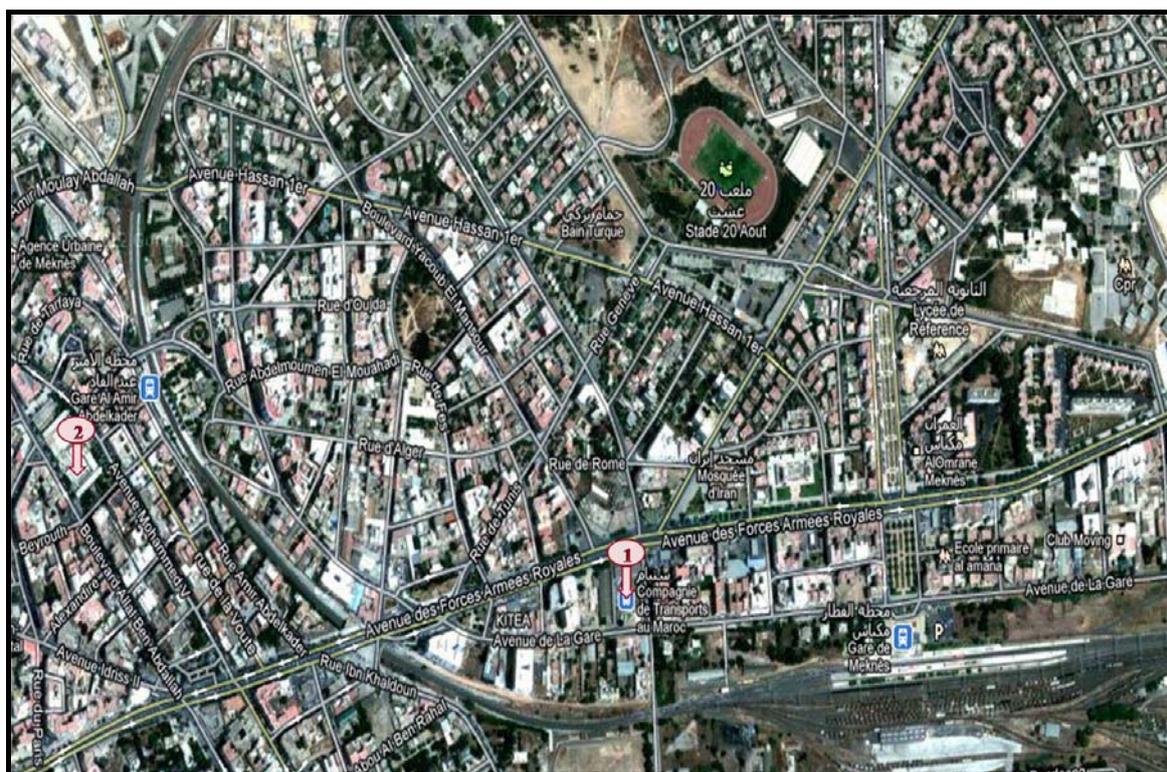


Figure 1. Localization of sampling site (1: CTM; 2: Allal Ben Abdallah College).

### 2.1.1. XRF analysis

For XRF analysis, the used technique is Total Reflection X-Ray Fluorescence X. This technique is a variant of Energy Dispersive X-Ray Fluorescence (EDXRF) (Van Grieken & Markowicz, 2002). The used TXRF system in this work is that installed in National Center of Energy, Sciences and Nuclear Techniques (CNESTEN) in Rabat in Morocco. This system is equipped with a 2 kW power fine-focus X ray tube with a molybdenum anode operating in most case at 30 mA and 50 kV. X ray beam is monochromatized by using a multilayer (W/C) crystal. The fluorescent X rays of the sample are detected by a Si(Li) detector with a resolution of 165 eV at 5.9 keV and next analyzed by a Canberra S100 multi-channel analyzer card coupled to a computer for data storage and analysis.

The filters analyzed with this technique were those collected with Gent sampler. The collected samples were digested in 10 ml of HNO<sub>3</sub> in microwave digestion system of type Mars 5 of CEM.

### 2.1.3. NAA analysis

NAA is one of the most sensitive methods used to measure the concentration of trace amounts of many elements in a variety of sample types (Weizhi, 2000).

The NAA analyses were carried out in Nuclear Engineering Teaching Laboratory (NETL) in Texas University in Austin (USA). The NETL reactor,

designed by General Atomics, is a TRIGA Mark II nuclear research reactor. The NETL reactor has in-core irradiation facilities and five beam ports. The reactor is capable of steady-state operation at power levels up to 1 MW or pulsing mode operation where powers as high as 1500 MW are achieved for about 10 msec.

The NETL facilities utilize a set of gamma detectors that include a Compton suppression system, gamma coincidence based analysis, and several more traditional high efficiency detection systems for the measurement and assay of radioactive materials. The filters analyzed with this technique were those collected during 24, 26 and 28 July 2008 using Partisol and Dichotomous samplers. The sampling was carried out during 24 hours.

## 2. RESULTS AND DISCUSSION

### 2.1. Comparison between Partisol and Dichotomous results

NAA technique permits as to evaluate the concentrations of the following elements in the filters collected with Partisol and Dichotomous samplers: As, Ba, Br, Co, I, Mg, Na, Sb, V, Ca, Cl, Cr, Cu, Fe, Mn, Sr, Ti and Zn.

Figure 2 correlates the averages contents (expressed in ng/Nm<sup>3</sup>) of chemical elements evaluated in filter samples of Partisol sampler with those of Dichotomous samplers.

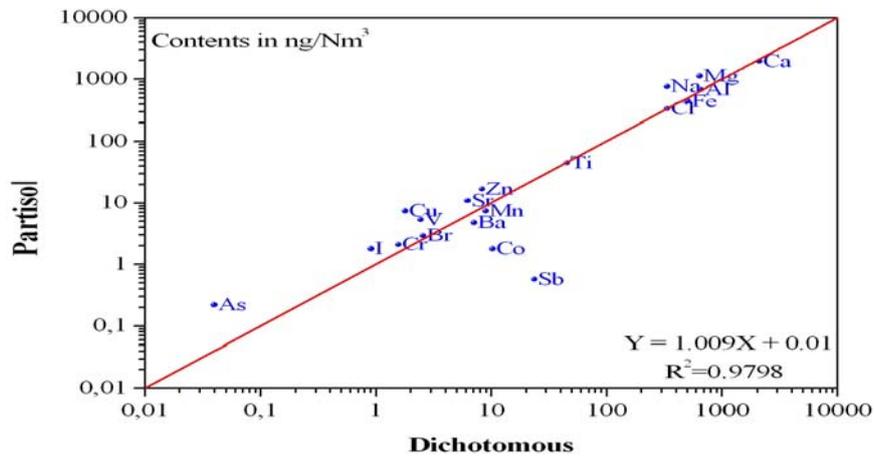


Figure 2. Elemental compositions, obtained with NAA, of filters collected with Partisol and Dichotomous samplers.

Figure 2 shows that, for almost all the analyzed elements, the concentrations evaluated from filters collected with Partisol sampler are correlated and equivalent to those collected with Dichotomous sampler, during the same period of sampling. So, this means that the use of Partisol or Dichotomous as sampler would influence significantly the obtained results. The treatment of the results of the chemical composition of the analyzed filters by NAA revealed that Ca is the predominant element with average concentration higher than 2000 ng/Nm<sup>3</sup>, i.e. a percentage of 43% of the global composition; followed by Al, Cl, Fe,

Mg and Na with 11% of abundance and average concentrations greater than 500 ng/Nm<sup>3</sup>.

Then, Ti with 1% of abundance (42 ng/Nm<sup>3</sup>) and finally (As, Ba, Br, Co, Cr, Cu, I, Mn, Sb, Sr, Zn and V) with average concentrations equal to 8 ng/Nm<sup>3</sup> corresponding to an abundance lower than 1% of the total composition. A probable origin of the more abundant elements (Al, Mg, Ca, Ti and Co) could be crustal or they could come from cement industry. However, As, B, Ba, Br, Cu, Sb, V and Zn are considered as tracers of automobile traffic (Mouthon, 2003; Vecchi et al., 2004; Vecchi et al., 2007; Zibing et al., 2006).

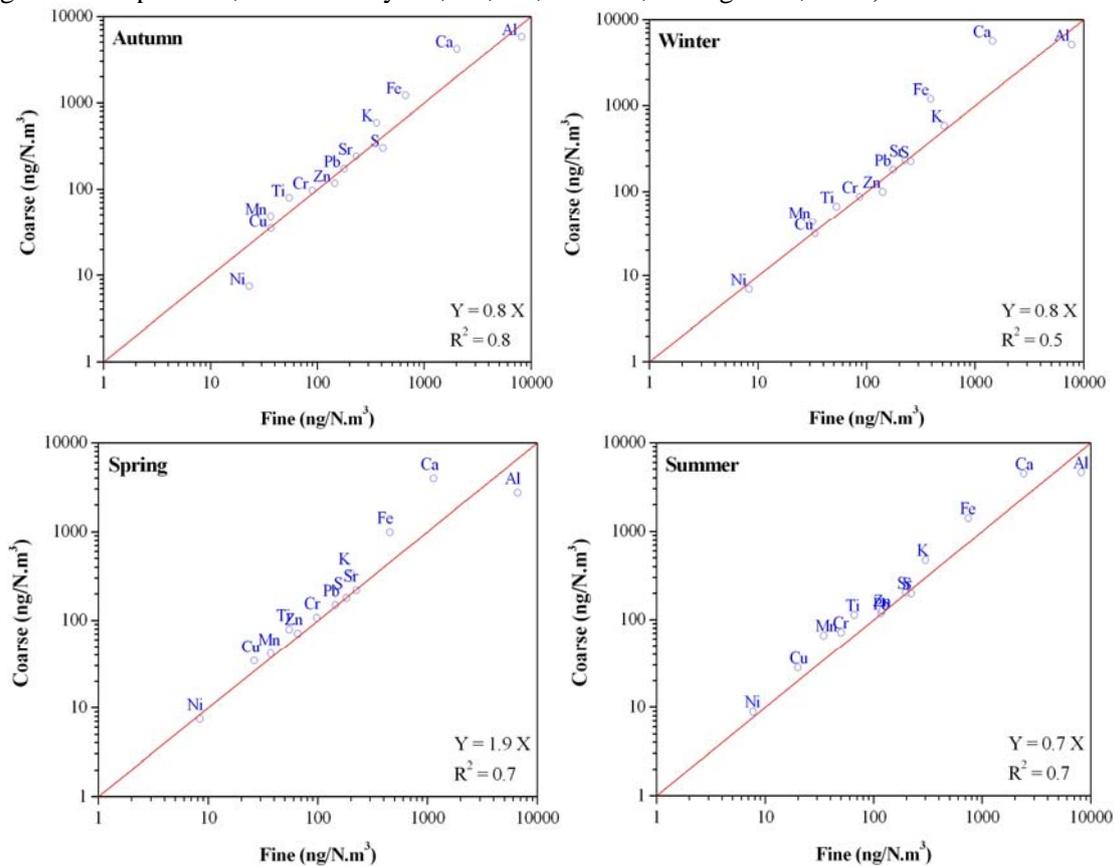


Figure 3. Repartition of element concentrations between coarse and fine fractions during the sampling period.

## 2.2. Seasonal variation of element mass concentration

Figure 3 shows the repartition of the studied elements between fine and coarse fractions for the four seasons.

As shown in Figure 3, the variation of the repartition of element concentrations between fine and coarse fractions is generally not very significant. Nevertheless, the repartition or distribution of some elements differs between the two fractions. So, Ca and Fe are more distributed in coarse fractions. Al, contrary to what we expect, is more concentrated in fine fractions. For, Cl, Cr, Cu, Pb, S, Sr, Ti and Zn, they are equally distributed on the both fractions. However, K, Mn and Ni distributions change from one season to another. Thus, Ni in winter and summer has the same repartition on fine and coarse fractions; while during spring and autumn, it has a slight tendency towards fine fractions. For Mn, it is mostly distributed on coarse fractions except in spring where it was found to be more distributed in fine fractions. Finally, K in winter and spring is similarly distributed on the two fractions, while in summer and autumn; it has a preferential distribution on coarse fractions.

In the Table 1, we have reported the values of (Fine/Coarse) ratios calculated for the four seasons. So, when the ratio (Fine/Coarse) is equal to 0.5, we say that we have an equal distribution between fine and coarse fractions. If this ratio exceeds 0.5, the element is more distributed in coarse fractions (case of terrigenous or natural elements). Whereas, if the ratio is less than 0.5, the element is more important on the fine fractions (case of anthropogenic sources). So, the conclusions obtained by the analysis of Table 1 are agreed with those of Figure 3 and confirm the deductions made from this figure.

## 2.3. Source apportionment by enrichment factors

Enrichment factor (EF) is widely used to identify the anthropogenic source of metallic elements and it is generally applied to show the degree of enrichment of a given element compared

to the relative abundance of that element in crustal material (Gao et al., 1992). In the present study, crustal  $E_F$ 's are calculated with Al as reference element. In the absence of data about the soil of the studied region, the average composition of continental crust was considered to calculate the EF (Squalli et al., 2007). The formula used for enrichment factor calculation is:

$$EF_i = (i/j)_{air}/(i/j)_{crust}, (1)$$

where  $EF_i$  is the enrichment factor of species  $i$ ;  $j$  is the reference element for crustal material;  $(i/j)_{air}$  is the ratio of species  $i$  to species  $j$  in the aerosol sample and  $(i/j)_{crust}$  is the ratio of species  $i$  to species  $j$  in the crust (Quiterio et al., 2004).

It is believed that the element should be enriched as if the EF of each element is greater than 10, indicating that atmospheric particulate matters come mainly from human activities. The greater value of enrichment factor shows the greater possibility of air pollution from anthropogenic sources. As the EF of an element is close to 1, it suggests that atmospheric particulate matters come mainly from soil or weathered rocks (Huang et al., 2010).

Figures 4 and 5 show the EF values obtained for elements (Ca, Cr, Cu, K, Fe, Mn, Ni, Pb and Zn) of coarse and fine particulate matter related to crustal concentrations.

For the both fractions, the elements with enrichment factors less than 1 were K and Fe which is an indication that these elements came mainly from natural sources; Ca, Mn and Ni with EF between 1 and 10 were mainly from natural sources but we cannot ignore the influences of human activities. The elements with EF higher than 10 were Cr, Cu, Zn and Pb which indicates that their origins are mainly anthropogenic.

For seasonal variations of EF in the studied area, the more significant variations were observed for coarse than fine fractions. Moreover, the higher pollution levels for coarse fractions were detected in June for sampling elaborated in 2007 and in March for sampling carried out in 2008.

Table 1. Calculated values of (Fine/Coarse) ratios.

Season	Al	Ca	Cl	Cr	Cu	Fe	K	Mn	Ni	Pb	S	Sr	Ti	Zn
Autumn	0.58	0.34	0.56	0.47	0.51	0.37	0.41	0.45	0.53	0.51	0.56	0.48	0.39	0.55
Winter	0.61	0.22	0.55	0.50	0.51	0.25	0.44	0.41	0.60	0.49	0.52	0.50	0.42	0.55
Spring	0.70	0.23	0.45	0.51	0.48	0.32	0.40	0.47	0.54	0.50	0.54	0.50	0.42	0.48
Summer	0.66	0.35	0.46	0.42	0.43	0.35	0.39	0.35	0.50	0.50	0.53	0.49	0.39	0.48
Year	0.65	0.29	0.50	0.47	0.48	0.32	0.41	0.42	0.54	0.50	0.54	0.49	0.40	0.52

## 2.4. Source apportionment by PCA

Principal Component Analysis with Varimax rotation was performed with selected 9 elements and 58 samples to extract the major sources of trace elements and the technique yielded a number of key factors. The factor loadings obtained for coarse and fine particles are presented in table 2.

The analysis of factor loadings of the coarse particles showed a significant correlation among elements of crustal source (Al, Ca, Fe, K, Mn and Zn). The second factor regroups Cr and Cu which are mostly used in abundance in the industries of textile, dyes and intermediates; it is suspected that this could be the source of emission of these elements having high loadings in the second factor of coarse particles. The third and last factor was found with high loadings of Pb which could be from the nearby road traffic. For the PCA study of fine particles, other factors were found different from that identified for coarse particles. So, the first factor

was associated with Cr, Cu, Mn and Pb which could be related to the road traffic emissions. The second factor with the group constituted of Ca, Fe and K could be identified as crustal source.

The last factor indicated a strong correlation between Al and Zn and could be considered as natural sources but we cannot neglect the contribution of anthropogenic activities.

## 3. CONCLUSIONS

Coarse and fine particulate matters (PM<sub>2.5</sub> and PM<sub>2.5-10</sub>) were collected at an urban area near road traffic axis in Meknes City. The aim of this study was to evaluate the air quality and the mainly sources of emission in this Moroccan.

TXRF and NAA techniques were used as elemental techniques to determine the concentration levels of elements in the collected filter samples during the period from March 2007 to November 2008.

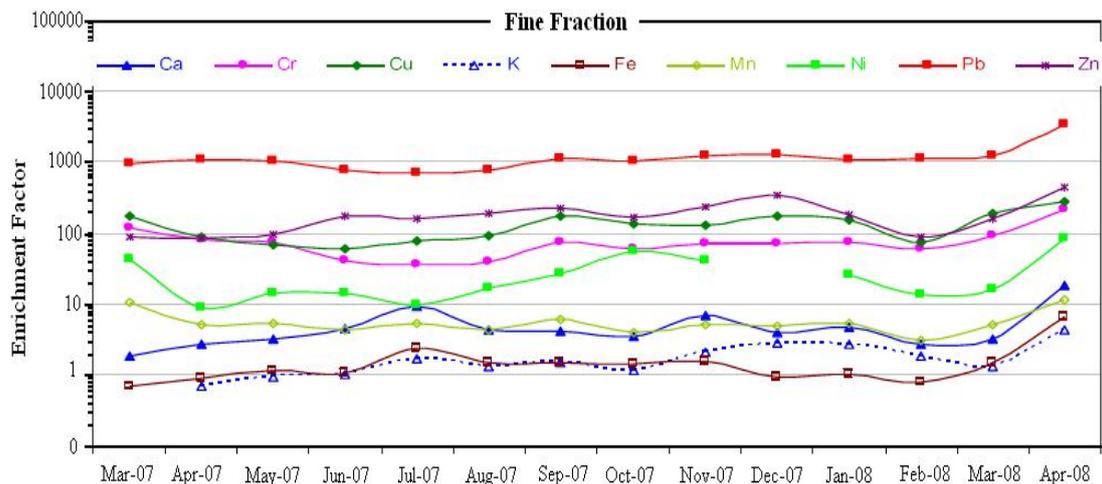


Figure 4. Seasonal variations of enrichment factors in fine fractions.

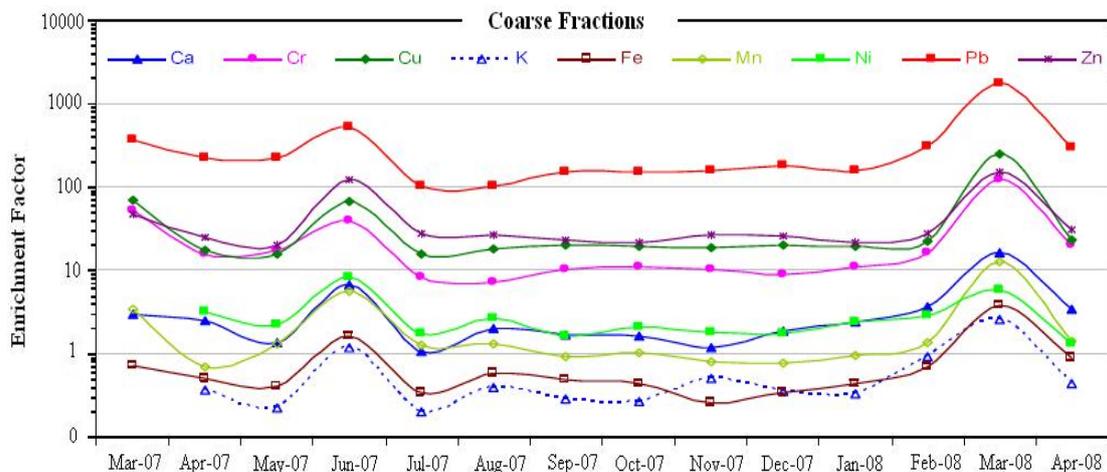


Figure 5. Seasonal variations of enrichment factors in coarse fractions.

Table 2. Varimax rotated PCA factor loadings for fine and coarse particles.

Element	Fine particles			Coarse particles		
	F1	F2	F3	F1	F2	F3
Al	0.09	0.01	<b>0.71</b>	<b>0.54</b>	0.04	0.03
Ca	-0.23	<b>0.79</b>	0.28	<b>0.73</b>	0.01	0.51
Cr	<b>0.83</b>	-0.22	-0.12	-0.18	<b>0.91</b>	0.13
Cu	<b>0.68</b>	-0.05	0.40	0.43	<b>0.76</b>	0.16
Fe	0.08	<b>0.84</b>	0.00	<b>0.85</b>	-0.12	0.22
K	0.52	<b>0.54</b>	0.23	<b>0.81</b>	0.03	0.42
Mn	<b>0.59</b>	0.44	-0.14	<b>0.68</b>	0.45	-0.33
Pb	<b>0.70</b>	0.14	0.36	0.17	0.22	<b>0.91</b>
Zn	0.08	0.24	<b>0.83</b>	<b>0.74</b>	0.31	0.05
Eigen Values	2.34	1.94	<b>1.63</b>	<b>3.46</b>	1.78	1.47
% Variance	25.99	21.60	<b>18.16</b>	<b>38.42</b>	19.76	16.30

The seasonal variations of element contents between fine and coarse fractions indicated that most of the analyzed elements (Cl, Cr, Cu, Pb, S, Sr, Ti and Zn) are equally distributed on both fractions; except for some elements like Ca that was more distributed in coarse fractions and Ni that was more concentrated in fine fractions. These results were supported by the calculation of the ratios (Fine/Coarse) of each element for the four seasons. In fact, the values of the calculated ratios, for most studied elements, were less than 0.5.

The enrichment factor analysis indicated that, for both fractions, Fe and K came mainly from natural sources; Ca, Mn and Ni were mainly from natural sources but we cannot ignore the influences of human activities; Cr, Cu, Zn and Pb which came mainly from anthropogenic sources. PCA studies identified three common emission sources for coarse and fine particulate matters. These sources were identified as crustal, road traffic and industries emissions.

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