

C. Spiteri  
V. Kalinski  
W. Rösler  
V. Hoffmann  
E. Appel  
MAGPROX team

## Magnetic screening of a pollution hotspot in the Lausitz area, Eastern Germany: correlation analysis between magnetic proxies and heavy metal contamination in soils

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C. Spiteri · V. Kalinski · W. Rösler  
V. Hoffmann (✉) · E. Appel  
MAGPROX team  
Institute for Geosciences,  
University of Tübingen, 72076  
Tübingen, Sigwartstrasse 10, Germany  
E-mail: viktor.hoffmann@uni-tuebingen.de  
Tel.: +49-7071-2974697  
Fax: +49-7071-295842

**Abstract** This investigation was carried out within the scope of EU-FP5 project MAGPROX. In parallel with the work of Kalinski et al. (2004, submitted), in which the magnetic signatures of the same soil profiles were analysed in more detail. The 'hot spot' under investigation was situated in the Lausitz area, Eastern Germany, between two major power plants, *Schwarze Pumpe* and *Boxberg*. This heavily industrialized region is known as the *Black Triangle*, named after the large lignite deposits and the old-technology power plants, among other petrochemical plants, refineries, textile manufacturing and glasswork

industries. The relationship between magnetic parameters and heavy metal concentrations (Fe, Mn, Zn, Pb, Cu, Cr, Cd, Co and Ni) in soil profiles was determined statistically using linear regression analysis. Strong positive correlation was observed between heavy metal concentrations as viewed preliminarily from the heavy metal and magnetic susceptibility distributions with depth (soil profiles), and from the correlation coefficients obtained.

**Keywords** Magnetic proxies · Heavy metals · Soil pollution · Lausitz area · Germany

### Introduction

In recent years, applications of environmental magnetism have been increasingly extended to pollution studies. Magnetic properties of soils, in particular magnetic susceptibility ( $\chi$ ), have become an important auxiliary tool in estimating the extent of contamination due to industrial activity. It provides a rapid, cost-effective, efficient and highly sensitive way of analysing pollution in large scales for review purposes and, through more systematic sampling procedures, it is targeted to smaller areas previously identified as pollution hotspots. In order to properly interpret the magnetic data, it is imperative to distinguish the atmospheric input from the natural background (Petrovsky and Ellwood 1999). Moreover, the various pollution sources have to be identified and discriminated. This represents one of the major problems in soil magnetometry, especially in less

contaminated areas, where the magnetic signal is not significantly high.

Soil magnetometry in areas affected by anthropogenic dustfall originating from combustion processes is based on the knowledge that ferrimagnetic particles, namely magnetite-like phases, are produced from pyrite during fossil fuel combustion. Pyrite and other iron impurities in the pulverized coal are transformed into molten spheres of magnetic iron oxides (i.e. magnetite, maghemite, haematite— $\text{Fe}_2\text{O}_3$  and mixtures) and iron, liberating  $\text{SO}_x$  as gaseous components. Thus, a fraction of the iron is ultimately converted into ferrimagnetic minerals, which are in turn emitted into the atmosphere together with other phases (Magiera and Strzyszczyk 1999). Depending on shape and size, the anthropogenic ferrimagnetic minerals from stack emissions are transported as dusts or aerosols over variable distances before depositing on soils surfaces, the most important sink of

anthropogenic airborne particulate matter (Dedik et al. 1992).

Several studies suggest a relation between magnetic susceptibility and heavy metal concentrations in polluted soils (Thompson and Oldfield 1986; Āurža 1999; Hoffmann et al. 1999). Nevertheless, the relationship between magnetic particles and heavy metals in fly ashes, industrial particulates and vehicle emissions is still not fully understood.

One of the mechanisms proposed is the surface adsorption of metal oxides (e. g. magnetic phases) during fly ash formation (Georgeaud et al. 1997). The authors also refer to a preferential adsorption of Pb, Zn, Cu, Cr, Cd on the surface of iron(III) oxyhydroxides as an alternative mechanism to describe the association with heavy metals. Other studies draw attention to the enrichment of first transition group elements (V, Cr, Mn, Fe, Co, Ni, Cu and Zn) in the magnetic fraction of coal fly ash (Hansen et al. 1981; Hunt et al. 1984), in the form of substituted spinels of  $Fe_{3-x}M_xO_4$ . The central background of the potential link can be the “same source–same distribution pathway” principle.

In various studies, the relationship between magnetic susceptibility and heavy metal concentrations was further analysed by statistical methods (Beckwith et al. 1986; Dekkers and Pietersen 1992; Strzyszcz and Magiera 1993; Āurža 1999; Hanesch et al. 2001). In the work of Knab et al. (2001), magnetic susceptibility values along a highway were found to be positively correlated with heavy metals, in particular Pb, Cu, Cd and Zn. Fuzzy C-means cluster analysis was employed to discriminate between anthropogenic and pedogenic/geogenic background values in shallow sections along soil profiles. In areas with multiple pollution sources, the link between the magnetic signal and heavy metal concentration was found to be too complex to be described by simple linear statistics, but could fit log normal distributions, in which the logarithmic values of chemical concentration and magnetic susceptibility were used as input for multivariate statistical analyses (Hanesch et al. 2001).

In the present study, correlations between measured concentrations of heavy metals along soil profiles collected from the *Schwarze Pumpe-Boxberg* area were investigated using linear regression analysis (LRA). Heavy metal concentrations were detected using atomic absorption spectroscopy (AAS). In this way, the potential use of magnetic susceptibility as a preliminary, semi-quantitative proxy for detecting heavy metal concentrations in polluted soils, prior to the setup of time-consuming and expensive chemical methods, could be evaluated.

The geochemical methods were complemented by SEM/EDX (scanning electron microscopy and elemental) analyses, which provided additional valuable

information with regard to morphology, grain size and chemical signature of the magnetic particles of interest.

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## Materials and methods

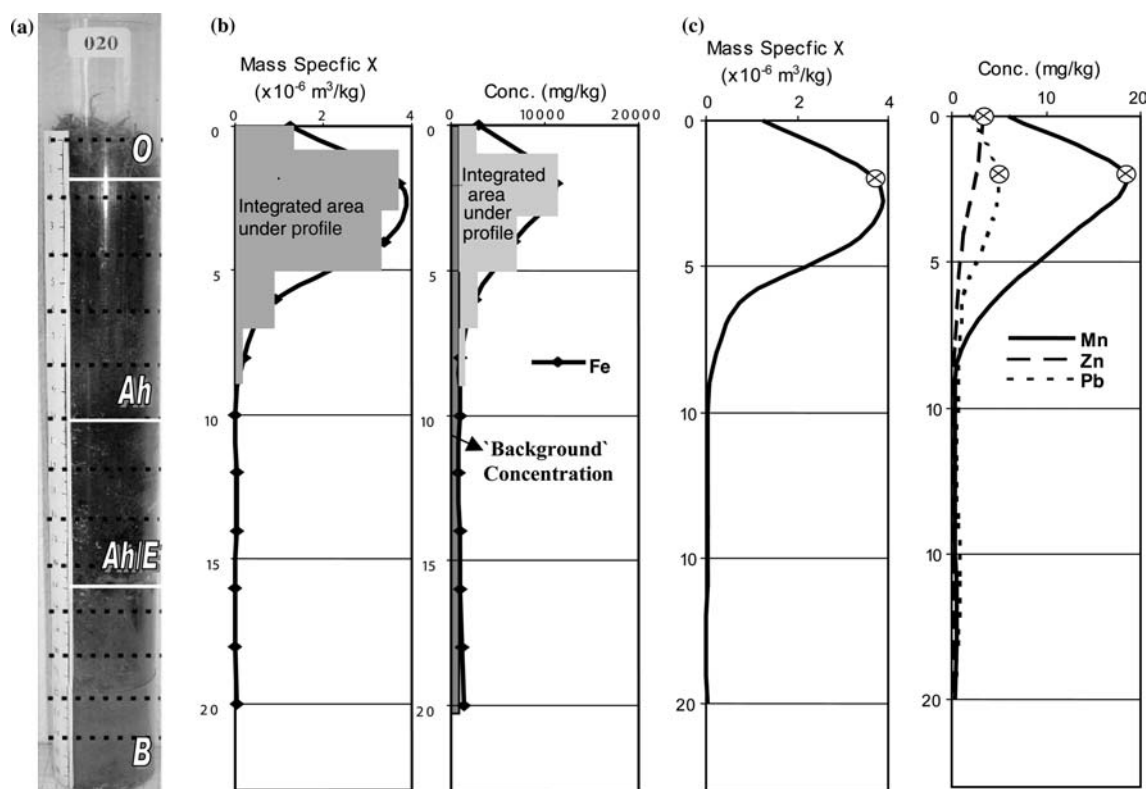
A total of 140 measuring points were sampled following an average grid density of 1×1 km. The sampling points were mostly located in forest areas with an age of 40 years or older, situated at least 50 m from major roads and 50 m away from forest margins.

Field measurements of magnetic susceptibility were performed by using a Bartington susceptibility meter connected to a loop sensor (MS2D), following the MAGPROX methodology (Schibler et al. 2002). The exact geographical position of each measurement was recorded using a Trimble GPS Total Station 4700. Topsoil magnetic measurements were later used for compiling magnetic maps of the area, as explained in further detail by Kalinski et al. (2004, submitted). A Humax soil corer with plastic tubes was used for taking two vertical soil cores (30 cm long and 3.5 cm diameter cores) at each sampling point (Fig. 1a). Vertical magnetic susceptibility profiles were recorded in-situ for each site using the new magnetic susceptibility profiling instrument, SM400 (ZH-Instruments), developed within the framework of MAGPROX (Petrovsky et al. 2004).

Field magnetic measurements were verified with laboratory readings of soil cores using a Bartington MS2C sensor. A detailed account on the magnetic susceptibility values measured with the various instruments (field and laboratory measurements with SM400, KLY2, Bartington) and data of mineral-magnetic analyses (thermo-magnetic analyses with KLY3, IRM) can be found in Kalinski et al. (2004, submitted). Readings of magnetic susceptibility (measured on KLY2) were obtained on individual samples cut from cores at 2 cm intervals. Individual sample readings are required because SM400 values refer to the material outside the extracted soil core rather than to the soil profile itself.

Sub-samples from 16 cores (same samples as used for KLY2-magnetic measurements) were sieved and dissolved in 2 M HNO<sub>3</sub> and photometrically analysed for the following heavy metals using Perkin-Elmer 1100B Atomic Absorption Spectrophotometer (AAS) at the Institute of Environmental Engineering, Zabrze, Poland: Fe, Mn, Zn, Pb, Cd, Cu, Ni, Cr and Co. Sample preparation was carried out according to the ‘*Wegleitung für die Probenahme und Analyse von Schadstoffen im Boden*’, (Guidelines on sampling and analysis of pollutants in soils), issued by the “Deutsches Bundesamt für Umwelt, Wald und Landschaft” (1989).

The relations between mass-specific magnetic susceptibility ( $\chi$ ) values and heavy metal concentrations were determined using simple linear regression analysis (LRA) (Knab et al. 2001), in three different ways, i.e. on



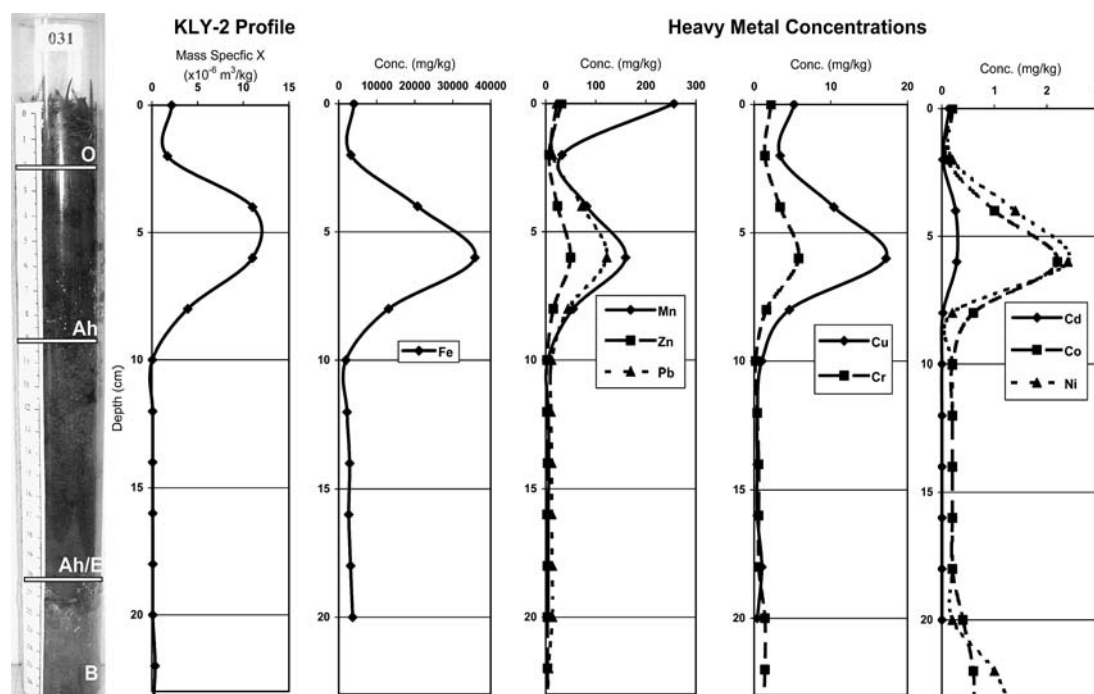
**Fig. 1** a Photo of Core 20, depicting subsamples referred to as *Individual sample - sample analysis*; b KLY-2 magnetic susceptibility profiles (individual samples measured with KLY-2) and heavy metal profiles showing integrated area and 'background' area; c KLY-2 and heavy metal profiles showing measured peak value, marked with

an individual sample-sample basis, integrated curves and peak values of vertical distribution. For the integrated curves, the areas under the  $\chi$  and the heavy metal profiles are compared (Fig. 1b, c). In this context, 'background' concentration does not refer to the lithogenic background but to the value of the 'plateau' of the profile. The area under the profile gives a measure of the total input of magnetic phases and heavy metals and accounts for different depth characteristics of deposition/migration. Peak values are chosen from sample measurements and not from fitted curves (Fig. 1c). Correlations were interpreted in terms of the correlation coefficient  $R^2$ .

Morphological details of individual particles provide valuable information on the possible pollution sources and on the origin of particles in general. Soil samples and magnetic extracts (prepared using a hand magnet) were scanned under scanning electron microscope (SEM- LEO 1450 VP). Semi-quantitative analysis of the composition was performed by energy dispersive X-ray (EDX) detector (Oxford) coupled to the SEM, set on the backscattered electron mode (BSE).

## Results

Enhanced magnetic susceptibility levels are located in the uppermost horizons of the soil profiles, namely in the O and Ah horizons, decreasing downwards to the so-called 'background values'. Heavy metal profiles show the same trend (Figs. 1, 2, 3). It is interesting to note that in all of the 16 analysed cores, both the magnetic susceptibility and the heavy metal peaks occur either in the organic (O) or in the humic horizon (Ah) below, irrespective of the depth of these two soil horizons. The location of the magnetic susceptibility and chemical concentration 'maxima' was influenced by both the thickness of these two horizons and the position of the sampling point relative to the pollution emitter. In the area closer to the power plant, the peak magnetic susceptibility values occur at deeper levels. In the case of power plants in East Germany, which were not previously equipped with dust filters or electro-precipitators, the quantity of dust emitted and deposited in the vicinity of the power plants was significant. In addition, the quantity of dust fall depending on distance from a power plant is controlled by the stack height. Since the magnetic peak is mostly located within the Ah horizon, the thickness of this horizon should be considered as a dominant variable, which controls the depth of the magnetic susceptibility peak. Figures 2 and 3 show examples of the different



**Fig. 2** KLY-2 magnetic susceptibility profiles (individual samples measured with KLY-2) and corresponding heavy metal profiles for selected core 31

chemical profiles compared to the magnetic susceptibility values for two selected soil cores.

Concentrations of Fe (ranging from 300 mg/kg to 40,000 mg/kg) are found to be 2 orders of magnitude higher than of other heavy metals, i. e. Mn, Zn and Pb (0–500 mg/kg), Cu and Cr (0–20 mg/kg), Cd, Co and Ni (0–3 mg/kg).

The correlation coefficients of LRA between mass specific magnetic susceptibility values and heavy metal concentrations for individual samples from selected cores are given in Table 1. An example of a set of correlation plots for a particular core is shown in Fig. 4. Table 2 shows correlation coefficients for (1) integrated curves and (2) peak values of the various cores.

Figure 5 shows the thickness of the O/Ah soil horizons versus the location of the ‘maxima’ of magnetic susceptibility and heavy metals, respectively. The reasonable linear correlation shows that the signal distribution along the soil profile is dominated by deposition/accumulation of dust particles (e.g. magnetic particles), while the role of translocation can be considered negligible.

Scanning electron microscopic observations were conducted on selected samples, both ‘raw’ topsoil material and magnetic extracts. In both cases, bright, spherical particles were the particles of interest. The spherical shape is frequently the result of particle formation at high temperatures in a suspended mode and

generally indicates an anthropogenic origin (Fig. 6). In BSE, areas with higher average element number (essentially differences in Fe-concentration in the grains and other heavy metals) appear brighter than other areas, against a dark background.

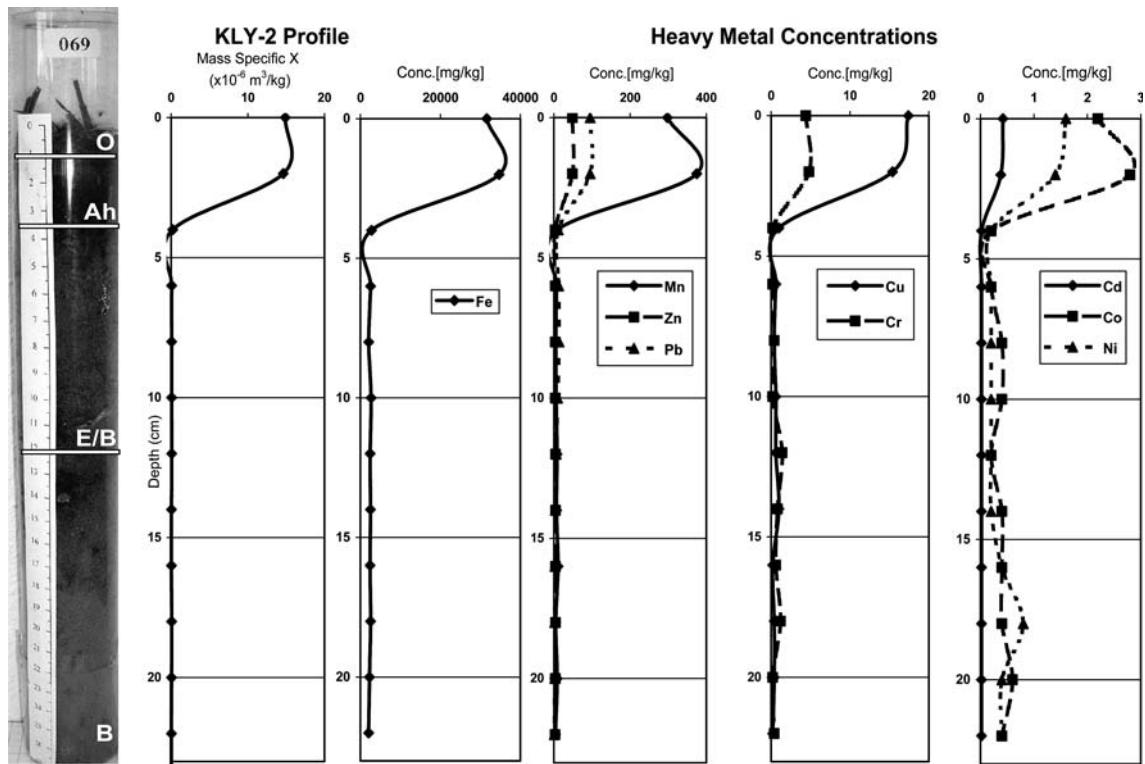
Spheres detected in topsoil could be particulates from fly ashes, such as cenospheres or hollow spheres (Fig. 7), or plerospheres (filled with smaller spheres), as documented by Fisher et al. (1976). On the contrary, the magnetite spherules in fly ash have a typical morphology, characterized by a rough ‘orange-peel’ surface (Fig. 8), having different magnetic parameters which is related to the type of burnt coal and the combustion conditions (Petrovský et al. 2000).

The two groups were distinguished on the basis of morphology and size, as shown in the SEM micrographs (Figs. 7, 8).

Energy dispersive X-ray spectra of ‘raw’ topsoil samples and magnetic particles revealed the presence of other elements such as Sn, Hg and Mn, besides the ones detected previously using AAS (Fig. 9). These metals are not commonly associated with fly ash particles from power plants but rather with the presence of other possible sources in the same region such as paint, chemical and metallurgical industries.

## Discussion

The main observation drawn from the above results is that a strong, positive correlation exists between magnetic susceptibility and heavy metals concentration in



**Fig. 3** KLY-2 magnetic susceptibility profiles (individual samples measured with KLY-2) and corresponding heavy metal profiles for selected core 69

most soil profiles. This can be viewed not only visually, through the combined plot of heavy metal depth distributions and magnetic susceptibility profiles (Figs. 2,

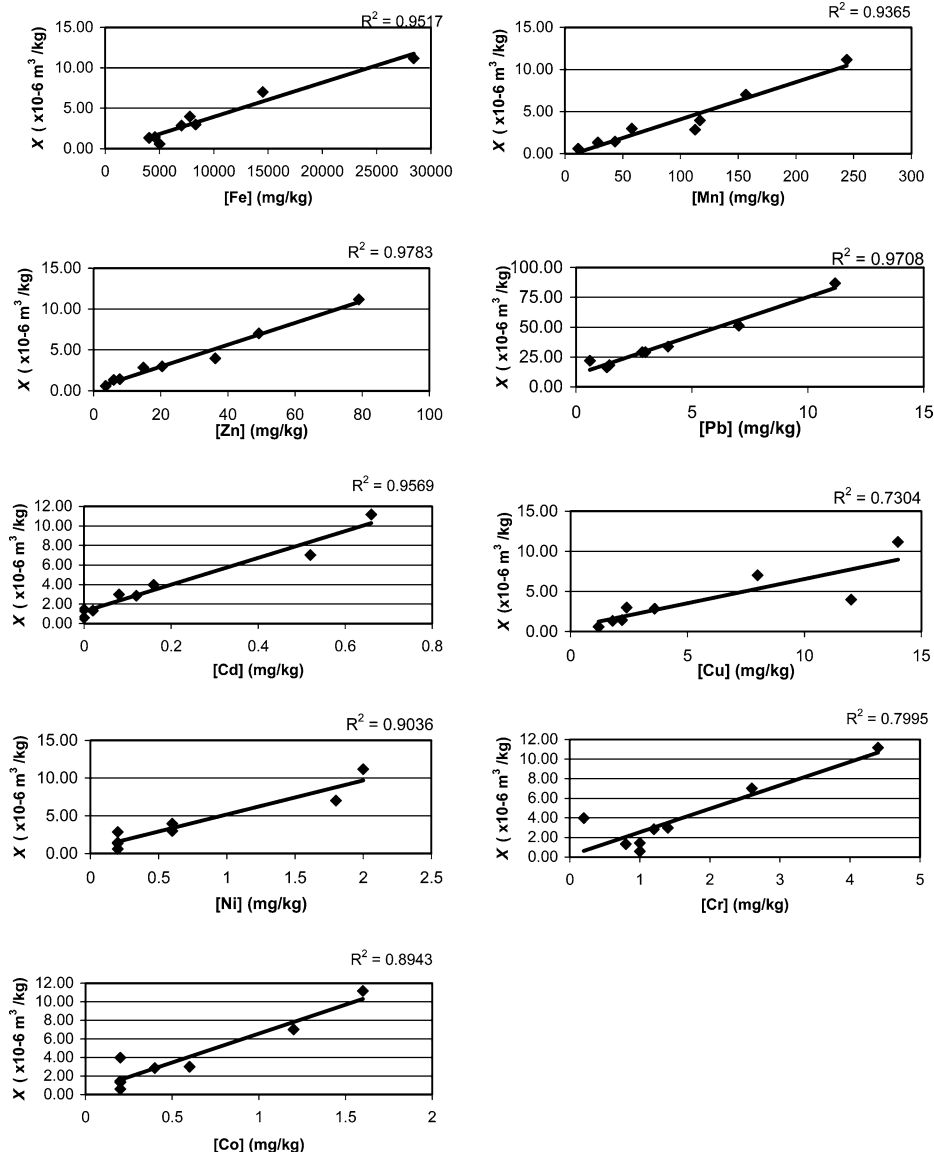
3) but also quantitatively, through the various statistical correlations (Tables 1, 2). Enhanced magnetic susceptibility and heavy metal concentrations were located in the uppermost soil horizons (O and Ah horizons), decreasing downwards to 'background' values. In few cases, maxima of Cu, Co and Cr were observed at depths of around 20 cm. These heavy metal peaks occurred in the B horizon, just underneath the E horizon and could have thus resulted from leaching during podzolisation. Those metal peaks, which were not aligned with the magnetic susceptibility could have resulted from mobilization/transportation of the heavy metals downwards through the soil, like for e.g. Mn and Cu. Further information on the metal specification,

**Table 1** Correlation coefficients ( $R^2$ ) between KLY-2 mass specific magnetic susceptibility values and heavy metal concentrations of individual 2 cm subsamples from each core.

Core	Fe	Mn	Zn	Pb	Cd	Cu	Ni	Cr	Co
<b>Spremborg Area</b>									
11	0,61	0,71	0,57	0,3	0,59	0,9	0,55	0,92	–
14	0,87	0,91	0,79	0,95	0,93	0,72	0,80	0,59	0,64
15	0,96	0,99	0,94	0,95	0,01	0,79	0,41	0,7	0,31
20	0,96	0,8	0,79	0,98	0,99	0,83	0,94	0,95	0,91
31	0,88	0,29	0,69	0,9	0,89	0,91	0,53	0,82	0,69
35	0,97	0,81	0,68	0,78	0,75	0,81	–	0,67	0,13
36	0,95	0,94	0,98	0,97	0,96	0,73	0,9	0,80	0,89
42	0,93	0,97	0,44	0,97	0,53	0,54	0,5	0,66	–
68	0,89	0,83	0,68	0,96	0,72	0,63	0,48	0,61	0,55
69	0,99	0,98	0,99	0,98	0,99	0,99	0,87	0,93	0,95
78	0,76	0,98	0,49	0,77	0,56	0,49	0,14	0,25	0,83
82	0,98	0,87	0,82	0,98	0,97	0,85	0,09	0,82	0,63
135	0,88	0,99	0,99	0,93	0,99	0,95	–	0,98	–
<b>Weißwasser Area</b>									
114	0,98	0,99	0,99	0,98	0,39	0,99	0,95	0,98	–
117	0,88	0,5	0,38	0,63	0,24	0,53	0,41	–	–
122	0,9	0,51	0,95	0,71	0,98	0,97	0,31	0,92	–

**Table 2** (i)  $R^2$  values for integrated KLY-2 magnetic susceptibility values with integrated heavy metal concentration (second column) and (ii) peak magnetic susceptibility values against peak heavy metal concentration (third column) of the various cores

Metal	(i) $R^2$	(ii) $R^2$
Fe	0,88	0,8
Mn	0,72	0,1
Zn	0,84	0,44
Pb	0,67	0,23
Cd	0,76	0,55
Cu	0,7	–
Ni	–	–
Cr	0,47	–
Co	0,48	0,60



**Fig. 4** Thickness of O/Ah horizons (cm) versus location of “maxima” of magnetic susceptibility/heavy metals in soil profiles (cm)

valency and mineral phase would be required to explain the occurrence of such peaks and the mechanisms controlling the mobility of the metals.

As expected, correlation coefficients between Fe content and magnetic susceptibility values were found to be the highest in all cores with all the three methods of statistical analysis. This implies that the magnetic susceptibility and Fe profiles follow a similar pattern, with values above background in the top subhorizons of the soils. Mn, Zn, Pb and Cu also showed significant positive linear correlation with  $\chi$ , with most  $R^2$  values ranging from 0.70 to 0.98. For the other metals, namely Co, Cr and especially Ni, unsatisfactory  $R^2$  values were

obtained. Since these metals occur in very low concentrations, close to the response limit of the AAS method (0.02 mg/kg), a small deviation in the measurement will cause a considerable effect in the correlation coefficients.

Results for Cd did not reveal particular trends. Some correlations gave high coefficients, others extremely low or no correlation at all. More details about the specification of this metal would be helpful to explain this outcome.

Higher correlations were observed for cores with relatively high magnetic susceptibility values. As a general rule, it can be said that when magnetic susceptibility and heavy metal concentrations are both high, the source is most likely anthropogenic. Conversely, when magnetic susceptibilities are high but heavy metal concentrations are low, the source is most likely a natural one, indicating the presence of ‘geogenic’ anomalies. In

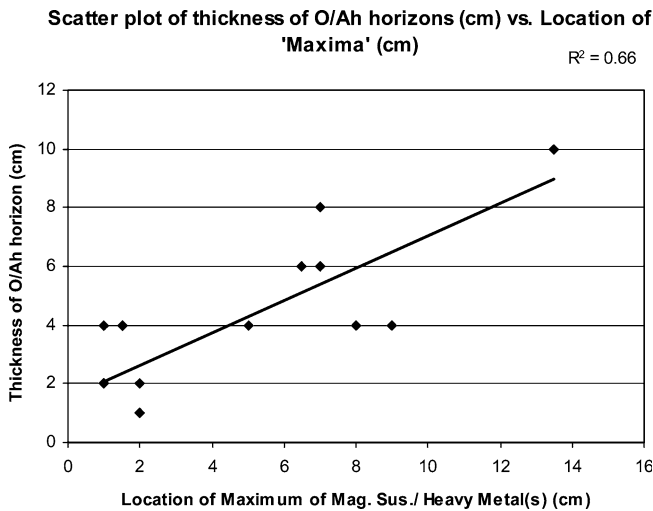
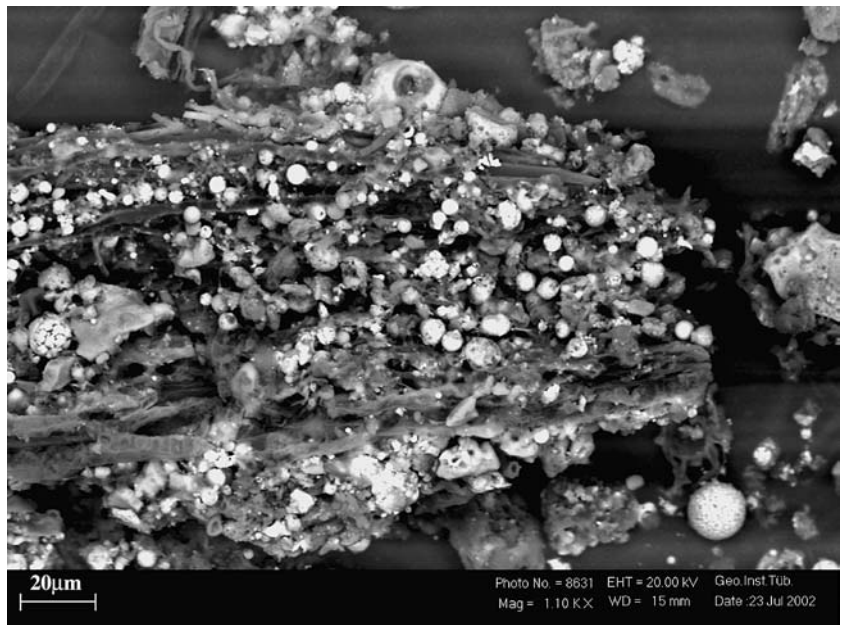


Fig. 5 Correlation plots of  $\chi$  versus heavy metal concentration for Core 36 with regression lines and correlation coefficients ( $R^2$ )

this work, as a rule of the thumb, 'high magnetic susceptibility' refers to values greater than  $2 \times 10^{-6}$  SI (bulk susceptibility). This value was found after performing a series of statistical analysis using SPSS software version 11.5. In the case of heavy metal concentrations, the classification between 'high' and 'low' must be viewed with respect to the limit values stated in official guidelines and standards. Different levels of limit values (precautionary, trigger and action values) are stated by the German Federal Soil Protection Act (Höper 1998). Nearly all values for Ni, Cu and Cr fall

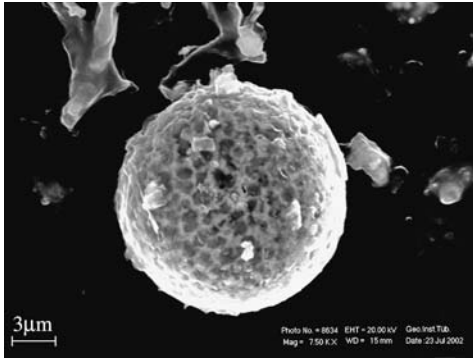
Fig. 6 SEM micrograph: a general view of a multitude of smaller, bright spherules (magnetic extract-sample 65; Ah horizon)



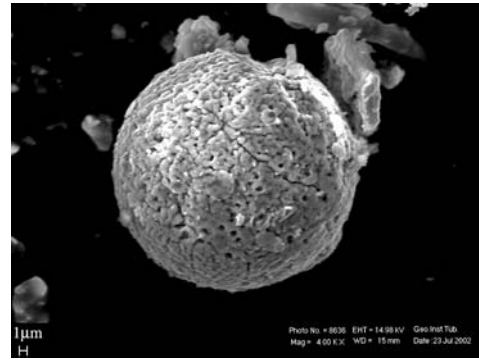
below the precaution limit values set for sandy soils. Some values for Zn, Cd and especially Pb exceed the precaution values, as observed in particular cores that also show relatively high magnetic susceptibility values. Nevertheless, they still fall below the trigger values established for industrial and commercial areas.

The heavy metals detected in the soils were compared to the composition of brown coal ashes (as documented by Fiedler and Rösler (1988), for brown coal fly ashes in the former GDR), since the two major candidate pollution sources in the area are believed to be the lignite-fired *Schwarze Pumpe* and *Boxberg* power plants. Although the exact chemical composition of fly ashes depends on both the type of coal and the conditions of combustion (temperature, oxygen content), the similarity between certain metal concentrations, (Cd, Zn and Co) in fly ashes and soil samples suggests that fly ashes could be the main source of heavy metal contamination and consequently also of magnetic susceptibility in topsoils. Other elements present in brown coal fly ashes, e.g. Hg, Tl and Ti were not analysed by AAS, but were detected through EDX analysis.

The first test (individual sample—sample analysis) was found to give the highest correlation coefficients. Nonetheless, the results from correlating the integrated profiles of  $\chi$  and heavy metals were also found to be significantly high. Unlike the first test, the latter analysis combines all cores from different sampling points, in which the influence of other variables, (variability in background contents, differences in transport of magnetic particles and heavy metals, the effect of other pollution sources) could render the comparison more complex.



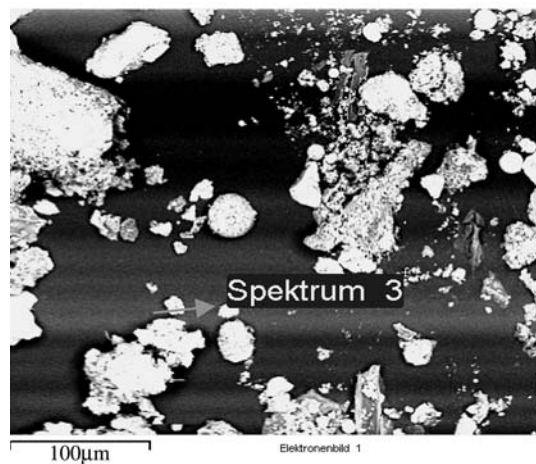
**Fig. 7** SEM micrograph of a sphere with hollow structure, possibly a cenosphere (topsoil 135; O horizon)



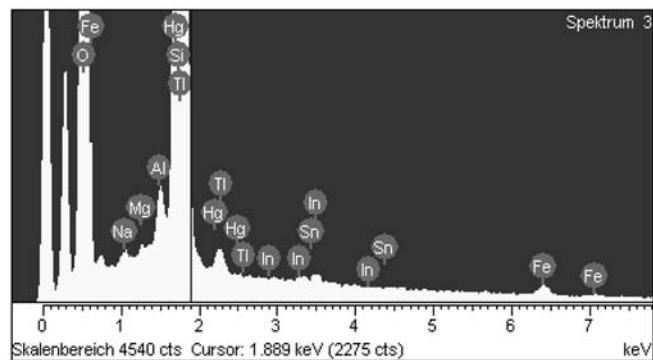
**Fig. 8** SEM micrograph of a magnetite spherule with 'orange-peel' surface structure, (magnetic extract-sample 65; Ah horizon)

The peak magnetic signal and the peak heavy metal concentrations did not correlate very well, as indicated by the relatively low correlation factors obtained (Table 2). The combination of different sampling sites within the same correlation analysis might also be affected by the transition function between the 'magnetic plume' and the 'heavy metal plume', which varies with the distance from the source. Accordingly, the use of simple linear correlation methods for relating peak values might not be suitable.

**Fig. 9** EDX spectrum of bright topsoil particles from sample 135 and a table presenting the detected elements (cts (counts) [keV])



Element	Weight %	Atom %	Comp %	Formula
Na K	0.20	0.18	0.27	Na <sub>2</sub> O
Mg K	0.11	0.09	0.18	MgO
Al K	0.59	0.45	1.11	Al <sub>2</sub> O <sub>3</sub>
Si K	44.01	32.32	94.15	SiO <sub>2</sub>
Fe K	0.97	0.36	1.25	FeO
In L	0.46	0.08	0.55	In <sub>2</sub> O <sub>3</sub>
Sn L	0.36	0.06	0.46	SnO <sub>2</sub>
Hg M	1.25	0.13	1.35	HgO
Tl M	0.66	0.07	0.69	Tl <sub>2</sub> O
O	51.40	66.26		
Total	100.00			





metals that occur at concentrations well above the response limit of the AAS and for individual sample-linear correlation analysis.

Anthropogenic, magnetic particles can be treated as tracers for the presence of heavy metals, based on the knowledge that they both originate from the same combustion source. Thus, soil magnetometry could serve as an efficient, 'first stage prescreening' method of

outlining pollution in soils and optimizing the selection of sampling points for the more time-consuming and expensive chemical methods.

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