Sampling and characterization of resuspended and respirable road dust

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Abstract

Urban air quality is severely affected by traffic related particulate matter, including direct emissions from exhaust, brake pad, tire wear and road dust resuspended by vehicular motions. Deposited road dust can also be resuspended by wind force or other anthropogenic activities, and overall it may contribute up to 30% to urban PM10. A mobile resuspended road dust PM10 sampler was developed and constructed which simulates the effects of traffic or gusting winds on road surfaces and collects resuspended PM10 samples in a cyclone separator and PM1 samples on filters. The sampler was tested by collecting resuspended road dust at kerbside locations in Veszprém, Hungary. The collected PM1 and PM10 fractions were analysed by various analytical methods to show the potential of size-selective on-line sample collection combined with the chemical characterization of resuspended road dust. The main constituents of the resuspended road dust were crustal elements, and it was also possible to determine the mineral phase composition of PM10 dust which is generally not feasible from samples collected on filter substrate. The application of the sampling and analysis methods may facilitate the evaluation of resuspended road dust sources in cities as well as help constrain a better source apportionment of urban PM10.

1. Introduction

Air pollution in cities is a very complex issue, showing strong seasonality and dependence on meteorological factors, sometimes culminating in severe and dangerous smog episodes which require intervention by local authorities. The urban particulate matter with aerodynamic diameter less than 10 µm (PM10) is now identified as one of the most dangerous air pollutants on human health because its size range overlaps with the range of respirable particles. Prolonged exposure to respirable urban PM10 has been associated with adverse health effects and linked to an increased risk of respiratory illnesses (Pope, 1996).

Resuspension of road dust is now an established source of urban particulate matter. Fugitive dust from paved and unpaved roads and bare grounds as well as construction and demolition works was shown to be important contributors to
both PM$_{10}$ and PM$_{2.5}$ in urban aerosol (Chow & Watson, 2002). A source apportionment study based on elemental composition of particles using Al as a tracer for crustal matter estimated a total contribution of 10% (m/m) in PM$_{10}$ and 6% in PM$_{2.5}$ in Hong Kong (Ho et al., 2003). On the other hand, in Mexico City fugitive dust was estimated to account for as much as 54% (m/m) of PM$_{10}$ (Vega et al., 2001). Its contribution strongly depends on local conditions, including meteorology, road surface, traffic, etc.

In addition, dust particles preserve cumulative signatures of particles that had once been airborne and deposited from the atmosphere at the location of sampling. Its detailed analysis may provide qualitative and quantitative information on the significance of various local and regional aerosol sources. Fugitive dust sources are extremely difficult to measure and quantify in urban air due to their diffuse nature and strong dependence on sampling conditions (Cowherd & Grelinger, 1992; Watson & Chow, 2001).

Detailed source profile studies on this fraction of aerosol that can be used for receptor modeling is known (Ho et al., 2003; Vega et al., 2001; Amato et al., 2010). The mass size distribution of primary particles of fugitive dust is poorly characterized. Scanning electron microscopic examination of PM$_{10}$ particles in Shanghai showed that most of particles were originally released from construction sites, cement plants, vehicle exhaust, coal boilers and steel mills (Li et al., 2003). Such studies typically used the source type “paved road dust” as a collective term for all types of urban dust particles, including freshly emitted or resuspended particles, without regard to their ultimate origin (Schauer et al., 1996).

Sampling of resuspended urban particulate matter has usually been performed by simply sweeping and sieving deposited road dust (Wei et al., 2009; Han et al., 2007; Manno et al., 2006; Ho et al., 2003). In a few studies the collected bulk samples were resuspended in the laboratory and the size-distribution of the particles was determined (Zhao et al., 2006; Chow et al., 2004). These procedures and sample treatments were affected by the loss of fine particles owing to the difficulties of collecting all deposited material and to the electrostatic adhesion of particles to brush hairs and sieve meshes. A recent study has shown that aerosol particles can be quantitatively resuspended from road dust using a specific sampling device (Amato et al., 2009). The objective of this study is to develop and test a mobile sampler that simulates the effect of strong winds and heavy traffic and on-line collects the resuspended PM$_{10}$ particles. Furthermore the potential of various analytical methods are evaluated in the use of the samples for source profiling of resuspended road dust in source apportionment of urban PM$_{10}$.

2. Material and methods

2.1. Resuspension and sampling unit

This work involved the development and construction of a mobile resuspended road dust PM$_{10}$ sampler which induces resuspension and collects particles on-line directly from road surfaces (see Fig. 1). A rectangular stainless-steel hood (length: 600 mm, width: 400 mm, height: 170 mm) was fixed to the front of a laboratory cart 0.5 cm above the road surface. A leaf blower (Makita UB1101, 600 W) was connected to the hood through two facing nozzles at an angle of 65° (of 30° correlates
to the surface) via a split flexible hose. This setup was designed to induce turbulence inside the hood which can resuspend a part of the road dust from solid surfaces. In this configuration the system is not air-tight, but there is a constant outflow from under the hood inside which the mass concentration of resuspended road dust is much higher (by a factor of 1000) than that of PM$_{10}$ in ambient air. The air velocity was measured at the nozzles with an anemometer, and was found to be $\sim 65$ km hr$^{-1}$. The hood was connected to the duct system with a stainless steel tube (length: 200 mm, diameter: 100 mm). The duct system (length: 1.8 m, internal diameter: 30 mm) was designed for an air velocity in the vertical duct section at which particles larger than $\sim 110$ $\mu$m aerodynamic diameter can effectively settle out by gravitational settling and do not overload the PM$_{10}$ sampling head of the sampler. The duct was connected to an alumina housing (height: 350 mm, diameter: 350 mm) containing the R&P PM$_{10}$ inlet. This housing was composed of two parts to facilitate cleaning. The height of the bottom part was 100 mm and that of the upper one was 250 mm, the diameter of this housing was 350 mm. During sampling the two parts was closed hermetically, the volume of the housing was approximately 24.5 dm$^3$.

A PARTISOL-FRM MODEL 2000 sampler (operating at the flow rate of 16.7 l min$^{-1}$) was mounted on a mobile sampling cart. This sampler collected resuspended PM$_{1-10}$ samples in a cyclone separator and PM$_1$ samples on filters. The sampling unit was powered with a portable electrical power generator (Honda EU10i), also mounted on the platform. The mobile sampling unit has been successfully deployed on the site of the catastrophic red mud spillage near Ajka-Kolontár, Hungary (Gelencsér et al., 2011).

### 2.2. Sample transfer and gravimetric determination

The PM$_1$ fraction was collected on 47 mm diameter Whatman quartz filter, and before and after sampling filters were weighted with a microbalance (Sartorius, 10 $\mu$g sensitivity). The PM$_{1-10}$ fraction was deposited in the sharp-cut cyclone-separator of the PARTISOL sampler. The collected dust samples were transferred with a PTFE-coated spatula into pre-weighted clean vials. Then the vials were weighted again with a microbalance. The total mass of the PM$_{1-10}$ fraction was determined by difference. It should be noted that though sample collection from the surface is not quantitative, mass concentration data above the surface during the sampling can be derived for comparative purposes. The resuspended road dust samples were tagged and stored in the freezer until analysis. The collected PM$_{1-10}$ fractions were analysed by various analytical methods to establish chemical composition, to identify major source types and to assess the potential contribution of major sources to deposited dust.

### 2.3. Chemical analyses

The direct determination of phase composition of particulate matter has not generally been available in ambient aerosol studies due to limited sample size and the presence of a filter matrix. However, quantification of phase composition would be an invaluable supplement to source apportionment studies of specific aerosol types such as resuspended road dust. The X-ray diffraction (XRD) analyses were carried out with a Philips PW 3710 type diffractometer equipped with a PW 3020 vertical goniometer and curved graphite diffracted beam monochromator. The radiation applied was CuKa from a broad focus Cu tube, operating at 50 kV and 40 mA. The samples were measured in continuous scan mode with 0.02 $^\circ$ sec$^{-1}$ scanning speed. Each small powder sample (approximately 7 mg) was deposited on a glass plate from a slurry of the sample in isopropyl alcohol (Bish & Post, 1989). The quantitative determination was carried out with Rietveld analysis which is a full-pattern fitting method. In this method the measured diffraction profile and the calculated profile are compared, and the difference is minimized. Crystal structure data, such as atom positions are necessary to the standard Rietveld refinement. In addition the scale factors calculated for the mixture of phases are proportional to weight fractions.

Due to the limited amounts of resuspended road dust samples particle morphologies were studied and elemental composition (of elements with $Z > 5$) were determined by environmental scanning electron microscopy (ESEM, Philips XL30) with an attached EDAX energy-dispersive X-ray spectrometer (EDS). ESEM was operated at 20 kV accelerating voltage in low-vacuum environmental mode due to the non-conductive character of the samples. PM$_{1-10}$ powder samples were sprinkled on cylindrical Al stubs covered by double coated conductive carbon tape. Determination of mean elemental composition (except C) was performed on 3 different areas ($200 \times 200 \mu$m) of each prepared sample. Secondary electron (SE) images of hundreds of particles in each PM$_{1-10}$ fraction were taken to characterize of particle sizes and morphologies.

The total carbon (TC) concentration of PM$_{1-10}$ samples were analysed by high-temperature catalytic oxidation, using Zellweger Astro TOC 2100 total carbon analyzer with NDIR detector. The TC was determined by catalytic burning at 680 $^\circ$C in oxygen followed by non-dispersive infrared detection of the evolved CO$_2$. Approximately 0.5 mg of bulk dust samples were oxidized in the combustion boat. Calibration of the instrument was performed with reagent-grade potassium hydrogen phthalate (VWR International) dissolved in high-purity water (MilliQ).

The water-soluble inorganic ions (Na$^+$, NH$_4^+$, K$^+$, Mg$^{2+}$, Ca$^{2+}$, Cl$^-$, NO$_3^-$ and SO$_4^{2-}$) were measured by Dionex DX300 gradient chromatographic system that consists of a CHA-6 high pressure chromatographic module, Dionex EDM eluent degas module and gradient pump equipped with a conductivity detector CDM-II. Separations were carried out by a Dionex IonPac AS12A column for anions and a Dionex IonPac CS12A column for cations. The conductivity of the eluent was suppressed by a Dionex CSRS 300 4 mm suppressor. Approximately 1 mg samples were extracted with 1 ml high-purity water. The detection limit of each ion was 10 ppb.
A series of 16 polycyclic aromatic hydrocarbons (PAHs) regulated by the US Environmental Protection Agency (US-EPA) were analysed. PAHs were determined by liquid chromatography with Jasco PU-980 pump, Jasco LG-980-02 gradient unit and Waters 470 scanning fluorescence detector. Separations were carried out by a LiChrospher PAH 5 μm column with RP18 silica gel charge, and its temperature was held at 35 °C with a Waters thermostat. Approximately 6 mg dust samples were extracted with 1 ml of dichloromethane:methanol (80:20) solvent mixture. Extraction was performed in an ultrasonic bath for 20 min. The extracts were filtered by 0.22 Gv filters. The injection volume was 20 μl. The calibration was performed with TCL polynuclear aromatic hydrocarbons mix (SUPELCO) dissolved in acetonitrile:methanol (9:1) solvent. The standard solution containing the 16 EPA priority pollutant PAHs: naphthalene (Nap), acenaphthylen (Acy), acenaphthene (Ace), fluorine (Fl), phenanthrene (Phe), anthracene (Ant), fluoranthene (Fla), pyrene (Py), benz[a]anthracene (BaA), chryseene (Chry), benzo[b]fluoranthene (BbF), benzo[k]fluoranthene (BkF), benzo[a]pyrene (BaP), ideno[1,2,3-cd]pyrene (IndP), dibenz[a,h]anthracene (dBaAnt) and benzo[g,h,i]perylene (BghiPer). 15 PAHs (exception is acenaphthylene) were detected with fluorescence detector.

The TC and PAHs concentrations of PM10 fractions on quartz fiber filters were also determined with the above methods. TC concentrations were analyzed from filter spots of 10 mm in diameter which were cut with a special puncher and were oxidized directly in the combustion boat. PAHs concentrations were measured from the remaining filter samples. Other measurements were not carried out due to the limited sample size and the presence of the filter matrix.

3. Testing

3.1. Sampling

The sampling unit was tested by collecting resuspended particulate matter at three kerbside locations in Veszprém, Hungary. Veszprém lies approximately 110 km far from the capital of Hungary, Budapest. The city is lying in the embrace of Lake Balaton and the Bakony Hills, on both sides of the Séd creek. Veszprém has about 60 thousands inhabitants, its climate is continental. The daily average temperature is 20 °C in summer and −1 °C in winter.

Each sampling involved collection of resuspended road dust from a sampling area of about 840 m2. The samples were collected on the sidewalks ~2 m from the roads for a period of 2 h each. The sampling was carried out on three consecutive days between 22 and 24 September 2010. The sampling locations were characterized with high traffic of passenger cars and busses, but heavy-duty trucks were not allowed. The speed limit is 50 km h⁻¹.

The meteorological and air quality parameters before and during sampling days were recorded by the local meteorological station of Veszprém and by the Hungarian Air Quality Monitoring Network. During this period the daily mean temperature varied between 12 and 18 °C. The sampling took place three days after a rainy period between 16 and 19 September, under generally dry conditions. On the days of sampling the daily mean PM10 concentrations were 28.4, 29.2 μg m⁻³ and absent data on 24 September, and the NOx concentrations were 62.6, 60.7 and 43.1 μg m⁻³.

3.2. Mass proportion of resuspended road dust

Even if the collection of resuspended PM10 is not quantitative, a surface load of resuspended PM10 can be estimated from the measured data by taking into account the flow rate of the blower and that of the PM10 sampler. The estimated surface load of PM10 was found to be 3.4–4.9 mg m⁻² at the three sites. This value compares well with surface loads estimated from measured emissions caused by passing vehicles (0.3–3 mg m⁻²) (Etyemezian et al., 2003). The absolute mass concentrations of the PM10 and PM1 fractions inside the sampling hood were determined by weighted masses on filters and in vials. The mass concentration of the PM10 was 15.4–19.6 mg m⁻³ and of the PM1 fraction was 0.2–2.7 mg m⁻³ (Table 1).

The PM1 fraction constituted on average 9.3% of the PM10 fraction in the resuspended road dust which compares well with the mass ratio of fine (PM2.5) to total (PM10) particles found in soil and paved road dust (10–30%) (Ho et al., 2003). In addition the particulate matter with aerodynamic diameter less than 1 μm (PM1) was sampled directly and was much less subject to losses during sampling.

Table 1

<table>
<thead>
<tr>
<th>Code</th>
<th>Location</th>
<th>GPS</th>
<th>Date</th>
<th>Absolute mass concentrations</th>
<th>PM10 [mg m⁻³]</th>
<th>PM1 [mg m⁻³]</th>
<th>Surface loads of PM10 [mg m⁻²]</th>
</tr>
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<td>22 Sept.</td>
<td>36.35</td>
<td>5.12</td>
<td>18.2</td>
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<td>23 Sept.</td>
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<td>5.36</td>
<td>19.6</td>
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<tr>
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<td>0.46</td>
<td>15.4</td>
<td>0.23</td>
</tr>
</tbody>
</table>
**Fig. 2.** The mineral phase composition of resuspended road dust (m/m%).

**Fig. 3.** SEM images of the PM$_{1-10}$ fraction of resuspended road dust. (a) Sample 1, (b) sample 2, and (c) sample 3. The particles form loose aggregates. White arrows indicate euhedral crystals with rounded edges.
3.3. Phase composition

In addition to source profiling based on elemental composition the direct determination of mineral phase composition is also feasible from resuspended bulk PM$_{1-10}$ road dust samples. Similarly to the quantification of oxygen, this is also not available on dust samples collected on filter substrates. The mineral phase composition could, on one hand, be used to cross-check mass balance calculations based on elemental compositions. But more importantly, it can be used in the source identification of resuspended road dust itself, as thermally altered phases can be directly identified and quantified by this method. Moreover, by comparing mineral phase compositions of resuspended native soils in the areas, building materials resuspended road dust may also be possible to estimate the relative contribution of demolition and construction works to the resuspended road dust concentrations in cities.

In the resuspended bulk PM$_{1-10}$ road dust samples the major mineral phases identified were dolomite [CaMg(CO$_3$)$_2$], calcite [CaCO$_3$], clinocllore [[(Mg,Fe)$_6$(Si,Al)$_4$O$_{10}$(OH)$_8$]], quartz [SiO$_2$], muscovite [KAl$_2$Si$_3$O$_{10}$(OH)$_2$], albite [NaAlSi$_3$O$_8$], orthoclase [KAlSi$_3$O$_8$] and gypsum [CaSO$_4$·2H$_2$O], in the mass ratios shown in Fig. 2.

The main mineral phase in the resuspended road dust is dolomite which forms the rock bed in the entire region around Veszprém. There are dolomite mines in operation upwind the town of Veszprém and all the unpaved roads in the region are covered with ground dolomite. It is therefore not surprising that the predominant mineral phase in the resuspended road dust is dolomite. Calcite, quartz and gypsum are much less related to the phase composition of local soils, they mostly come from industry, and building activities such as construction and demolition works. The other mineral phases (clinocllore, albite, orthoclase and muscovite) in lower amounts indicate the crustal origin, originate from the regional soils or abrasion of surfaces.

3.4. Morphologies of individual particles

The morphologies of individual particles in the PM$_{1-10}$ fraction are similar in each sample as observed in the SEM. Most of the particles have irregular shape, their geometric sizes vary between 1 and 5 μm. The smallest particles form fluffy aggregates and, the larger ones also appear as loose aggregates. There are some individual grains which have nearly euhedral crystal shape. Although the original crystal shape has been eroded by the weathering process as it is manifested in the rounded edges and growth terraces some crystals have rhombohedral shape. This shape is typical for carbonate minerals which form the main mineral phase of the resuspended PM$_{1-10}$ based on the XRD measurements Fig. 3.

3.5. Chemical composition

Due to the limited sample amount the elemental composition of resuspended PM$_{1-10}$ was determined by EDS and TC analyses. The mass concentrations of the identified elements are given in units of mg g$^{-1}$ (in Table 2). The main constituents of the PM$_{1-10}$ fraction were O, Si, Ca, C, Al, Mg, Fe, S, K and Na. Crustal elements such as Si, Al, Ca, Fe have been identified in other studies (Vega et al., 2001; Zhao et al., 2006) as important constituents of the resuspended dust, but collection and analyses of bulk dust samples offers the possibility of measuring concentrations of oxygen which is generally not feasible in the analyses of filter samples. Direct measurement of mass concentration of oxygen can be a useful complement of mass balance calculations necessary for source apportionment studies. Furthermore, the sensitivity of the method and the lack of filter matrix allow the determination of less abundant elements. For example, in the resuspended PM$_{1-10}$ samples P, Cl and Ti were also quantified. Titanium is known to result from resuspension of soil and road pavement erosion (Amato et al., 2009; Guo et al., 2009). The contribution of phosphorus in the resuspended PM$_{1-10}$ was significant compared to its mass proportion in the PM$_{2-10}$ fraction of urban aerosol (0.08 mg/m$^3$) (Salma et al., 2002). It might be possible that phosphorous originate from microbiological activities taking place in the deposited dust and thus might serve as a tracer for resuspended dust. Such hypotheses, however, require further targeted studies and are beyond the scope of the present study. It should be pointed out that the suggested methods of sample collection and analyses open the possibility of better source profiling of resuspended road dust component for urban PM$_{10}$ studies.

The average TC concentration of PM$_{1}$ fraction was 114.4 mg g$^{-1}$ (standard deviation 14.68 mg g$^{-1}$) which may be influenced by soot particles from vehicular emission.

As part of routine aerosol analyses water-soluble inorganic ions can also be determined in the bulk PM$_{1-10}$ resuspended road dust samples. This feature is also available for PM$_{10}$ samples collected on filter substrate. Note that the analyses of the bulk samples yields concentrations in units of mg g$^{-1}$ which allows source profiles to be established. It is generally not expected that water-soluble ions would be predominant components of resuspended road dust. The concentrations of the water-soluble inorganic ions in PM$_{1-10}$ fractions are shown in Table 2.

Similarly, as part of the routine aerosol analyses, PAHs can also be determined in the resuspended bulk PM$_{1-10}$ and PM$_{1}$ fractions on filter. The concentrations of PAHs are given in units of μg g$^{-1}$ (in Table 2). The average mass concentration in the PM$_{1-10}$ fraction was 2.38 μg g$^{-1}$ (standard deviation 1.02 μg g$^{-1}$), and in the PM$_{1}$ fraction was 16.83 μg g$^{-1}$ (standard deviation 16.34 μg g$^{-1}$). It may be expected that as a result of cumulative deposition and the proximity of tailpipe emission sources, PAHs that are characteristic of vehicular emission (Diesel or gasoline) will be enriched in the PM$_{1-10}$ fraction of road dust relative to that...
in the coarse fraction of urban particulate matter. This may increase the health risk associated with the inhalation of resuspended road dust, but may also provide a tool for fingerprinting resuspended road dust in the coarse fraction of urban PM10.

4. Conclusions

A novel method for direct sampling of resuspended and respirable urban particulate matter has been developed and tested successfully. Although the collection method is not designed to be quantitative, it simulates the possible effects of environmental factors on road dust resuspension. Therefore it is capable of producing a representative aliquot of resuspended PM10 in two size bins (PM1–10 and PM1). The use of sharp-cut cyclone separator allows the collection of bulk particulate matter in the PM1–10 fraction. The availability of bulk PM1–10 facilitates the application of low-cost analytical methods to determine the elemental, chemical and phase composition of resuspended particulate matter, as it has been demonstrated in this study.

Contrary to other indirect methods of resuspended road dust characterization, such as brushing, sieving or laboratory resuspension of collected bulk particulate matter, the methodology presented may offer unbiased collection and characterization of resuspended PM10. The determination of the chemical and phase composition of the samples offers the possibility of more reliable source apportionment of resuspended urban PM10 that poses a potential health risk for the population. Furthermore, chemical source profiling of this poorly characterized fugitive source type is also possible by this methodology. Chemical source profiles are indispensable for reliable source apportionment studies of urban PM10 that serve

Table 2
Chemical composition of PM1–10 and PM1 fractions of resuspended road dust.

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LOD: limit of detection, LOQ: limit of quantification.
as input to air quality management in cities. In addition, planned developments in automobile industry aiming at reducing non-exhaust (fugitive) emissions from vehicles may also benefit from the application of the proposed methodology.

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References


