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ORIGINAL ARTICLE

Investigation of the pollution in the street dust at Xuzhou, China, using magnetic, micro-morphological and Mössbauer spectra analyses

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Abstract This study reports a detailed examination of the magnetic mineralogy of street dust samples collected in Xuzhou, a heavily industrial city in China. Several different approaches have been utilized in the characterization, from standard mineral magnetic techniques such as magnetic hysteresis and microscopy to non-standard techniques such as Mössbauer spectroscopy. Magnetic properties and the concentrations of four metals including Cu, Zn, Ba and Pb were reported for each sample. The primary magnetic mineral was found to be multidomain magnetite-like material. Scanning electron microscope analyses of street dust revealed: spherule-shaped iron oxides and angular particles with complex chemical composition. The concentrations of Cu, Zn, Pb and Ba were 3, 4.8, 2.8 and 1.3 times higher, respectively, than the soil background values of this study area. Both Cu and Ba showed significant correlations with the magnetic concentration parameters χ_{ARM} (susceptibility of anhysteretic remanent magnetization) and saturation isothermal remanent magnetization. The combination of the various methods offers a powerful analytical technique in the study of magnetic properties of street dust.

Keywords Environmental magnetism · Geochemical analyses · Street dust · Xuzhou city

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Introduction

Magnetic particulate matter (MPM) is not only dangerous in itself due to iron's and iron oxides' catalytic nature, but it is also associated with other hazardous pollutants emitted into the atmosphere during combustion (Muxworthy et al. 2002). Traffic is considered to be one of the major sources of MPM in urban area. Several recent studies have suggested that the influence of traffic is accompanied by significant emissions of strongly magnetic, iron-containing particles (Bucko et al. 2011, 2013; Kim et al. 2007, 2009; Wang et al. 2012). Apart from direct combustion-produced particles, wear of tires and brakes is another important source of magnetic particles (Kim et al. 2007; Bucko et al. 2011).

In addition to magnetic methods, Mössbauer spectroscopy technology is frequently applied to characterize the iron speciation. Muxworthy et al. (2002) applied Mössbauer spectroscopy and magnetic measurements to characterize urban atmospheric particulate matter collected in Munich (Germany) and found the primary magnetic minerals were maghemite and metallic iron in their samples. Baghdadi et al. (2012) applied Mössbauer spectroscopy to characterize the iron speciation in roadside soil and found the relative abundance of magnetite $Fe^{2.5+}$, magnetite Fe^{3+} and hematite was 10.9, 39.5 and 35.3 %, respectively. Wang (2014) used Mössbauer measurement to study the iron speciation of fly ash.

Street dust is an important pathway of pollution material in the urban environment, acting as a sink for vehicle exhaust and as a source of atmospheric PM (Xie et al. 1999; Kim et al. 2009). Human's health risks due to street dust, especially for children, were reported (Harrison and Yin 2000). Several studies of street dust have mainly focused on elemental concentrations and source identification (De Miguel et al. 1997; Xie et al. 2001; Sutherland 2003; Adachi and Tainosho 2004). The magnetic properties of urban dust collected directly from streets have been studied by several workers (Xie et al. 1999; Shilton et al. 2005; Kim et al. 2009; Wang et al. 2012).

Xuzhou, located in Eastern China, is a densely populated city with heavy traffic loads and major industrial plants within the metropolitan area. In the present study, we combined X-ray diffraction (XRD), environmental scanning electron microscopy (SEM) and Mössbauer spectroscopy (MS) and magnetic results in a detailed characterization of magnetic properties of street dust collected in Xuzhou, China. The concentrations of 4 heavy metals including Cu, Zn, Pb and Ba in street dust samples were also determined.

Materials and methods

Sample collection

Street dust samples were collected in Xuzhou (China) according to the procedure applied by Adachi and Tainosho (2004). A total of 49 street dust samples (Fig. 1) were collected within the city of Xuzhou (China). At each sampling site, three subsamples were taken and then mixed to obtain a bulk sample.

XRD, SEM–EDS and Mössbauer spectroscopy

Magnetic extracts were isolated from selected street dust samples by wet magnetic method (Kukier et al. 2003). Mineralogical analysis for the magnetic extracts was performed by an X-ray diffraction (XRD) technique (D8 Advance diffractometer, Germany). Mineral identification was based on reference X-ray diffraction data provided in the JCPDS-ICDD manual.

Scanning electron microscopy (SEM) technology is frequently applied to provide information on the composition and morphological characteristics associated with particles (Kukier et al. 2003; Magiera et al. 2011; Wang 2014). The microstructure and elemental compositions of the magnetic extracts were characterized by environmental scanning electron microscopy (SEM, Quanta^{TM} 250, USA) equipped with an energy-dispersive X-ray spectroscopy (EDS, Quantax 400-10, Germany).

All Mössbauer spectroscopic measurements were taken using a Mössbauer spectrometer at room temperature. A detailed description of the method can be found in Wang (2014). In the current study, only one street dust sample with the highest χlf value was selected for Mössbauer spectroscopic measurements.

Determination of metals using XRF

Metal concentrations including Cu, Zn, Pb and Ba in dust samples were determined by X-ray fluorescence (XRF, S8 Tiger, Bruker) spectrometry. The determination method together with the control of precision and accuracy was described in details in Wang (2014).

Determination of magnetic parameters

Magnetic susceptibility was measured at low frequency (470 Hz, χlf, 10^{-8} m^3/kg) and high frequency (χhf, 4700 Hz, 10^{-8} m^3/kg), using a Bartington Instruments MS2 susceptibility meter. The frequency dependence susceptibility (χfd, %) is calculated using Eq. (1) (Thompson and Oldfield 1986):

$$\chi\text{fd}(\%) = [(\chi\text{lf} - \chi\text{hf})/\chi\text{lf}] \times 100 \quad (1)$$

Anhysteretic remanent magnetization (ARM) was measured in a Molspin magnetometer after magnetization with an AF demagnetizer. The final values of ARM were expressed as the susceptibility of ARM (χ_{ARM}, 10^{-8} m^3/kg) by dividing ARM by the steady field. Acquisition of isothermal remanent magnetization (IRM) in the fields of 1 T (SIRM, 10^{-5} A m^2/kg) and −0.1 T (reverse) ($IRM_{-0.1\ T}$) was carried out using a Molspin pulse magnetizer. The parameter S-ratio can be used to gain information about the magnetic mineralogy (Bloemendal et al. 1992) and is calculated as (Thompson and Oldfield 1986)

$$S = IRM_{-0.1\ T}/SIRM \quad (2)$$

Thermomagnetic analyses were conducted using an AGICO KLY-4S kappameter with a CS-3 furnace from room temperature to 700 °C in an ambient atmosphere. Three representative street dust samples with high to medium χlf values were selected for hysteresis loop measurements and thermomagnetic analyses.

Statistical analysis

In order to quantitatively analyze and confirm the relationship among variables (i.e., magnetic parameters and metal concentrations), a Pearson's correlation analysis was applied. Pearson's correlation analysis was carried out using SPSS software for Windows (release Ver. 16, Inc, Chicago, IL).

Results and discussion

Magnetic properties

Table 1 presents the results of magnetic parameters of street dust samples. The samples are dominated by a

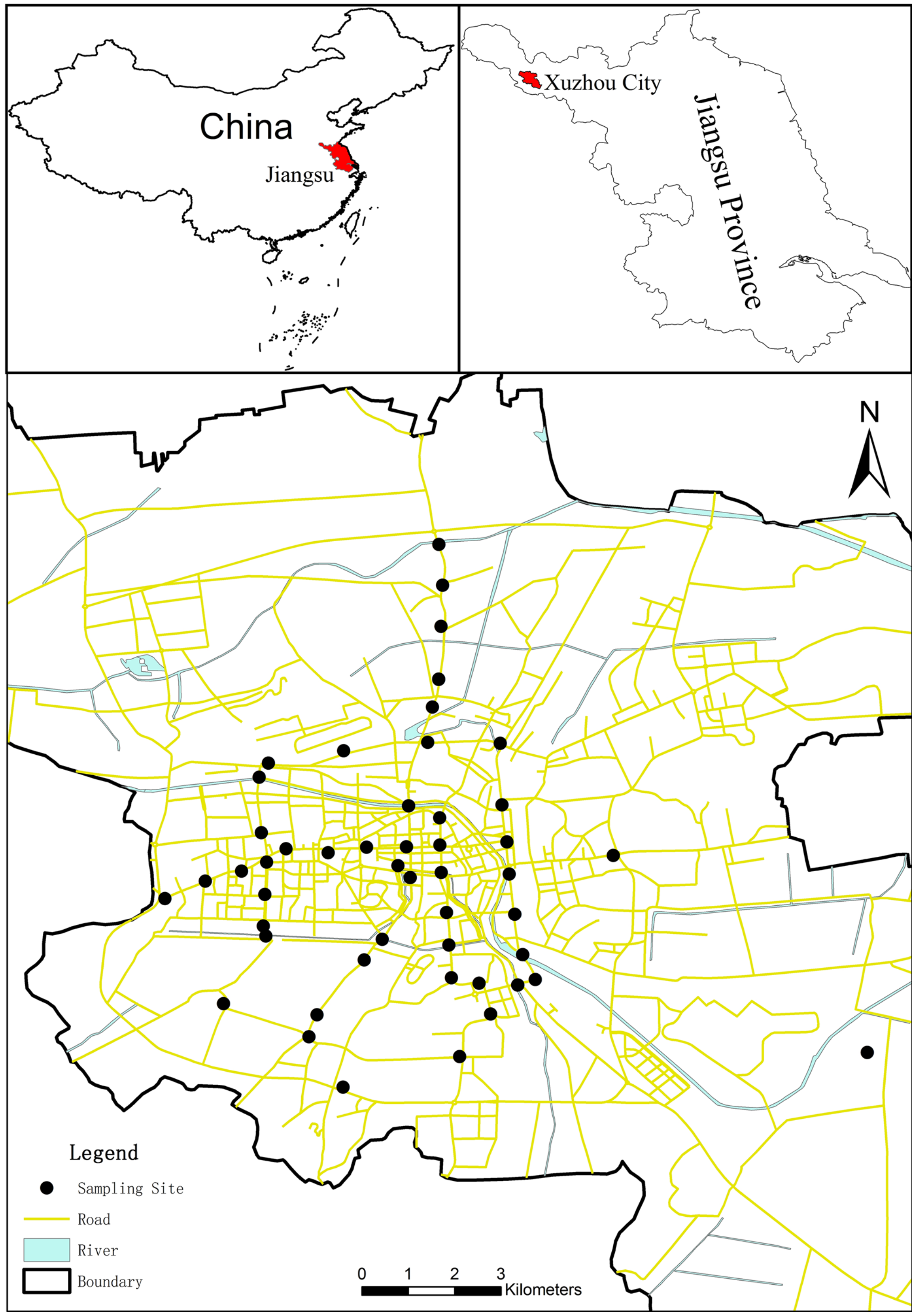


Fig. 1 Map of the study area showing the sampling locations

Table 1 Mineral magnetic results for the street dust samples ($N = 49$)

	χlf 10^{-8} m^3/kg	χfd %	χ_{ARM} 10^{-8} m^3/kg	SIRM 10^{-5} A m^2/kg	S-ratio %
Mean	421	0.49	357	2692	82
SE of mean	16	0.19	11.5	102	1.1
Median	405	0.12	348	2660	81
Range	247–676	0–6.91	196–551	1517–4687	56–100

ferrimagnetic mineralogy characterized by the χlf values. The samples yield an average χlf value of 421 $\times$ 10^{-8} m^3/kg, which is indicative of predominantly secondary ferrimagnetic minerals.

The parameter of χfd is sensitive to the superparamagnetic (SP) grains (Dearing et al. 1996). The values of χfd in our samples varied from 0 to 6.91 % with a mean of 0.49 %, indicating that magnetic carriers in the dust are predominately coarse-grained particles and the proportion of SP particles is much lower (Hay et al. 1997). Similar result was obtained by Bucko et al. (2011). They made an identification of magnetic particulates in road dust accumulated on roadside snow and found that the investigated road dust was dominated by a coarse fraction (multidomain, MD) and the contribution of SP and SD (single domain) grains was of minor importance.

The parameter χ_{ARM} is grossly proportional to the concentration of stable single domain (SSD) and fine pseudosingle domain (PSD) ferrimagnetic components. SIRM approximates the total remanence carrying mineral assemblage but is controlled largely by the SSD magnetite concentration (Thompson and Oldfield 1986). The means of χ_{ARM} and SIRM were 357 $\times$ 10^{-8} m^3/kg and 2692 $\times$ 10^{-5} A m^2/kg, respectively. A significant correlation between SIRM and χlf is found in Fig. 2, suggesting that ferrimagnetic minerals were dominating χlf in our dust samples.

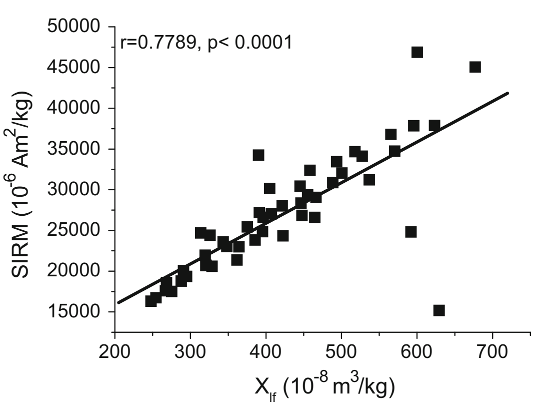


Fig. 2 Correlation between SIRM and χ

The parameter S-ratio is frequently applied to estimate the contribution of antiferromagnetic minerals in samples, with values >0.7 indicating the dominance of ferrimagnetic components (Robinson 1986). The high S-ratio (mean = 0.82) indicates the magnetic enhancement of the street dust was dominated by the presence of ferrimagnetic minerals.

Table 2 compares magnetic results in street dust samples in various locations throughout the world. χlf data from this work match the findings in Lanzhou (China), slightly higher than those found in Wuhan (China), West Midlands (UK) and Liverpool (UK), and finally match lower than those found in Zhuzhou (China) and Hezhang (China). The values of S-ratio and χ_{ARM} in the present work are generally similar to those reported for Liverpool (UK), while χfd and SIRM values are generally lower for other cities. In the absence of knowledge regarding industrial and traffic activities in the various cities or countries, comments on the causes of the differences between magnetic results are unjustified. However, it is generally agreed that traffic emissions accounted for the magnetic enhancement as all the samples were collected in street or road dust.

Hysteresis loop measurements were made between maximum fields of 1 T at room temperature. Hysteresis loops for three representative street dust samples are characterized by steep narrow curves (Fig. 3), indicative of the contribution of low coercivity ferrimagnetic minerals, in agreement with the S-ratio results. Similar results were reported by Zhu et al. (2012) and Robertson et al. (2003).

Thermomagnetic curves for three representative samples are shown in Fig. 4. Generally, the curves displayed similar behavior, that is, on heating there was a wide peak near 500 °C, before approaching to the Curie point (T_c) (T_c = 580 °C, Dunlop and Ozdemir 1997) suggesting the existence of a magnetite-like phase. The wide peak close to 500 °C is probably ascribed to a different grain size distribution of small particles of possibly various mineralogy

Table 2 The mean of the magnetic parameters in street dust samples from different cities in the world

Location	Sample number	χlf 10^{-8} m^3/kg	χfd %	χ_{ARM} 10^{-8} m^3/kg	SIRM 10^{-5} A m^2/kg	S-ratio %	References
Zhuzhou (China)	43	663	3.3		7400		Zhu et al. (2013)
Hezhang (China)	26	527	4.4		6600		Zhu et al. (2013)
Lanzhou (China)	71	449.88	2.22	956.49	6618.41		Wang et al. (2012)
Wuhan (China)	97	131.68	1		11,898		Yang et al. (2010)
West Midlands (UK)	216	157.1	3.36	21.8	4746.1	75	Shilton et al. (2005)
Liverpool (UK)		237	1.7	462	3433		Xie et al. (1999)
Liverpool (UK)	9	271	2.47			77	Robertson et al. (2003)
Xuzhou (China)	49	421	0.49	357	2692	82	Present work

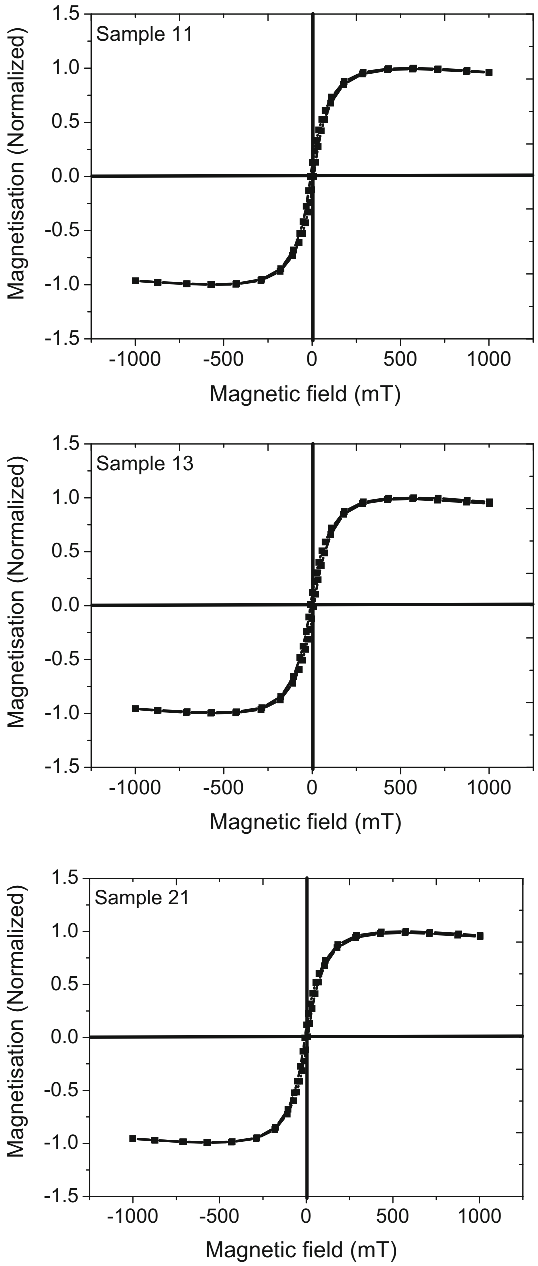


Fig. 3 Typical hysteresis loops displayed by the samples. Note the steep gradient and narrow loop

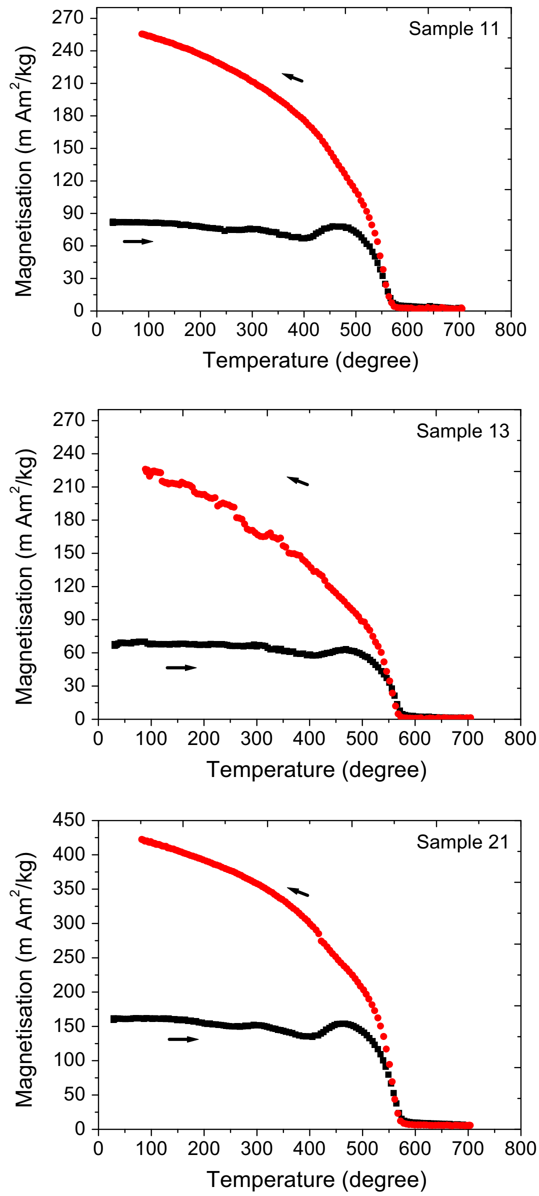


Fig. 4 High-temperature thermomagnetic curves for three representative samples

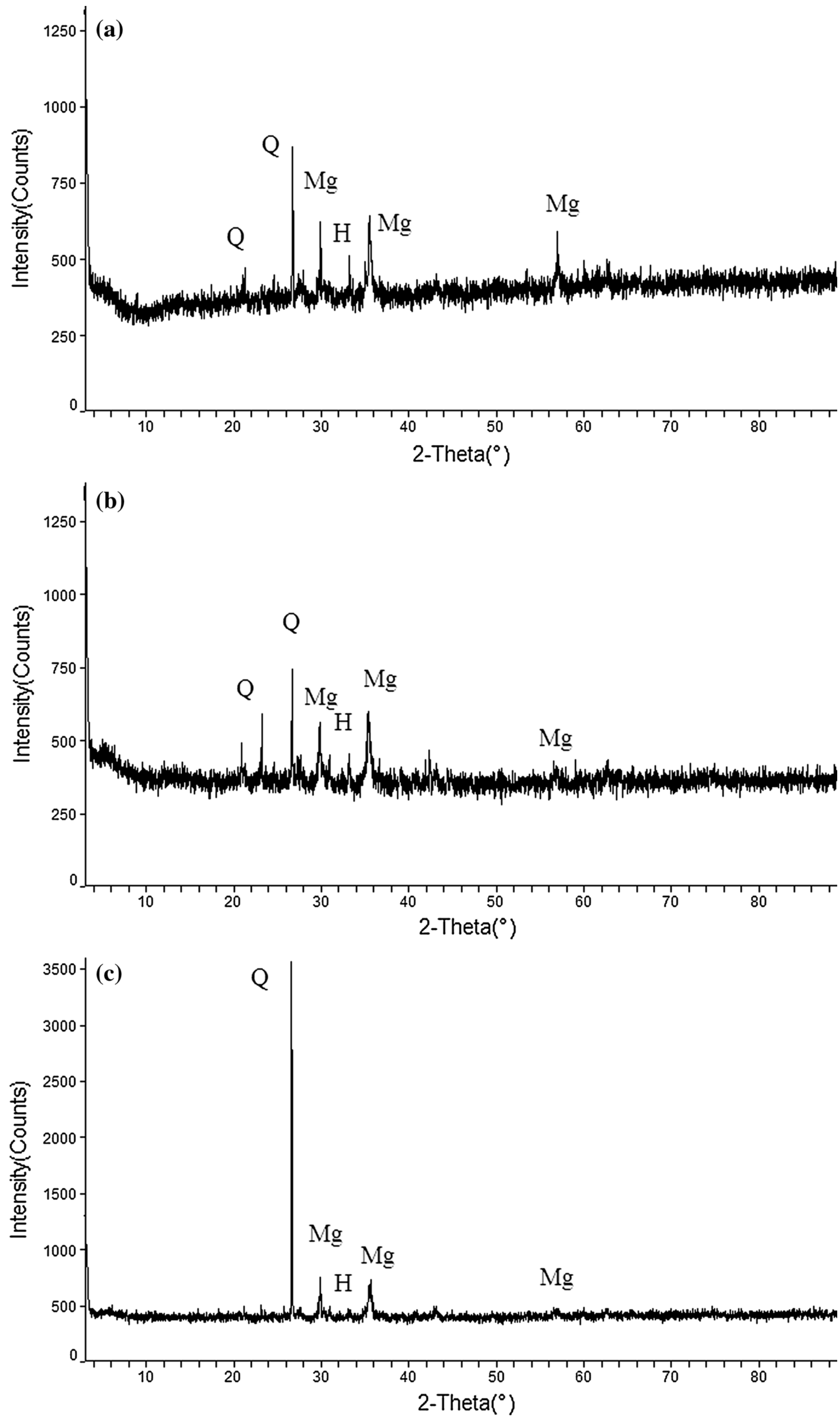
(Muxworthy et al. 2002). On cooling, the intensity of the magnetization was seen to sharply increase for these three street dust samples, suggesting the formation of a new magnetic phase (e.g., magnetite and/or thermally stable maghemite) (Zhang et al. 2012).

SEM–EDX and XRD analysis

The XRD analyses of magnetic extracts (Fig. 5) from the street dust were in accordance with the results of magnetic measurements. The X-ray diffraction peaks matched well with the diffraction lines of magnetite (Fe_3O_4), hematite (Fe_2O_3) and quartz (SiO_2), respectively. The magnetite peak intensity was greater than that of hematite, suggesting that crystalline iron in the magnetic extracts was mainly composed of magnetite. In addition, quartz was also found in the magnetic extracts. Similar results were obtained by Zhu et al. (2012). They investigated the relationship between magnetic parameters and heavy metal contents of indoor dust in e-waste recycling impacted area, Southeast, China.

SEM observations in conjunction with EDS analyses were performed on magnetic extracts separated from the

Fig. 5 X-ray diffraction patterns of magnetic extracts (*Q* quartz, *Mg* magnetite, *H* hematite)



street dust collected in the present study (Fig. 6). Overall, two groups of magnetic particles were identified: spherule-shaped iron oxides (diameter of 10–150 μm) and angular particles ($d \sim$ 30–200 μm) with complex chemical composition. Apart from elemental iron, the presence of Al, Ti, K, Ca, Si, Na, Mg and C was found in these magnetic extracts. These results are in accordance with studies by Kim et al. (2009), who identified by EDS analysis that

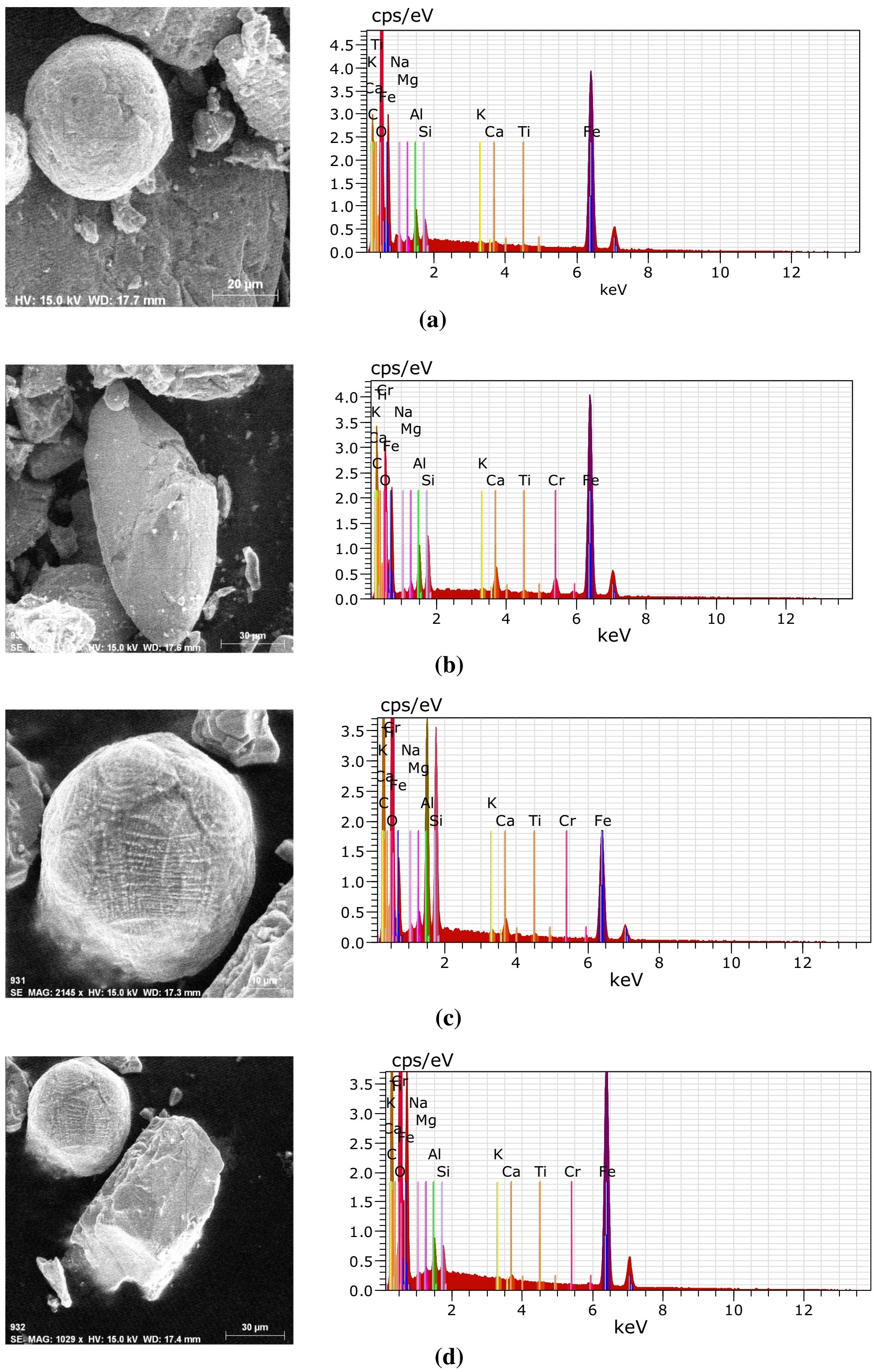
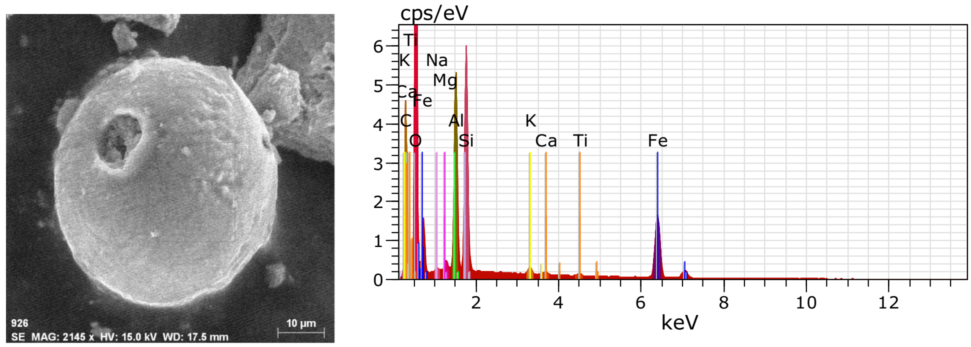
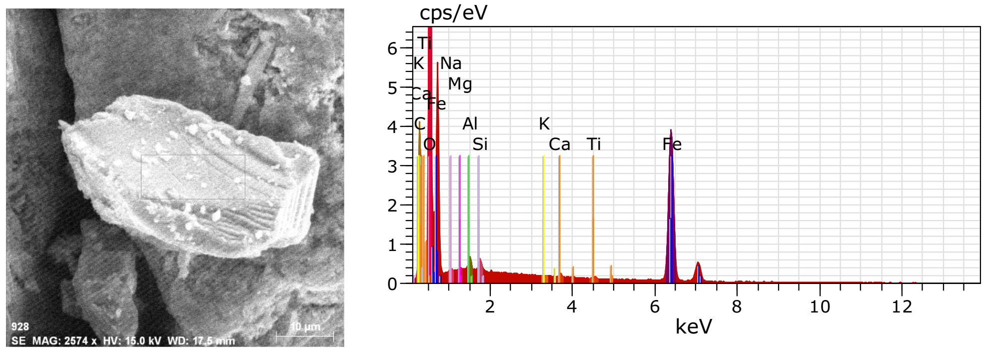


Fig. 6 SEM–EDS images of magnetic extracts from selected samples. *Squares* indicate the spots of EDS analysis

the spherical magnetic particulates separated from urban roadside dust were dominantly composed of Fe, O and C and additional minor elements including Al, Ca, Mg and Si. Chemical analyses of composition of particulate matter from exhaust revealed the presence of these minor elements as well (Rodriguez-Navarro and Sebastian 1996). It should be noted that all the magnetic particulates contain abundant C, suggesting their anthropogenic origin resulted



(e)



(f)

Fig. 6 continued

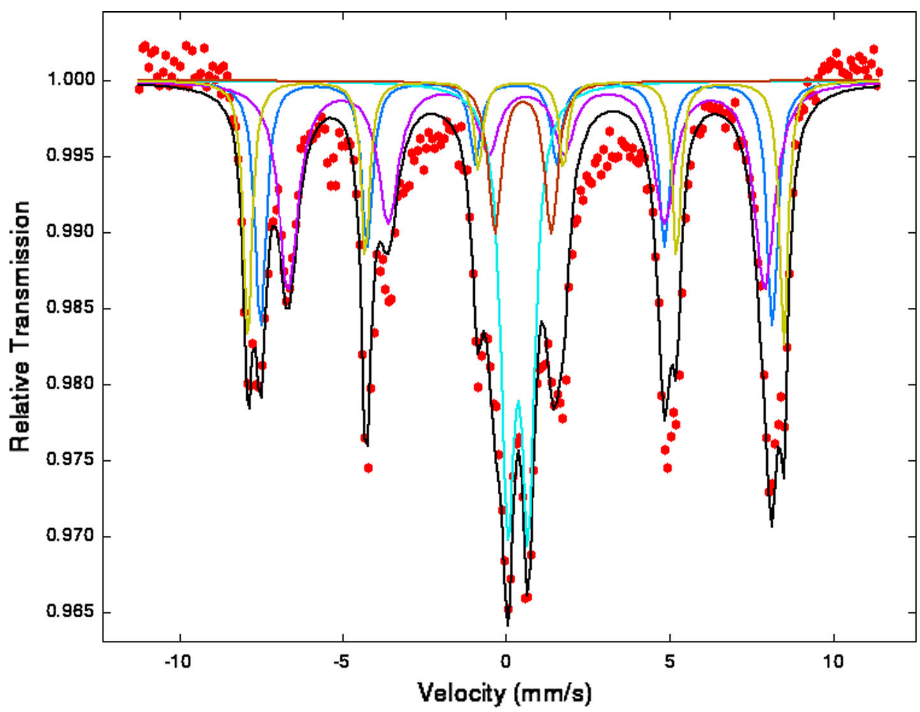


Fig. 7 Mössbauer spectra measured for street dust at room temperature

from fossil fuel combustion (Kim et al. 2007). Additionally, contents of other elements including Al, Ca, Mg and Si were likely dependent on the compositions of burning fossil fuels (Kim et al. 2009 and references therein).

It has been demonstrated that Cr is one of the most critical air toxics. The presence of particles containing Cr (Fig. 6b–d) was observed in street dust samples. These results are in good agreement with studies by Bucko et al.

(2011), who reported an accumulation of chromium-rich in magnetic extracts from the road dust sampled in roadside snow in Finland. Utsunomiya et al. (2004) also found Fe–Cr phase in airborne particles in Detroit urban atmosphere and suggested that these Cr-bearing particles were likely from traffic emissions.

^{57}Fe Mössbauer spectroscopy

Mössbauer spectrum was measured for one representative dust sample at room temperature and is shown in Fig. 7. The fitted parameters are listed in Table 3.

The central doublet with isomer shift IS = 0.373 mm/s is typical for high-spin Fe^{3+}, while the other one with IS = 0.526 mm/s is characteristic of Fe^{2+} (Muxworthy et al. 2002). It is very likely that the Fe^{2+} and Fe^{3+} ions are in silicates and/or carbonates originating from soils. This interpretation is supported by the large quadrupole splitting QS(Fe^{2+}) = 1.724 mm/s which is a typical feature of Fe^{2+} in oxides (Murad and Cashion 2004). For both components, the line widths (W) are above those expected for stoichiometric chemical compounds where $W \sim 0.25$–0.35 mm/s. Hence, each of the experimental doublet environments of the Fe^{2+} and Fe^{3+} ions is comparatively inhomogeneous due to the presence of other metal cations (Muxworthy et al. 2002). From relative abundance of both experimental doublets (Table 3), it is clear that the Fe^{3+} component is more common than Fe^{2+}.

The Mössbauer spectrum can also be fitted with two sextets (Fig. 7; Table 3), one corresponding to high-spin Fe^{3+} on the tetrahedral sites of magnetite ($B_{hf} = 48.7$ T) and the other to $Fe^{2.5+}$ ($Fe^{3+} + Fe^{2+}$) on the octahedral sites of magnetite (Cornell and Schwertmann 2003). Electron dislocation causes the nucleus to sense on average valence ($B_{hf} = 45.28$ T). Mixed $M + Q$ with a magnetic hyperfine field of $B_{hf} = 51.03$ T is indicative of α-Fe_2O_3 (hematite) (Murad and Cashion 2004; Cornell and Schwertmann 2003).

Similar results were obtained by Baghdadi et al. (2012), who investigated soil magnetic susceptibility in urban roadside soil of Beni Mellal City (Morocco) and applied Mössbauer spectroscopy to characterize the iron content within roadside soil. They detected magnetite $Fe^{2.5+}$ (relative abundance: 10.9 %), magnetite Fe^{3+} (39.5 %) and hematite (35.3 %) in their roadside soil samples.

Geochemical data and their relationship with magnetic concentration parameters

Dust samples ($N = 49$) collected were analyzed for four heavy metals (Cu, Zn, Ba and Pb). The obtained geochemical data are listed in Table 4. All measured metals in the present study were highly enriched: the concentrations of Cu, Zn, Pb and Ba were 3, 4.8, 2.8 and 1.3 times higher, respectively, than the background values. The high metal concentrations were likely due to traffic emissions.

The effectiveness of various magnetic parameters was tested as a proxy for heavy metal pollution in street dusts. The correlation coefficient between the heavy metal concentrations and magnetic concentration parameters is summarized in Table 5. Zn was found to show no correlation with χlf, χ_{ARM} and SIRM, suggesting that there is no link between magnetite formation and metal Zn in the street dusts. Cu and Ba showed significant correlations with χ_{ARM} and SIRM, while only Pb correlated well with χ_{ARM}.

In a study of roadside dusts in Seoul by Kim et al. (2007), Pb showed no correlations with χlf, ARM and

Table 4 Total concentrations (mg/kg) of metals in street dusts ($N = 49$)

	Cu	Zn	Ba	Pb
Mean	69	360	597	73
SE of mean	3.4	35.3	14.5	3.5
Median	66	302	572	69
Range	26–163	144–1540	495–1155	37–152
Background value[a]	22.6	74.2	469	26

[a] Mean values of different natural soils of China (China Environmental Monitoring General Station 1992)

Table 3 Fitted Mössbauer parameters for spectra of Fig. 6

	IS (mm/s)	QS (mm/s)	B_{hf} (T)	W (mm/s)	Relative abundance (%)	Assignment
Doublet I	0.373	0.624	–	0.498	21.4	Fe^{3+}
Doublet II	0.526	1.724	–	0.478	7.5	Fe^{2+}
Sextet I	0.307	–	48.7	0.403	20.9	Fe_3O_4–Fe^{3+} (Tetra)
Sextet II	0.627	–	45.28	0.751	32.6	Fe_3O_4– Fe^{3+} Fe^{2+} (Octa)
Mixed $M + Q$	0.376	−0.177	51.03	0.315	17.6	α-Fe_2O_3

IS isomer shift (with reference to metallic iron), *QS* quadrupole splitting, B_{hf} magnetic hyperfine field, W line width; relative abundance, % relative area fraction of a doublet or a sextet or a mixed one with respect to the area of the whole fitted spectrum

Table 5 Correlation coefficients between the magnetic concentration parameters and the metal concentrations for street dusts

Variables	Cu	Zn	Ba	Pb
χlf	0.276	0.062	0.281	0.148
χ_{ARM}	0.453**	0.148	0.479**	0.357*
SIRM	0.426**	0.088	0.390**	0.261

** Correlation is significant at the 0.01 level (2-tailed)

* Correlation is significant at the 0.05 level (2-tailed)

SIRM, but such a relationship with χlf, ARM and SIRM was significantly identified for Cr, Cu, Fe, Mn and Zn. A study on urban street dusts in Loudi, China, by Zhang et al. (2012) found a positive correlation between χlf and the metals Cu and Pb, a negative correlation with metal Ba, no association with metal Zn.

Conclusions

In this study, we combined Mössbauer spectroscopy, SEM–EDS and magnetic results in a detailed characterization of urban street dust collected in Xuzhou (China). The results of magnetic measurements of the street dust indicated that coarse-grained, multidomain magnetite is the primary magnetic mineral. SEM and EDS analyses identified two groups of magnetic materials: spherule-shaped iron oxides and angular particles with complex chemical composition. Cu and Ba showed significant correlations with χ_{ARM} and SIRM, while only Pb correlated well with χ_{ARM}. The present study suggests that a combination of the various methods could offer a powerful analytical technique in the study of magnetic properties of urban street dust.

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