

Accelerator based studies of atmospheric pollution processes

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Abstract

Accelerator based ion beam analysis techniques have been applied to the characterisation of fine particulate matter from open cut mining operations in New South Wales Australia as well as dust and sulphate emissions from deserts and industries in northern and eastern China.

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1. Introduction

Accelerator based ion beam analysis (IBA) techniques have been applied to the studies of airborne particulate matter for many years (Cohen, 1993; Cohen et al., 1996). They are well suited to such studies as they have several unique advantages; they have multi-elemental analysis capabilities (Cohen, 1998; Cohen et al., 2000, 2002a); low minimum detection limits (MDLs) for a very broad range of elements in the Periodic Table, and can quantitatively detect picograms of material in micrograms of sample (Cohen et al., 2002b). Recently the composition of airborne particulate matter (PM) with aerodynamic diameters less than $2.5\ \mu\text{m}$ (PM_{2.5}) has received considerable research interest (Malm et al., 1994). These fine particles are small enough to penetrate deep into the human lung and have direct access to the blood stream with obvious health impacts (Dockery et al., 1993); they have resident times in the atmosphere from days to weeks and travel hundreds if not thousands of kilometres across international borders (Jaffe et al., 1999); with sub-micron diameters they are very efficient light scatterers strongly influencing the public's perception of air pollution and more recently have been

strongly linked to negative climate forcing effects in global warming studies (IPCC, 2001).

Fine particles (PM_{2.5}) are mainly generated by combustion processes including emissions from motor vehicles, fossil fuel burning for power generation and large industrial processes such as ore and metals smelting. They may also include natural emissions such as fine windblown soils, emission from volcanos, sea spray and smoke from biomass burning following lightning strikes. Concentrations in air vary from a few ($\mu\text{g}/\text{m}^3$) in pristine areas to hundreds of ($\mu\text{g}/\text{m}^3$) close to smoke from major fires. Annual average PM_{2.5} concentrations in major cities around the globe will vary from around $10\ \mu\text{g}/\text{m}^3$ in Sydney to above $50\ \mu\text{g}/\text{m}^3$ in many Asian cities. In 1997 the US EPA has introduced an annual average fine particle PM_{2.5} goal of less than $15\ \mu\text{g}/\text{m}^3$ with a 24 h maximum average of $65\ \mu\text{g}/\text{m}^3$. Many cities around the world have followed the US EPA lead and introduced similar goals for urban areas. In Australia we plan, in the near future, to introduce a PM_{2.5} goal of $8\ \mu\text{g}/\text{m}^3$ annual average and $25\ \mu\text{g}/\text{m}^3$ 24 h maximum, significantly tighter than the current US EPA goals.

This paper discusses two examples of recent applications of these IBA techniques to studies on dust emissions from open cut mining operations and the characterisation of East Asian aerosols (ACE, 2001; Huebert et al., 2003) and their effects on climate forcing.

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2. Ion beam analysis techniques

The four IBA techniques of particle induced X-ray and gamma ray emission (PIXE & PIGE), particle elastic scattering analysis (PESA) and Rutherford backscattering (RBS) have been applied throughout the work described here. The four techniques were applied simultaneously using 8 mm diameter beams of 2.6 MeV protons and target currents of typically 10–15 nA. Each of these non-destructive methods has been described in detail elsewhere (Cohen, 1993, 1998; Cohen et al., 1996) and will not be reproduced again here. Collectively these four techniques cover the commonly occurring elements H, C, N, O, F, Na, Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Sr, Y, Zr, As, Se, Br and Pb with MDL's around 1–10 ng/m³ of air sampled. There are however some elemental interferences, which may make some weaker elemental signals undetectable in the presence of stronger overlapping signals. For example, 1 ng/m³ of As is not detectable on filter papers in the presence of Pb with concentrations above 5 ng/m³.

These IBA techniques provide high sample throughput coupled with multi-elemental information allowing statistical analysis such as principal components analysis (PCA) (Hopke, 1991) to be used to determine source elemental fingerprints and then the source contributions to the total fine mass fraction to be quantitatively determined (Cohen, 1998; Cohen et al., 2000).

3. The samples

For both examples discussed here atmospheric particulate matter was collected by pulling air through a filter for 24 h; usually from midnight to midnight. Samples were generally collected twice a week on Wednesdays and Sundays. Two types of samplers were employed; a cyclone system based on the US EPA IMPROVE system which collected PM_{2.5} particles on a 25 mm diameter Teflon filter (Malm et al., 1994) and a stacked filter system, based on the GENT sampler (Maenhaut et al., 1994; Hopke et al., 1997), using two Nuclepore 47 mm diameter polycarbonate filters to collect PM_{2.5} (the fine fraction) and 2.5–10 μm diameter (the coarse fraction) particles. In these studies particulate mass in air ranged from 5–250 μg/m³ providing masses between 100 and 8000 μg per filter for analysis.

4. Open cut mining

Australia has significant mining operations, in particular there are large open-cut mining operations associated with coal mining for power in the Hunter Valley regions of New South Wales on the eastern

seaboard several hundred kilometres north of Sydney. State governments require mining companies to monitor their particulate emissions to the atmosphere. There is currently a PM₁₀ goal of 50 μg/m³ over 24 h not to be exceeded on more than 5 days per year. Hence a key question for the mining interests was, can the soil component related to open-cut mining be quantitatively extracted from the total PM₁₀ mass collected during routine sampling around a mining area? To test this two mines in the vicinity of the town of Muswellbrook in the upper Hunter Valley region, 100 km inland from the coast and 200 km north of Sydney, were selected. Twenty-four hour samples were collected twice weekly, using the GENT stacked filter sampler, from April through to September 2001 at mine site 1 and from December 2001 to March 2002 at mine site 2 (Pickett et al., 2002).

The PM₁₀ mass collected at all sites is shown in the monthly box and whisker plot of Fig. 1. The shaded box represents 25–75% of the distribution of all points, the upper and lower whiskers represent 1.5 times the distribution shown by the box and the horizontal bar in the box is the median of the distribution for that month. All points lying outside the range defined by the whiskers are plotted as outlier dots. No mass events exceeded the 50 μg/m³ PM₁₀ total mass goals set by the Australian authorities with median values ranging from 10–18 μg/m³ during the study period at both mine sites. Previous work has shown that airborne soils can be estimated from oxides of elements such as Al, Si, Ca, Ti and Fe (Malm et al., 1994). This assumed that each of these elements does not contribute significantly to any other source of airborne particulate matter and that contributions from other elements were small. To test this hypothesis we have obtained enough samples (674) to produce correlation plots of six key soil elements (Al, K, Ca, Ti, Mn and Fe) against the fundamental soil element silicon. Typical individual correlation plots for Ca and Ti against Si are shown in Figs. 2 and 3, respectively. They show how well correlated these elements are to silicon and demonstrate that they are

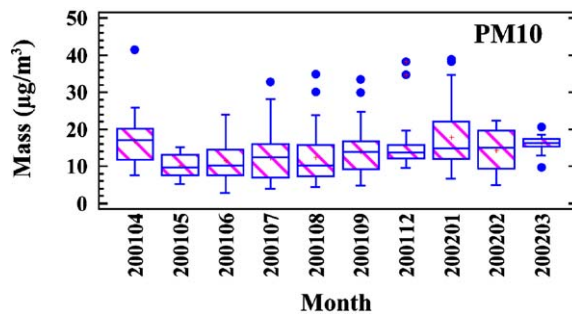


Fig. 1. Box and whisker plot of the monthly PM₁₀ mass for all eight sites operating during the study.

basically related to the one source, soil. The gradient of the best fit of each of these six elements to silicon was used to define a fractional elemental fingerprint for the local Muswellbrook soil. This fingerprint is shown in Fig. 4, the oxygen component was calculated assuming that each of these seven elements occurred in its natural oxide form. This fingerprint was unique to soils from our sampling region and was different to fingerprints

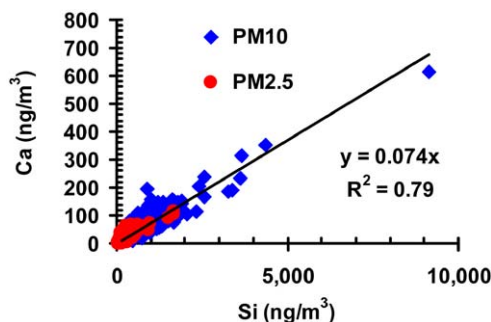


Fig. 2. Calcium versus silicon plot for PM10 and PM2.5 data for all eight sampling sites.

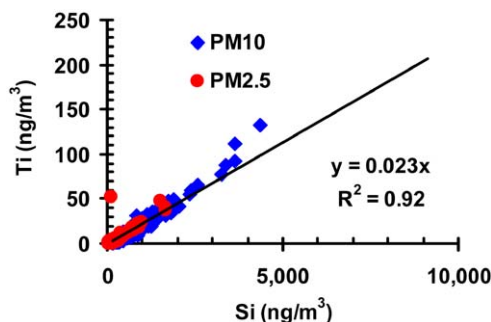


Fig. 3. Titanium versus silicon plot for PM10 and PM2.5 data for all eight sampling sites.

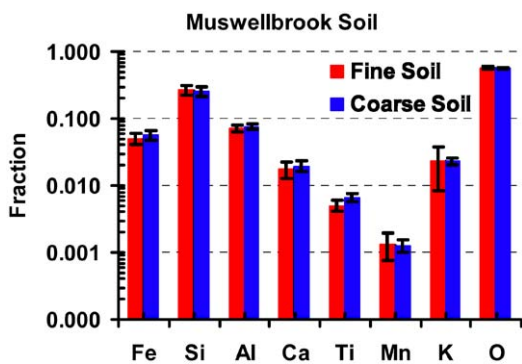


Fig. 4. Fractional elemental fingerprint for Muswellbrook airborne soils.

obtained in other sampling regions, even though the elements used may have been similar.

Fig. 5 is an estimate of the PM10 soil component of the PM10 mass given in Fig. 1 using the fingerprint fractions of Fig. 4. Clearly we could quantitatively estimate the soil component of the total mass using the IBA multi-elemental analysis techniques. Furthermore Fig. 5 shows the soil outlier events have a quite different distribution with time to the mass outlier events of Fig. 1. The mining interests could clearly see the soil contributions had median values around $5 \mu\text{g}/\text{m}^3$ but that on certain days values well above $10 \mu\text{g}/\text{m}^3$ were possible and maybe under these conditions mining operations should be minimised in order to reduce the impacts of dust emissions on nearby townships.

5. Long range transport of sea salt

During our studies of mining operations in the Muswellbrook region we also analysed all filters for elements associated with sea salt, namely Na and Cl. The sea salt estimates at Muswellbrook, an inland town, were estimated in the standard way from the Na concentrations on the PM2.5 fine fraction (Cohen et al., 2000). The fine fraction (PM2.5) from the stacked filter samplers was selected because this fraction was known to be transported large distances under appropriate weather conditions. The estimates of PM2.5 sea

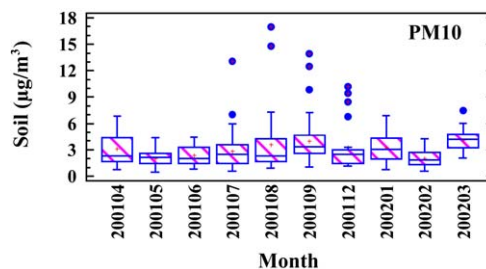


Fig. 5. Box and whisker plot of the monthly PM10 soil for all eight sites operating during the study.

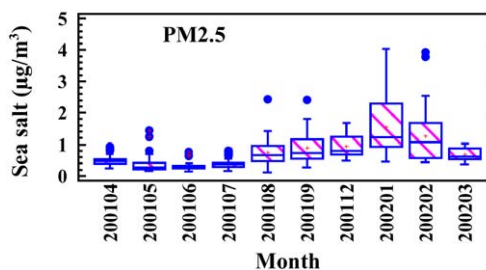


Fig. 6. Box and whisker plot of the monthly PM2.5 sea salt for all eight sites operating during the study.

salt mass at Muswellbrook are shown in Fig. 6 for the same sampling period as the open cut mining soil data. Close inspection of outlier events (dots) showed that sea salt at Muswellbrook had two possible origins. The first was the shortest and expected route and represented wind trajectories blowing directly onto the coast (from the east-south quadrant) up the Hunter Valley to Muswellbrook from the coastal City of Newcastle. This was well demonstrated by the maximum outlier event in Fig. 6, the dot shown for the 6 February 2002. Five day back trajectories (Draxler and Rolph, 2003) for the 6 February 2002, Fig. 7, show that this parcel of air originated well out into the Tasman Sea between Australia and New Zealand. It explained the high levels of sea salt measured on this day at Muswellbrook.

The other significant outlier event (dot) of Fig. 6 occurred on 19 August 2001 with PM_{2.5} sea salt concentrations well above 2 µg/m³. Similar 5-day back trajectories, shown in Fig. 8, show this sea salt event

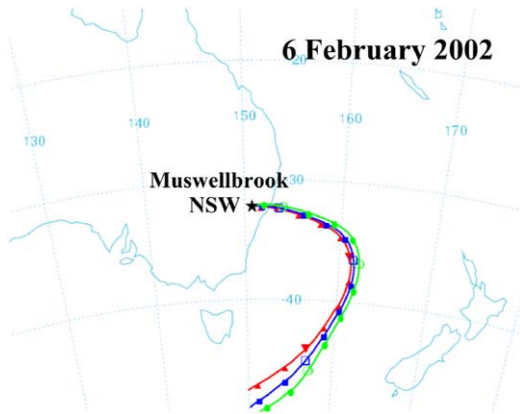


Fig. 7. Five day back trajectories from Muswellbrook site for 100, 200 and 500 m above ground for 6 February 2002.

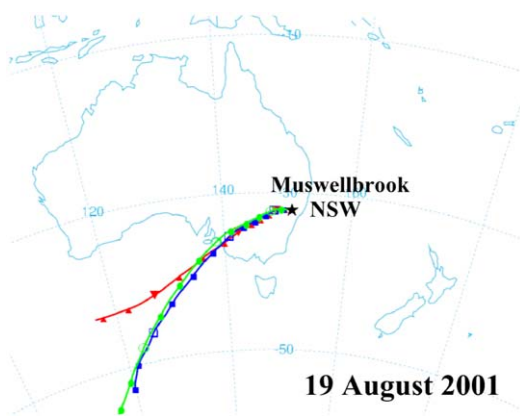


Fig. 8. Five day back trajectories for 100, 200 and 500 m above ground for 19 August 2001.

originated from the Great Australian Bight and deep in the Southern Ocean. It crossed the Australian coast just south of Adelaide, travelling 1500 km across land and reaching Muswellbrook from the west; the opposite direction to what might generally be expected. These two examples demonstrate the ability of fine (PM_{2.5}) particles to travel large distances under the appropriate wind and weather conditions.

6. ACE Asia

IBA techniques have also been applied to the study of atmospheric fine particles on a global scale in the international collaboration known as the aerosol characterisation experiment in Asia or ACE Asia (ACE, 2001; Huebert et al., 2003). One of the aims of this experiment is to quantify the regional and seasonal variations in the natural and anthropogenic fine particle atmospheric aerosols originating from eastern China and being transported into Vietnam, Hong Kong and across the ocean to Korea, Japan and even around the globe to North America.

We have been sampling and analysing PM_{2.5} (using the cyclone sampler) and PM₁₀ particulate matter (using the GENT sampler) at five ACE Asia sites since January 2001 to study large scale dust and pollution events, mainly sulphate, elemental carbon and organic matter associated with the region. These five sites were in Manila Philippines, Hong Kong China, Cheju Island South Korea, Hanoi Vietnam and Sado Island off the west coast of Japan. A map of the site locations within Asia is given in Fig. 9. The more northern sites of Cheju and Sado Islands are expected to be influenced by transport of dust from deserts in northern China and Mongolia and the more southern sites at Hanoi, Hong

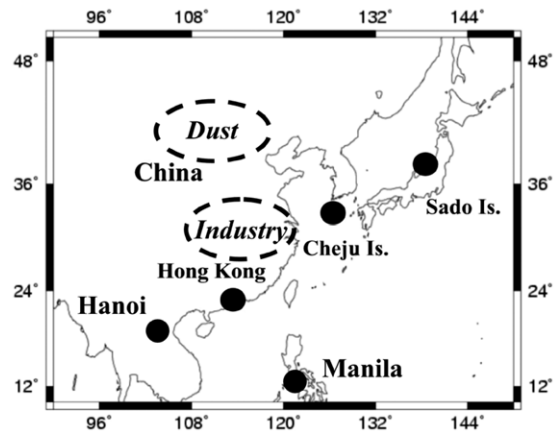


Fig. 9. ACE Asia site location map showing emissions sources for dust and industry in China.

Kong and Manila are expected to be more influenced by anthropogenic emissions from eastern and southern China. This is schematically shown by the *Dust* and *Industry* dashed oval shapes on the map of Fig. 9 roughly representing the source locations. The preliminary results have been discussed and presented in detail elsewhere and will not be repeated again here (Cheng et al., 2000; Kim et al., 2003; Cohen et al., 2002c, 2004).

Average PM_{2.5} composition of major aerosol components at the five ACE Asia sites together with their standard deviations for the sampling period from January 2001 to June 2003 inclusive are given in Table 1. The large standard deviations reflect the seasonal variations and not the IBA measurement or analysis errors, which were significantly lower at between 5% and 15% (Cohen et al., 2002b). The annual average fine masses were all above the US EPA PM_{2.5} goal of 15 µg/m³ except at the Sado Island site. At each site there were five major components making up the bulk of the fine fraction, elemental or black carbon (EltC or BC), windblown soil, ammonium sulphate, organic matter and sea salt. The sum of the mass of these five elements together with trace elements, called the reconstructed mass (RCM), accounted for between 79% and 96% of the total fine mass. This is relatively good *mass closure*, essential for meaningful source fingerprinting and apportionment calculations.

6.1. Trace and other elements

The IBA analysis techniques provided information on over 20 different chemical species in the fine mass fraction. Box and whisker plots for trace metal and other selected elemental concentrations are shown in Fig. 10(a)–(h) for all the ACE-Asia sites. Hong Kong tended to be high in Cr, Cheju and Sado Islands low in Cu, Hong Kong and Manila high in Ni and V and Hanoi high in Pb and Zn. As discussed later below, Ni and V were probably associated with diesel or oil burning and Zn and Pb with automobiles emissions. The parameter K_{smoke} was an estimate of the fine potassium associated with smoke from biomass burning and was

calculated by subtracting the potassium associated with soil and sea salt from the measured total potassium values. The Hanoi site was the most influenced by smoke from biomass burning with Hong Kong also having a significant number of outlier smoke events.

6.2. Dust events

Using the same seven elements and techniques described above a unique soil fingerprint was obtained for all ACE Asia sites (Cohen et al., 2002c, 2004). Using this fingerprint we have calculated the soil contribution to the total PM_{2.5} mass at the Sado Island site off the west coast of Japan. This is our site furthest from the sources of desert dust in northern China (see Fig. 9). Fig. 11 is a plot of the total PM_{2.5} mass at Sado against the number of days starting from 1 January 2001. The figure contains two horizontal dashed lines one at 15 µg/m³ and the other at 65 µg/m³ representing the current US EPA PM_{2.5} health goals. The numbered peaks correspond to significant PM_{2.5} mass events (days 434 = 10Mar02, 644 = 6Oct02, 815 = 26Mar03, 875 = 25May03) at the Sado site during the study period. Fig. 12 shows the estimated soil component at Sado Island for the same period as for Fig. 11. Clearly the events of day 434 (10 March 2002) had a significant soil component and back trajectories (see Fig. 13) confirm northern China/Mongolia as the origin. Also days 693 (24 November 2002) and 833 (13 April 2003) were major soil events at Sado but not a major mass events. The mass events on days 815 (26 March 2003) and 875 (25 May 2003) did not have significant soil components. Similar time series plots for other elements can help identify the composition of other non-soil events at Sado. For example, Fig. 14 is the plot for sulphur at Sado it shows that the high mass event of day 644 (6 October 2002) as well as being a relatively high soil day was also a high sulphate event at Sado. Back trajectories for 6 October 2002 are shown in Fig. 15 and show the origin of this event to be connected with emissions from mainland Japan and not eastern China.

Table 1

Average fine particle composition at the five ACE Asia sites for all good data collected between January 2001 and June 2003 inclusive

PM _{2.5} (µg/m ³)	Manila	Hong Kong	Cheju Island	Hanoi	Sado Island
Mass	42.2 ± 16	26.4 ± 15	19.0 ± 13	37.1 ± 20	13.2 ± 10
Elemental carbon	12.7 ± 5	1.9 ± 0.9	1.3 ± 1	3.6 ± 1	0.89 ± 0.5
Soil	11.8 ± 7	1.9 ± 2	1.7 ± 3	3.5 ± 3	1.0 ± 2
Ammonium sulphate	6.5 ± 4	11.9 ± 6	7.5 ± 4	9.7 ± 5	5.4 ± 4
Organics	19.4 ± 8	4.2 ± 6	3.6 ± 5	11.5 ± 7	2.1 ± 3
Sea salt	1.7 ± 1	2.3 ± 2	1.4 ± 1	1.8 ± 3	1.4 ± 1
Traces	0.24 ± 0.2	0.21 ± 0.2	0.91 ± 0.8	0.51 ± 0.4	0.06 ± 0.05
Number of samples	224	233	184	169	147
%RCM	96 ± 11	85 ± 15	84 ± 14	79 ± 9	79 ± 13

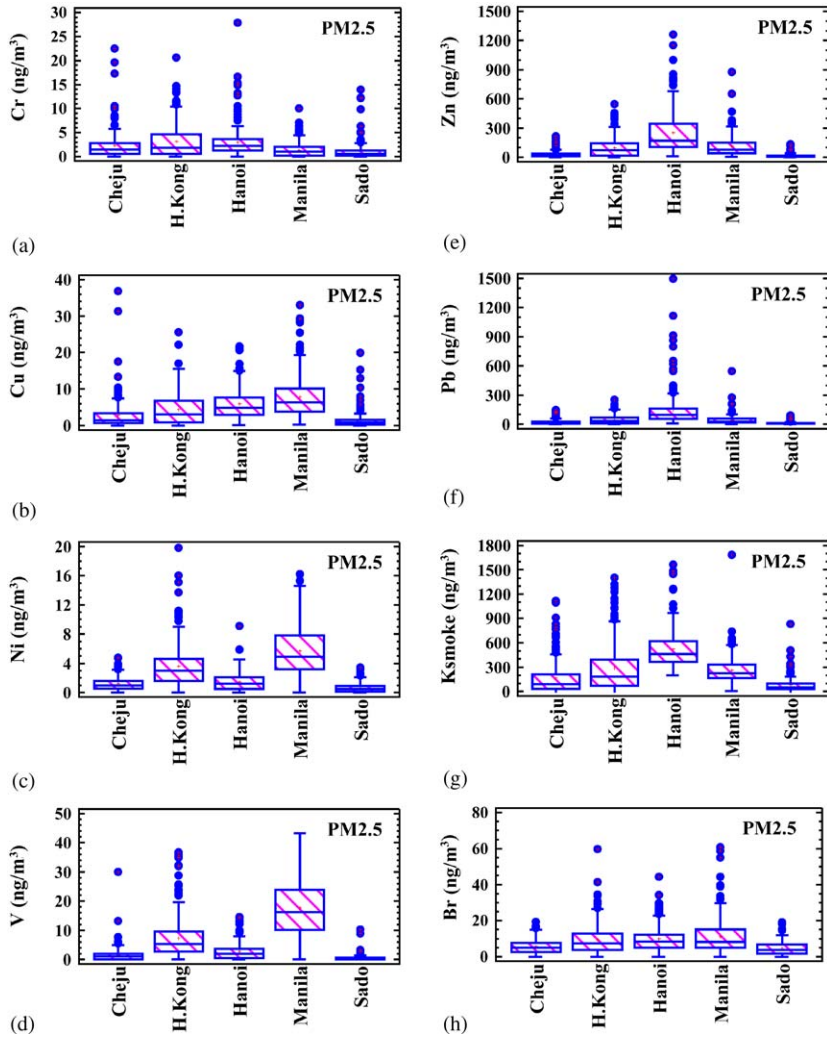


Fig. 10. (a–h) Trace element box and whisker plots for the 5 ACE Asia sites during the study period.

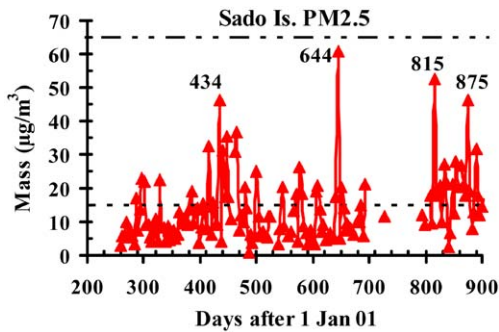


Fig. 11. Measured PM2.5 mass at Sado Island from April 2001 (day 100) to 30 June 2003 (day 910).

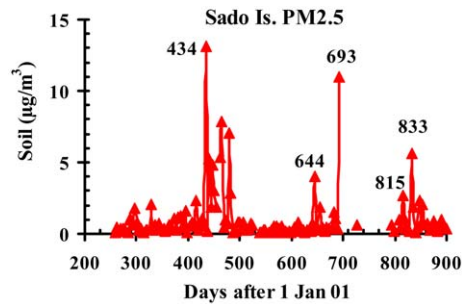


Fig. 12. Estimated PM2.5 soil at Sado Island from April 2001 (day 100) to 30 June 2003 (day 910).

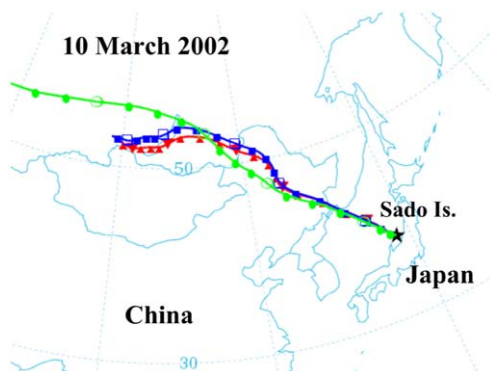


Fig. 13. Five day back trajectories from Sado Island site for 100, 200 and 500 m above ground for 10 March 2002.

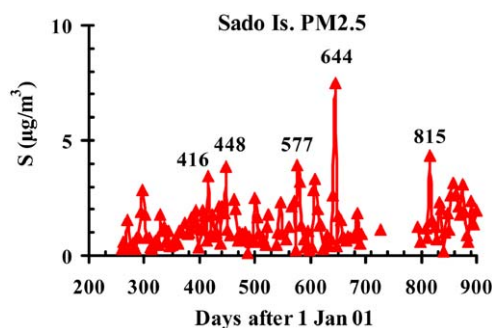


Fig. 14. Measured PM2.5 sulphur at Sado Island from April 2001 (day 100) to 30 June 2003 (day 910).

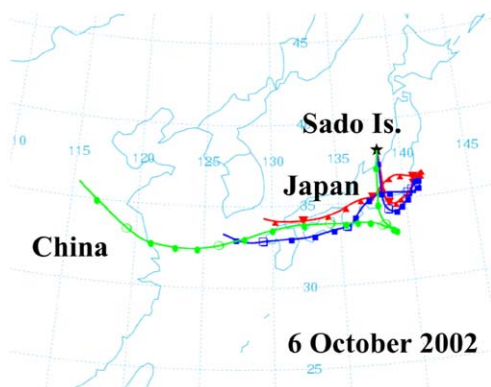


Fig. 15. Five day back trajectories from Sado Island site for 100, 200 and 500 m above ground for 6 October 2002.

6.3. Statistical analysis

Elements from H to Pb have been measured on hundreds of filters from the five ACE Asia sites. This large data matrix lends itself to impartial techniques like

Factor Analysis or Principal Components Analysis (PCA) to extract source element associations in a more rigorous statistical way (Hopke, 1991). PCA methods applied to all 957 ACE Asia data points for the entire study period required six factors to explain 84% of the variance. These factors and their corresponding factor loadings are plotted against each other in Figs. 16(a)–(d). These PCA methods reduced the multi-dimensional space of dozens of elements to a more manageable six dimensional factor space with several key elements associated with each factor. For example, Factor 1, which accounts for 42% of the total sample variance was clearly a *Soil* factor having high

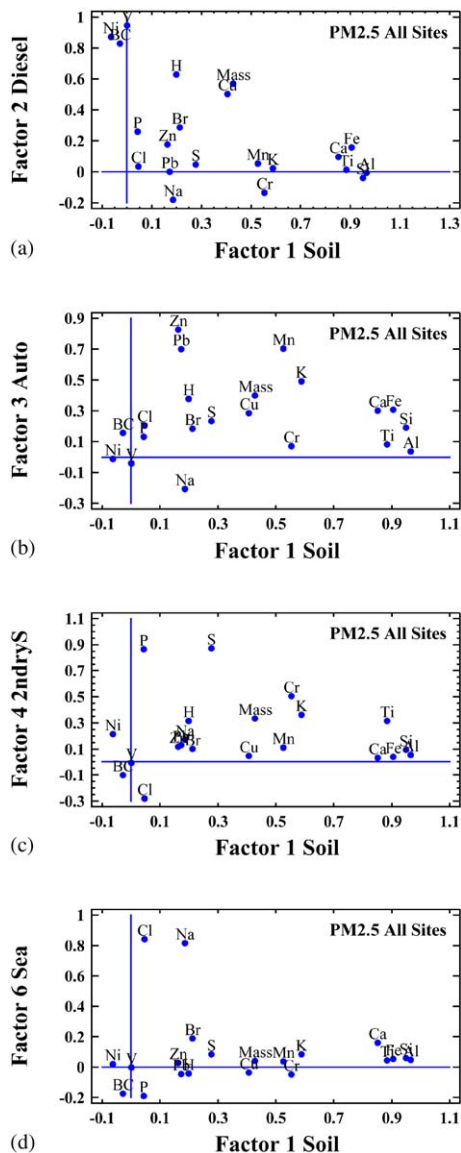


Fig. 16. (a–d) Plots of PCA factor loadings for data from all the ACE Asia sites to 30 June 2003.

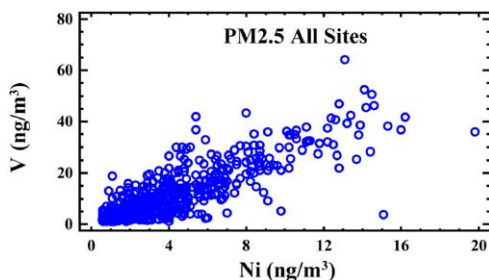


Fig. 17. Plot of V vs. Ni for all ACE Asia PM2.5 data to 30 June 2003.

factor loadings (>0.7) for Ca, Fe, Ti, Si and Al. Factor 2, named *Diesel*, was high in Ni, V, Black carbon (BC), and H from organic matter and accounted for 17% of the total sample variance. It was most probably associated with oil and fossil fuel combustion associated with power production and diesel motor vehicles. The third factor, which was probably also associated with motor vehicles, named *Auto*, was high in Zn, Pb and Mn and accounted for about 9% of the total sample variance. The remaining three factors were named secondary sulphate, industry and sea spray after inspection of their key elemental associations and accounted for 7%, 6% and 4% of the total sample variance, respectively.

The fact that PCA techniques impartially identify inter-element associations has been well demonstrated by Figs. 2 and 3 is further evidenced by plots like Fig. 17 where Ni and V with high factor loadings for the *Diesel* Factor 2 are plotted. The correlations were reasonable with (V:Ni) ratio around (2:1).

7. Summary

Ion beam analysis techniques have been shown to be appropriate methods for characterizing fine particulate matter collected on polycarbonate and Teflon filter papers. The broad range of elements and the large number of samples analysed provided a large data set that was able to be analysed using impartial statistical techniques like principal components analysis. Having identified inter-elemental associations quantitative anthropogenic and natural fine particle source fingerprints were obtained. Examples of dust from open cut mining operations and from the desert regions of northern China were given to demonstrate the power of these IBA techniques.

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