

SEM-EDX analysis of various sizes aerosols in Delhi India

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Abstract Scanning electron microscopy-energy dispersive X-ray analysis (SEM-EDX) was used to understand the differences in morphology, elemental composition and particle density of aerosols in different five size ranges to further investigate the potential sources as well as transport of pollutants from/at a much polluted and a very clean area of Delhi. Aerosol samples were obtained in five different size ranges viz. ≥ 10.9 , 10.9–5.4, 5.4–1.6, 1.6–0.7 and ≤ 0.7 μm from a considerably very clean and a much polluted area of Delhi. It was observed that at polluted area most of the particles irrespective of size are of anthropogenic origin. At clean area, in coarse size fractions particles are of natural origin while in fine size range the presence of anthropogenic particles suggests the transport of particles from one area to the other.

Keywords Scanning electron microscope · Energy dispersive X-ray · Size fraction · Aerosols · Metals · Delhi

Introduction

Trace metals are released into the atmosphere during combustion of fossil fuels and woods as well as during high temperature industrial processes and waste incineration (Allen et al. 2001). This results in elevated metal concentrations which can pose a serious risk to human health. Many epidemiological studies provide evidence of causal association between exposures to different size airborne particles and worst health effects, especially among the old age people. Particles of diameter less than 10 μm (PM10), especially the finer particles (PM2.5 and PM1) have been found to be associated with cardio-respiratory problems and mortality (Schwartz 1994; Pope et al. 1995; Chapman et al. 1997; Berube et al. 1999; Ostro et al. 1999).

The city of Delhi is highly polluted due to its immense numbers of gasoline, compressed natural gas (CNG), diesel fueled vehicles and industrial units. Suspended particulate matters, especially fine particulates have been identified as main component of vehicular borne particles in Delhi (Srivastava and Jain 2007a). The mutagenic processes of these different size particles to the health are not yet clear. For examples it is not known what aspects of mass, size, morphology, composition or the combination of these factors might be contributing to the health problems. Researches have shown that the equivalent mass of small particles is significantly more inflammatory to the lungs than the larger sized particles of the similar

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elemental composition (Osier and Oberdorster 1997; Donaldson et al. 1998).

Scanning electron microscopes with energy dispersive X-ray techniques are powerful tools to understand their morphology, elemental composition and particle density of aerosols and to give us a better insight about the origin of the particles that whether emitted from anthropogenic (internal combustion engines or industrial activities) or the natural processes (Esbert et al. 1996; Pope 2000; Petrovic et al. 2000; Oberdorster 2001; Conner et al. 2001; Conner and Williams 2004; Bernabe et al. 2005). Many studies have already been carried out to demonstrate and establish the relationship between the origin of the particles and potential adverse effects on human health. Some have been done using SEM-EDX techniques by the particle's morphology and elemental composition (Umbri et al. 1999; Chabas and Lefevre 2000; Ma et al. 2001; Bernabe and Carretero 2003; Liu et al. 2005). The elemental composition of atmospheric particles is some times more useful than their bulk elemental composition with a view to establish their origin and their potential effects on human health. Still, the studies on elemental composition of atmospheric particles are rather limited (Querol et al. 1999; Pina et al. 2000, 2002; Shi et al. 2003; Breed et al. 2002; Querol et al. 2002; Ekosse et al. 2004; Mathis et al. 2004; Suzuki 2006).

In Indian context no comprehensive study using SEM-EDX has been done, except one study in which Srivastava and Jain (2007b) used SEM for the morphological analysis of pollutants inside an indoor environment of Delhi. In the present study an application of elemental composition, morphology and particle density of aerosols by SEM-EDX techniques have been used, instead of the bulk elemental composition, to evaluate the potential sources of air pollution and their transport with respect to different sizes.

Material and methods

Area description

Sampling was carried out in Delhi, the capital city of India. Delhi, has over 14 million inhabitants, 4.8 millions of registered vehicles, three coal based thermal power plants (Badarpur, Rajghat and Indraprastha) and 125,000 industrial units (Government of

India 2001; Economic Survey of Delhi 2006). It lies in the subtropical belt between 76°50' E–77°23' E and 28°12' N–28°53' N. Its climate is semi-arid and consists of summer (March–May), monsoon (June–August) post-monsoon (September–November) and winter (December–February) seasons. It experiences a maximum temperature of ~45–48°C in June during summer and minimum of ~1–2°C in January during winter. Two different sites were chosen to obtain samples viz. Jawaharlal Nehru University (JNU) and Income Tax Office (ITO). These two sites are approximately 14 km away from each other (Fig. 1). Further descriptions of the sites are provided below.

JNU

Jawaharlal Nehru University (JNU) is India's premier educational institute. JNU campus is far off from any industrial activity, with no industrial unit nearby within several kilometers of it. It is densely vegetated and characterized by very low vehicular traffic. It is situated on the Aravali mountain (oldest mountain of India) ranges, which contains metamorphic quartzite rock (loosely bound) and has gone many phases of weathering. This is considerably one of the cleanest areas of Delhi.

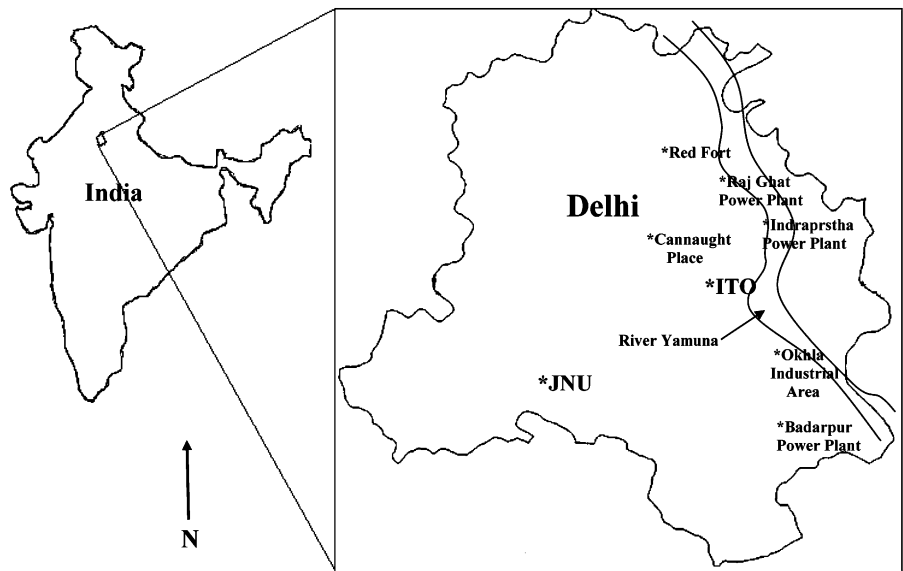
ITO

ITO is busiest traffic junction and has the heaviest vehicular traffic density of Delhi. About 210,000.00 vehicles pass everyday (24 h; CRRRI 2005). It is situated on the bank of river Yamuna. It is also closer to two thermal power plants (viz. Rajghat and Indraprastha) out of three in Delhi and not far away from Okhla industrial area. Some water and sewer treatment plants are also in the close vicinity. Cannaught Place and Red Fort area, the hub of Delhi's commercial activities, are also in the close proximity.

Ambient sampling

To determine the morphology, elemental composition, particle density and source contribution of aerosols, sampling was performed at aforementioned sites in first week of January 2006. This period is characterized by calm windy conditions and intense winter when occurrence of inversion is very frequent. A five-

Fig. 1 Map showing the sampling locations

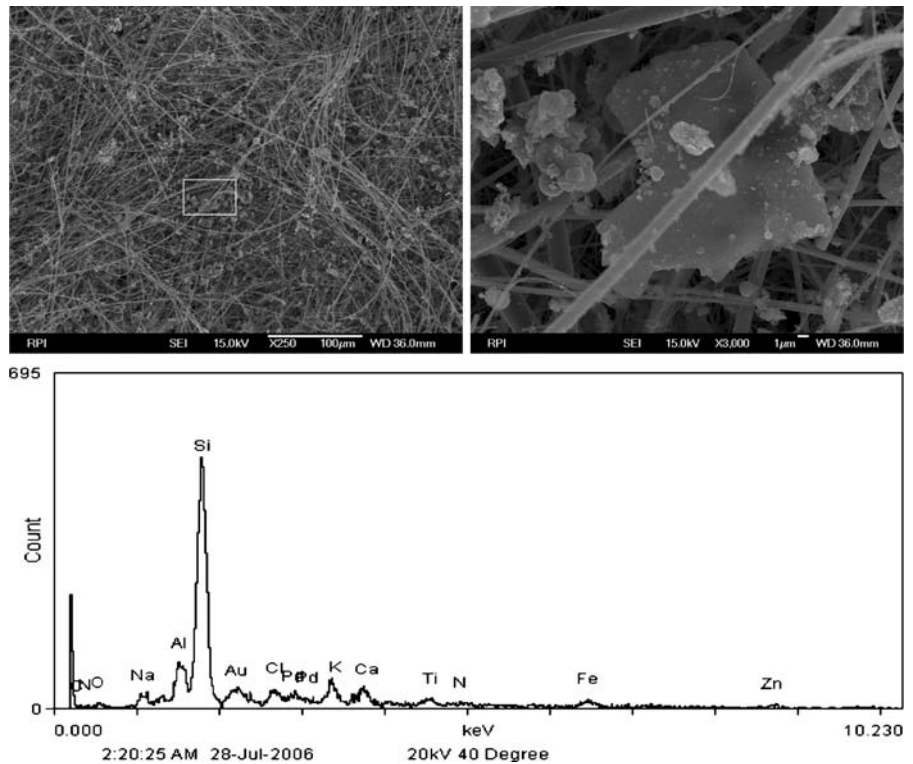


stage cascade particulate separator (CPS-105, Kimoto Electric Co. Ltd. Japan) was used at an average flow rate of 600 l m^{-1} . To collect sufficient amount of aerosols the sampling was done for 24 h at both the sites. CPS-105 collects particles in five different size

ranges viz. ≥ 10.9 (Stage-1), $10.9\text{--}5.4$ (Stage-2), $5.4\text{--}1.6$ (Stage-3), $1.6\text{--}0.7$ (Stage-4) and $\leq 0.7 \mu\text{m}$ (Stage-5).

The samples were collected on Whatman, EPM-2000, glass microfiber filters. Each set of samples consists of five different filters for various size ranges.

Fig. 2 Scanning electron micrographs and EDX-spectrum of aerosols in size range $\geq 10.9 \mu\text{m}$ at JNU



The filters were kept in vacuum desiccators for 24 h to remove any moisture content before mounting them on the air sampler. After the sampling the filters were immediately transferred to vacuum desiccators to again de-moisturize them in the same manner.

SEM-EDX measurement

The samples (dry filter papers) were randomly cut in 1 mm² size out of the main filter (Xie et al. 2005). A very thin film of gold and palladium (Au–Pd) was deposited on the surface of the samples to make them electrically conductive using vacuum coating unit. This extremely fine coating was done through the evaporation of Au–Pd plate under inert atmosphere (argon environment). These samples were mounted on electron microprobe stubs. The SEM-EDX analyses were carried out with the help of a computer controlled field emission SEM (JEOL JSM-6330F, JEOL Ltd., Akishima, Tokyo, Japan) equipped with a EVEX-EDX detection system, (State Road Princeton,

NJ, 8540). In the present investigation, the SEM was used in its most common mode the emissive mode. The other parameters are as follows, Detector: Si (Li), Limit of detection, 500,000; accuracy, 10%; energy, 20 kV (accelerating voltages, 0.5–30 kV) and spatial resolution, 2 nm (theoretical).

Results and discussion

In order to understand the sources and transport of pollutants, and to have better idea of vehicular pollution the biggest menace in Delhi, electron micrographs of aerosols in five different size ranges with EDX-spectra are provided in Figs. 2, 3, 4, 5, 6, 7, 8, 9, 10, and 11. Two micrographs in each size ranges at both the locations were taken. One is the micrograph of particles in bulk (left hand), while the other (right hand) is the zoomed-in view of the particles to understand the morphology. At the bottom of the micrographs is provided the bulk EDX in the

Fig. 3 Scanning electron micrographs and EDX-spectrum of aerosols in size range $\geq 10.9 \mu\text{m}$ at ITO

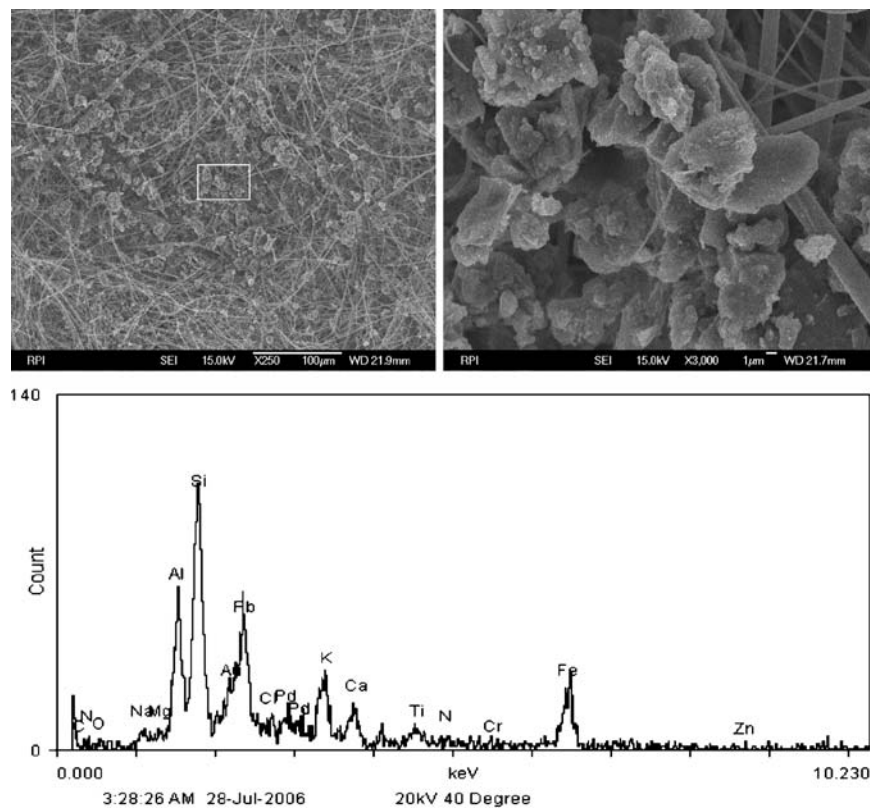


Fig. 4 Scanning electron micrographs and EDX-spectrum of aerosols in size range 10.9–5.4 μm at JNU

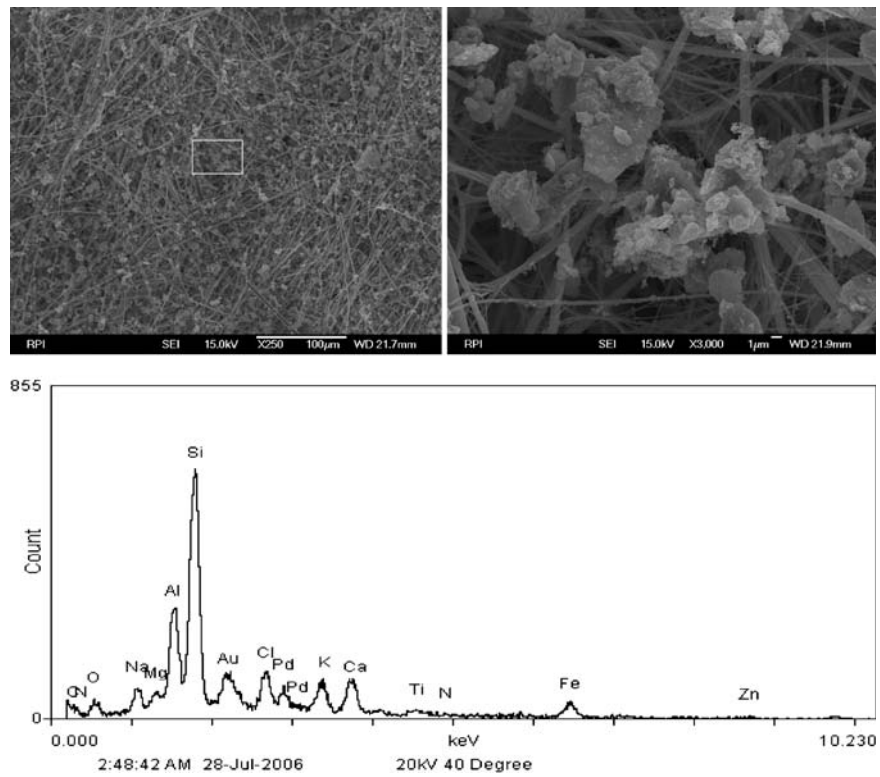


Fig. 5 Scanning electron micrographs and EDX-spectrum of aerosols in size range 10.9–5.4 μm at ITO

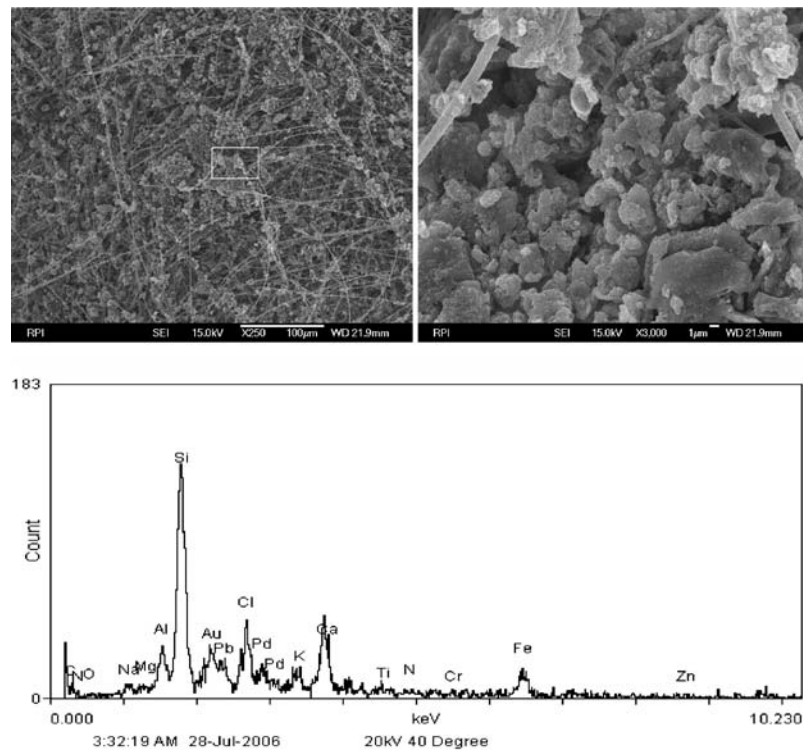


Fig. 6 Scanning electron micrographs and EDX-spectrum of aerosols in size range 5.4–1.6 μm at JNU

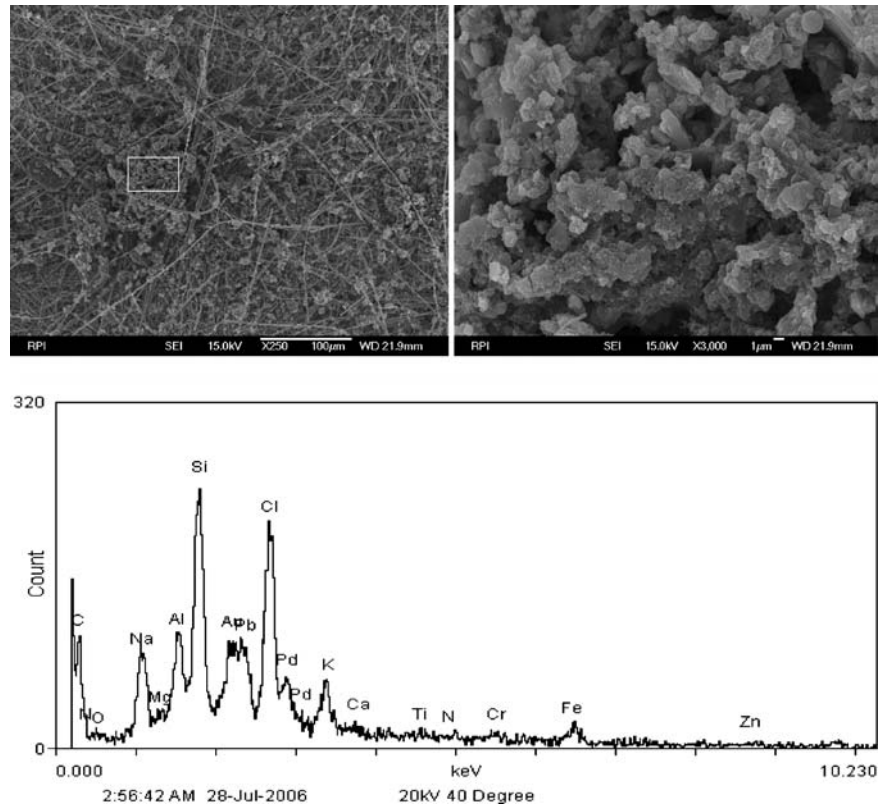


Fig. 7 Scanning electron micrographs and EDX-spectrum of aerosols in size range 5.4–1.6 μm at ITO

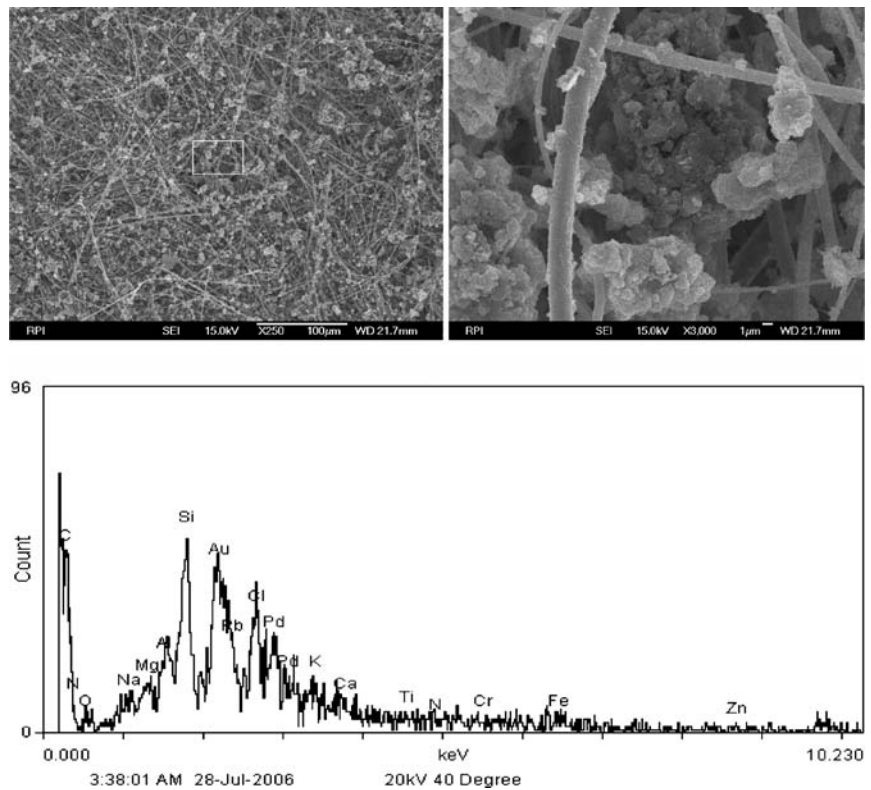


Fig. 8 Scanning electron micrographs and EDX-spectrum of aerosols in size range 1.6–0.7 μm at JNU

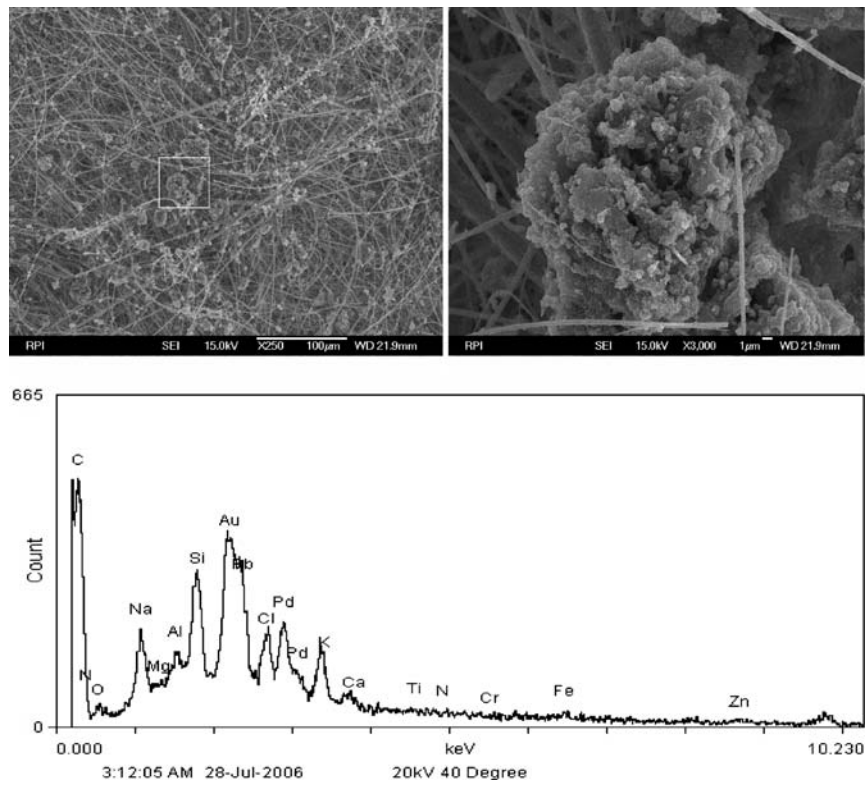
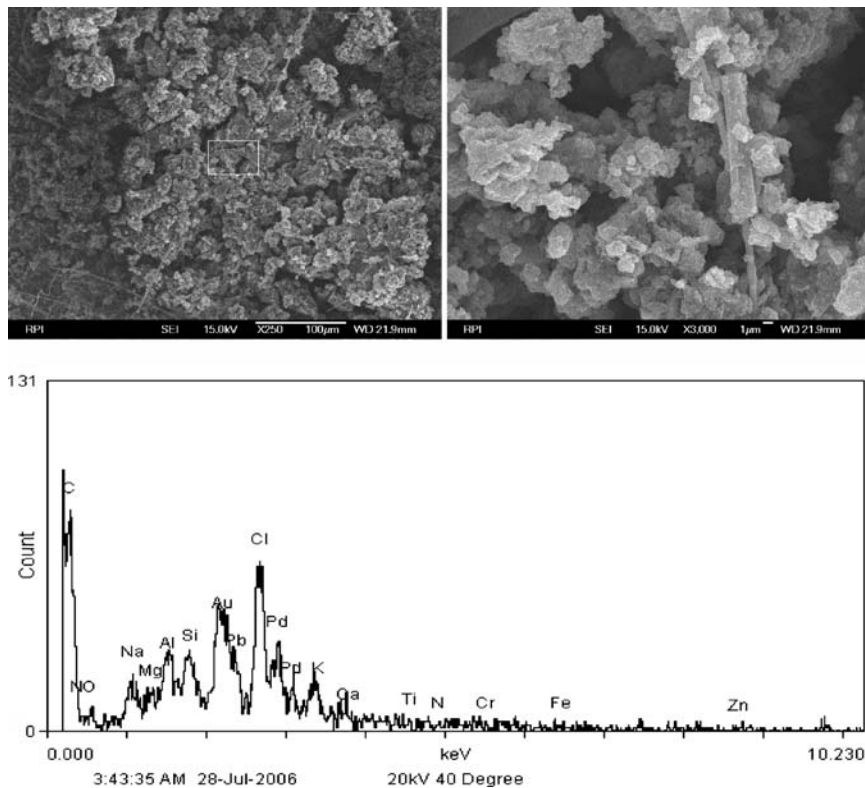


Fig. 9 Scanning electron micrographs and EDX-spectrum of aerosols in size range 1.6–0.7 μm at ITO



respective size range. Here, it is important to mention that the occurrence of gold (Au) and palladium (Pd) in EDX-spectra, especially in the lower size ranges, is due to the fact that Au–Pd was used for a very fine coating of all the samples to make them electrically conductive (refer “SEM-EDX measurement”). These two elements have not been included in the discussion. Each size range at both the locations has been discussed separately. EDX spectra have further been analyzed for concentration of different metals in all the size ranges at both sites (X-Ray Micro Analysis 2006). This has been presented in Table 2.

Size range $\geq 10.9 \mu\text{M}$ (Stage-1)

From Figs. 2 and 3 it can be inferred that in the size range of $\geq 10.9 \mu\text{m}$, at JNU and ITO there are differences between the particle morphology, density and elemental composition. At JNU the particles are flaky and crystal shaped. This is because JNU is highly vegetated and situated on the Aravali mountain ranges. The Aravali ranges contain huge amount of loosely bound quartzite, which are generally crystal and crystalline shaped. Elemental composition of the particles by Table 2 (EDX-spectrum) shows that Si and Al are dominating over all the elements. This confirms that at JNU in this particular size range most of the particles are of natural origin i.e. mountainous (dusts eroded from mountainous rocks) aerosols.

At ITO the case is different, here the particles are mostly rounded or crystalline shaped. The density also seems to be much higher than JNU. It can also be confirmed from Table 1 that the aerosols concentration in this size range at ITO is almost three times than at JNU. Elemental composition of the particles reveals the presence of many metals viz. Si, Al, Pb, K, Fe, Ca and C in appreciable quantity. The occurrence of these metals point towards the presence of sources other than natural. These sources could be vehicular, industrial and the two thermal power plants in the vicinity of sampling site (Venugopal and Luckey 1978; Mehra et al. 1998; Srivastava and Jain 2007a).

Size range 10.9–5.4 μM (Stage-2)

From Figs. 4 and 5 it is clear that, there is not much difference between two sites as far as particle's morphology and elemental composition are concerned.

The only little differences that can be observed are particle density at ITO is more than JNU (it is also clear from Table 1). Now the particles are rounded at JNU also. Elemental composition of the particles from Table 2 reveals that Si is predominant at both the places. Apart from Si the other elements present are Al, Pb, Cl, Ca, Na, Mg and Fe.

From the above discussions the only plausible inference that can be drawn is: transport of particles become significant in lower size ranges (especially PM10). Since JNU has absolutely no industries even in the far vicinity and characterized by very less vehicular activities, thus the occurrence of rounded shaped particles and above mentioned metals clearly indicate the presence of migratory particles originated by anthropogenic activities at other places (Venugopal and Luckey 1978; Srivastava and Jain 2007a).

Size range 5.4–1.6 μM (Stage-3)

From Figs. 6 and 7 and Table 2 it can be inferred that the dominance of carbon (soot) has started. However its dominance is fourth after Si, Cl, Na, and Al at JNU but it has dominated all the other elements at ITO. At ITO the occurrence of C in maximum amount is due to its nature of heavy vehicular traffic site. Apart from C and Si the presence of other elements viz. Pb, Al, Mg, K, Ca show the presence of sources like industrial and power plant other than natural and vehicular.

In case of JNU the situation is rather different in this size range, the main elements observed are Si, Cl, Na, Al, K, and C. Presence of C in this particular size range shows the dominance of vehicular source, whilst Si, Ca, Cl and Al confirms the presence of soil as a major source in this particular size range (Taylor and McLennan 1995; Rodstedth et al. 2003).

Size range 1.6–0.7 μM (Stage-4)

Figures 8 and 9 reveal not many differences between the particles. The only differences that can be observed are the particle density and mineralogical composition to some extent. The presence of C in maximum concentration at both the places reveals dominance of vehicular pollution in this particular size range. The next dominant elements at JNU are Si, Al, Cl, Pb and Na revealing the presence of soil and crustal dust. Here, it is pertinent to mention that crustal dust of Delhi still contains some amount of Pb, generated by leaded

Fig. 10 Scanning electron micrographs and EDX-spectrum of aerosols in size range $\leq 0.7 \mu\text{m}$ at JNU

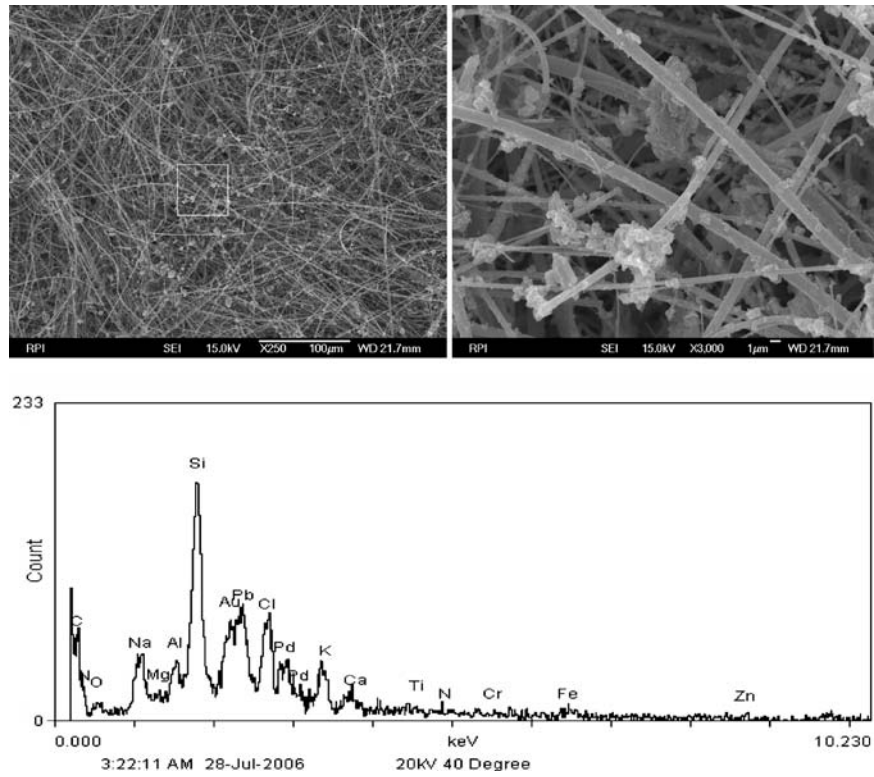


Fig. 11 Scanning electron micrographs and EDX-spectrum of aerosols in size range $\leq 0.7 \mu\text{m}$ at ITO

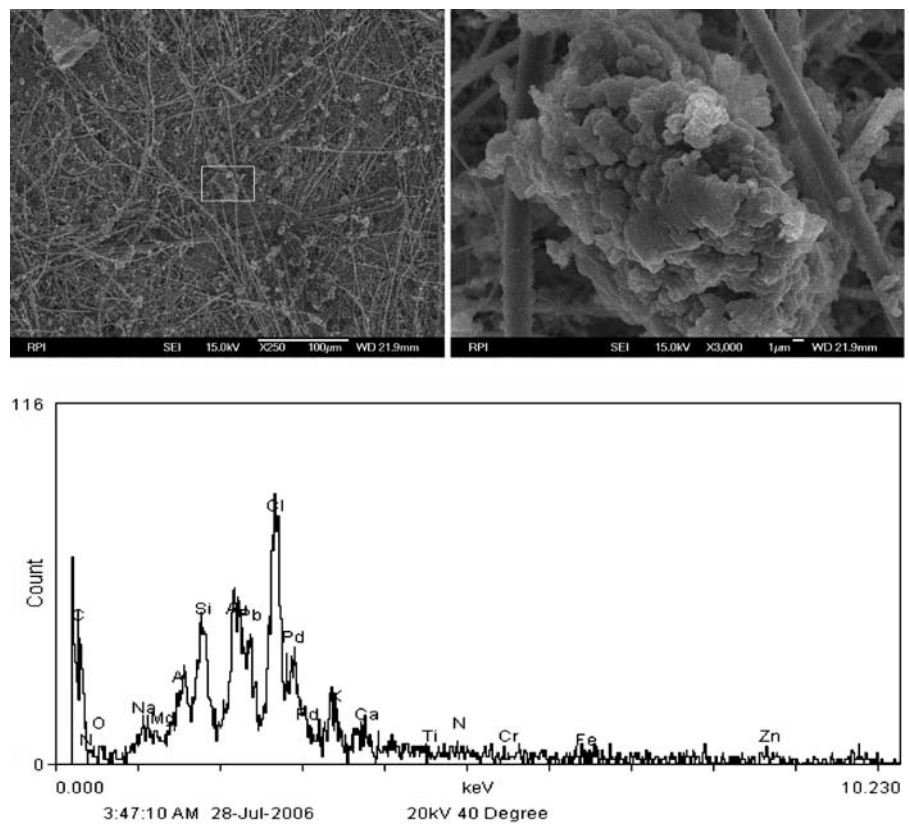


Table 1 Concentrations of aerosols in different size ranges at both the sampling locations

Size range (μm)	Conc. at JNU ($\mu\text{g m}^{-3}$)	Conc. at ITO ($\mu\text{g m}^{-3}$)
≥ 10.9	30.67	83.85
10.9–5.4	42.36	87.50
5.4–1.6	65.05	98.50
1.6–0.7	54.40	77.43
≤ 0.7	123.96	199.54
Total suspended particulate matters (TSPM)	316.44	546.82

gasoline, although it has completely been banned in Delhi since 1999 (Balachandran et al. 2000; Srivastava and Jain 2007a).

At ITO dominance of Cl has significantly exceeded other metals. The only plausible explanations for this is due to the presence of sewer and water treatment plants, and the river Yamuna in its close proximity, where use of chlorine (chloramines, chlorine dioxides etc.) as disinfectant and flocculating agent is very common (Holleman and Wiberg 2001; Fair et al. 1948). Small scale industries, manufacturing batteries and insecticides might also be contributing to the ambient Cl. Here, it is important to mention that at the flood plan of river Yamuna huge amount of vegetables are grown and the farmers use insecticide and pesticides in a large quantity. These insecticides and pesticides contain significant amount of Cl. After C and Cl at ITO the other metals present are Si, Al, Pb, K, Na and Mg, indicating toward the presence of crustal dust and anthropogenic activities as the other

sources but in a very less quantity (Venugopal and Luckey 1978; Srivastava and Jain 2007a).

Size range $\leq 0.7 \mu\text{M}$ (Stage-5)

From Figs. 10 and 11 and Table 2 it is clear that in this particular size range Si is maximum at JNU and Cl is maximum at ITO. This confirms the presence of vapor and solid phase aerosols containing Cl generated from various sources mentioned in the preceding section. The other source at both the locations in this particular size range is vehicular pollution. The other metals present are Pb, Al, Si, K and Ca at both the places, indicating anthropogenic activity as the tertiary sources.

Conclusions

Aerosols samples from a very clean area and a much polluted area were analyzed in five different size ranges using SEM-EDX techniques. The following main conclusions were drawn

- At polluted area the particles are mainly of anthropogenic origin irrespective of size range. However, particles from crustal dusts and natural origin can also be observed in insignificant amount, but in larger size ranges.
- At clean area in the larger size range most of the particles are natural.
- Particles originated from anthropogenic (mainly industrial and vehicular) activity may be trans-

Table 2 Percentage contribution of each element

Site	Stage	C	N	O	Na	Mg	Al	Si	Au	Pb	Cl	Pd	K	Ca	Ti	Cr	Fe	Zn
JNU	1	5.4	4.0	1.4	5.4	0	10.2	37.4	6.8	4.1	5.4	2.7	6.8	4.8	2.7	0	2.0	0.7
	2	2.1	1.3	2.1	4.7	3.8	12.8	34.2	10.7	0	9.8	4.7	4.3	5.1	1.7	0	2.1	0.4
	3	6.6	0	0.0	7.5	3.5	8.0	23.5	7.5	7.1	18.8	5.2	6.4	1.9	0.9	1.2	1.6	0.2
	4	19.6	4.2	1.5	9.8	5.1	7.4	14.7	27.0	16.7	10.3	10.8	9.6	3.7	2.7	2.0	2.2	1.0
	5	9.7	5.2	2.8	8.7	5.9	7.6	19.0	6.9	7.6	7.3	5.5	5.9	4.2	1.7	0.7	1.0	0.3
ITO	1	4.1	2.4	1.6	2.4	2.1	12.3	32.9	4.9	9.1	3.3	3.3	7.4	4.9	3.3	0.4	4.9	0.4
	2	2.4	0.9	0.5	2.4	0	8.5	33.0	8.5	7.5	9.4	6.1	3.3	11.3	1.9	0.9	2.8	0.5
	3	15.9	4.9	1.0	5.1	5.9	7.4	15.9	14.7	6.1	6.9	5.9	2.5	2.2	2.0	1.5	1.7	0.5
	4	19.4	3.3	2.2	5.2	2.8	6.9	6.7	11.1	7.5	16.1	10.0	5.0	1.7	0.8	0.6	0.3	0.3
	2	8.5	1.4	2.0	4.7	3.4	6.1	9.5	11.5	10.8	20.3	6.8	5.1	3.7	1.7	1.4	1.7	1.4

ported to longer distances but in fine size ranges. Thus, whether it is a clean or polluted area, concentration of pollutants associated with SPM in fine size range does not vary much.

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