

Investigation of Polluted Alluvial Soils by Magnetic Susceptibility Methods: a Case Study of the Litavka River

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Abstract

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Serious pollution of alluvial soils by a wide range of potentially toxic elements is usually observed in the valleys with mining and metallurgical industry. To outline areas of increased risk elements inputs, measurements of soil magnetic susceptibility can be used. This method is based on the measurement of the concentration of ferrimagnetic minerals of anthropogenic origin, mainly iron oxides, which are closely associated with risk elements such as heavy metals. The aim of this study is to examine the link between magnetic susceptibility of poorly drained Litavka River alluvial soils located in the mining/smeltering region of Příbram (Czech Republic) and the observed high concentration of risk elements in soils. Surface volume magnetic susceptibility and the vertical distribution of magnetic susceptibility in soil profiles were measured in situ. On the basis of field measurements, selected parts of soil cores were sampled for a detailed magnetic and chemical analysis. Our results demonstrate a statistically significant correlation between magnetic susceptibility and soil concentration of Cu, Pb, and Zn. The magnetically enhanced soil layer, relatively low frequency-dependent susceptibility, presence of Verwey transition, and scanning electron microscope observations suggested a prevalence of coarse magnetite/maghemite, likely of anthropogenic origin. However, the magnetic properties in-situ also reflected the natural soil conditions and soil processes. The high content of organic matter, varying reductive/oxidative condition, and vertical migration of magnetic phases were the main factors influencing the observed magnetic susceptibility values.

Keywords: environmental magnetism; Fluvisols; magnetite/maghemite; risk elements

Magnetic enhancement of topsoil and sediments due to the input of industrial magnetic particles and their connection with the elevated presence of some risk elements (RE) have been described by several authors (e.g. STRZYSCZ and MAGIERA 1998; HANESCH and SCHOLGER 2002). Magnetic susceptibility (MS) as a parameter reflecting concentration of ferrimagnets is widely used for assessment of soil pollution. This is based on the shared source and deposition of anthropogenic magnetic particles and RE. The incorporation of RE into the lattice and their adsorption on the surface of Fe oxides of an-

thropogenic origin is usually reported (HOFFMANN *et al.* 1996). Important sources of anthropogenic magnetic particles are fly ashes from the coal-fired power plants and metallurgical emissions. The input of anthropogenic magnetite/maghemite significantly increases the natural magnetic signal of soils given by the presence of different Fe oxides (goethite, lepidocrocite, maghemite, and magnetite). The effect of soil-forming processes and soil conditions on the presence of individual Fe phases is discussed by many authors (e.g. MAHER 1986; DEARING *et al.* 1996a).

Alluvial soils usually act as an important sink for a wide range of RE. Compared to areas with well-drained soils there is a lack of studies dealing with the magnetic monitoring of polluted waterlogged soils. However the results of magnetic studies of river sediments suggest that MS is a suitable parameter for RE detection (e.g. SCHOLGER 1998; ZHANG *et al.* 2011). Recently, YAN *et al.* (2011) and LU *et al.* (2012) have pointed to the water influenced Chinese paddy soils as a suitable target for magnetic proxy mapping in order to detect heavy-metal pollution. Therefore the aim of this study is to find the relationship between the presence of magnetic minerals, detected by MS measurements, and the concentrations of several RE in polluted soils from the Litavka River alluvium.

MATERIAL AND METHODS

The Litavka river valley is a part of the Příbram region situated in the Czech Republic, SW of Prague. The contamination of ecosystems in this area is closely connected with mining and ore processing lasting here already from the 14th century. In the study area, there are two main soils contamination sources: emissions from metallurgical industry and flood waters containing high amounts of contaminants due to repeated failures and release of effluents from metallurgical settling ponds during flood events. The detailed geochemistry of Litavka river soils and sediments is described in ETTLER *et al.* (2006).

The test site is located in one of the widest parts of the alluvial plain (close to Trhové Dušníky village) (Figure 1a). The soils were classified as Fluvisols (FL) and Gleyic Fluvisols (FLg). The land is covered with grass without agricultural use.

The values of surface volume MS (κ) were recorded in the field using a Bartington MS2D (Bartington, Oxon, UK). At the measuring points soil samples were collected from the topsoil (top 10 cm) using a soil auger. In total, 44 soil samples were collected and packed into plastic bags.

Vertical profiles of volume MS were measured using a susceptibility meter SM400 (ZH instruments, Brno, Czech Republic) (PETROVSKÝ *et al.* 2004). In total, 23 cores were taken using a ca. 40 cm long Humax soil corer (Martin Burch AG, Rothenburg, Switzerland), and stored in plastic tubes. The sampling scheme is depicted in Figure 1b.

Based on the susceptibility pattern measured in the soil profiles (verified by laboratory measurement with Bartington MS2C sensor (Bartington,

Oxon, UK), the selected parts of 15 soil cores were cut and the samples were used for further analysis. The thickness of the samples, separated from the plastic cores, was approximately 2–3 cm.

Soil samples were air dried, crushed, and passed through a 2-mm sieve prior to laboratory magnetic measurements and geochemical analyses.

Laboratory measurement of MS was carried out on the loose soil material packed in cylindrical plastic pots (10 cc) using a Bartington MS2 with dual frequency sensor (MS2B) and MFK1-FA Kappabridge susceptibility system (AGICO, Brno, Czech Republic). These values of MS are expressed as mass-specific MS (χ) taking into account the dry weight of the samples. Using an MS2B, the χ was measured at low (470 Hz) and at high (4700 Hz) frequencies. Next, in order to assess the significance of ultra-fine superparamagnetic magnetite of presumably pedogenic origin, the percentage frequency-dependent susceptibility ($\chi_{fd}\%$) was calculated according to DEARING *et al.* (1996b). With respect to the sensitivity of MS2B, the $\chi_{fd}\%$ was not determined for the subsoil samples with low values of volume MS (κ at low frequency $< 20 \times 10^{-5}$ SI) to avoid errors.

In order to determine the presence of magnetite/maghemite in soils, the thermomagnetic measurements of k were carried out on magnetic concentrates using KLY-3/CS-3 Kappa Bridge (AGICO, Brno, Czech Republic). Magnetic concentrates were obtained from homogenized soil samples, dispersed in isopropanol using permanent hand magnet separation. In addition, scanning electron microscopy (SEM) with wavelength dispersive spectroscopy (WDS) was performed on selected

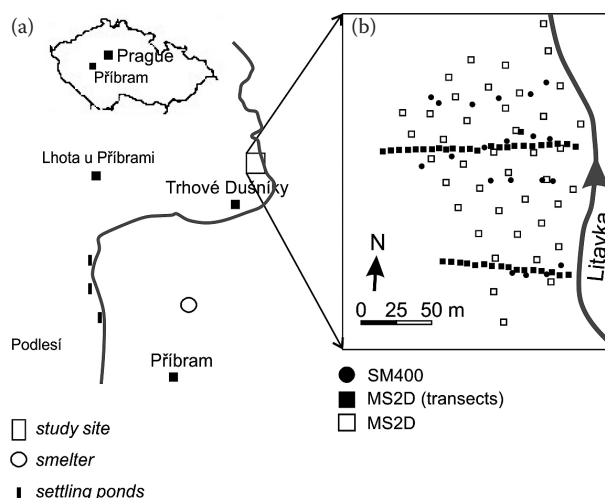


Figure 1. Location of study site (a); sampling scheme of field measurements (b)

magnetic concentrates from topsoil and magnetically enhanced samples.

The elements (Cd, Cu, Pb, Zn, and Fe) were extracted by cold 2M nitric acid (ratio of soil and reagent was 1:10, w/v). The amount of iron extracted with acid-oxalate (McKEAGUE & DAY 1966) and the iron extracted with dithionate-citrate was determined according to 6C2 procedure of the Soil Conversion Service, USDA stated in COURCHESNE and TURMEL (2008). The contents of elements in individual extractions were measured by flame atomic absorption spectrometry (FAAS) SpectrAA 280 FS (Varian, Mulgrave, Australia) under standard analytical conditions.

All obtained data were analyzed statistically using the software Statgraphics Centurion XV (StatPoint, Inc.). Relationships between the observed characteristics were tested by means of correlation analysis. The level of significance was set at $P < 0.05$ for all statistical analyses.

RESULTS AND DISCUSSION

Magnetic susceptibility. The values of surface κ range from 3.24×10^{-5} to 43.27×10^{-5} SI; average value is 18×10^{-5} SI. In comparison with other results of in situ κ measurements of polluted topsoil (e.g. HANESCH & SCHOLGER 2002; MAGIERA *et al.* 2008), the values at our study site are relatively low. The specific alluvial soils conditions strongly influenced by water and the input of pollution from non-ferrous smelter and from contaminated flood waters likely influenced the observed pattern of κ values. Although the grass was removed before measuring, the measured signal could be weakened by thick, poorly decomposed organic layer at the site due to the penetration depth characteristics of the MS2D probe (small response of layers below 6 cm). The higher values of surface κ close to the river bank suggest that the fluvial contamination with fine-grained waste material from ore process-

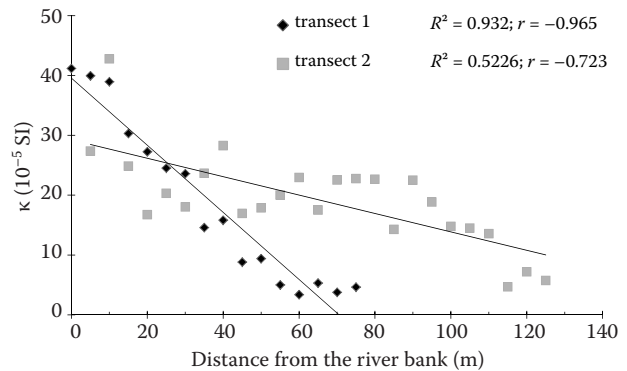


Figure 2. Relationship between the surface magnetic susceptibility measured in-situ and the distance of the sampling points from the river bank

ing can represent the main pollution source in that part of the Litavka alluvium (Figure 2).

On the basis of SM400 measurements two types of κ distribution in the soil profiles can be distinguished. The first group contains soil profiles with a significant peak of κ usually at the depth of 15–20 cm (Figure 3a). These profiles are located in the water-logged part of the study site and correspond to FLg. The second group of profiles shows relatively high values of κ in the uppermost layers (top 10 cm), only slightly increasing with depth (max. $\kappa 150 \times 10^{-5}$ SI), and then κ values gradually decrease in deeper horizons or are almost stable with depth (Figure 3b). This group represents mainly soil profiles located close to the river bank and corresponds to FL.

In-situ measurements suggest that there are differences between concentrations of strong ferrimagnetic minerals in topsoil and subsoil samples, reflected by different values of volume MS. The prominent peaks of κ in waterlogged profiles indicate the presence of a significant magnetically enhanced layer. To verify these observations laboratory measurements on selected parts of soil profiles were made.

The values of χ measured in topsoil (0–10 cm) were compared with values of χ from subsoil (lower

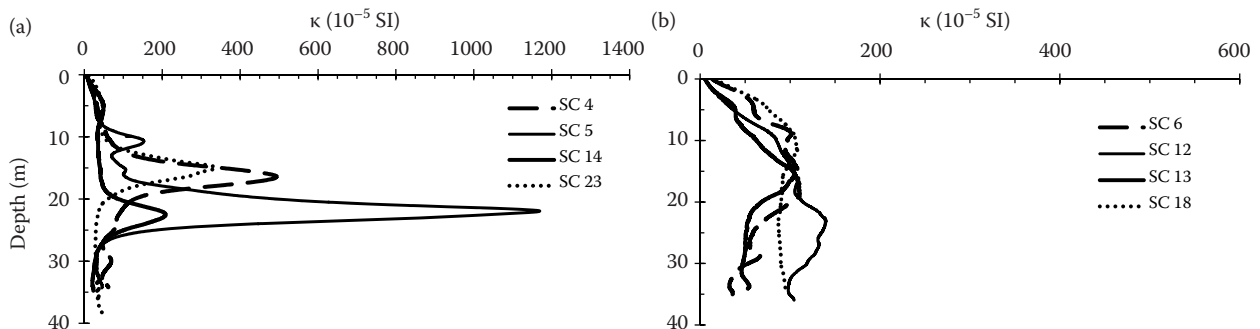


Figure 3. Vertical distribution of magnetic susceptibility values for selected soil profiles of Gleyic Fluvisols (a) and Fluvisols (b) measured by susceptibility meter SM400

than 30 cm). A significant difference between χ values of these groups is apparent from the box and whisker plot in Figure 4. According to the approach proposed by HANESCH and SCHOLGER (2002) and MAGIERA *et al.* (2006), the effect of alluvium lithology on magnetic properties of the studied soils is negligible.

The χ values of magnetically enhanced layers range between 78.4 to $1471.5 \times 10^{-8} \text{ m}^3/\text{kg}$; average and median values of χ are 362.4 and $156.7 \times 10^{-8} \text{ m}^3/\text{kg}$, respectively. These layers are located at the boundary between the lower, almost fully water-saturated layer, and the upper layer characterized by oxidative conditions. Below the layer with a high concentration of strong magnetic minerals, a rapid decrease in χ values was observed, most likely due to the prevailing reductive condition, where dissolution of magnetite is typical (e.g. MAHER 1986). Magnetically enriched layers in soil profiles associated with oxidative-reductive conditions caused by a fluctuating groundwater table were reported by RIJAL *et al.* (2010) and LU *et al.* (2012). Due to low values of frequency-dependent MS (discussed later) in these layers and no direct observation of small pedogenic magnetite/maghemite grains with SEM, formation of strong magnetic pedogenic minerals under these condition seems to be less probable here. The observed magnetic peak might be explained by a downward movement of the ferrimagnetic grains from the uppermost layer and their subsequent accumulation at layers of low permeability. This conclusion is in agreement with χ features observed in FL close to the river bank. Prevailing sandy material in these profiles enables the downward movement of magnetic particles, thus only a smooth decrease

and eventually almost stable magnetic signal in the whole profile was observed. Similar behaviour of magnetic grains in sandy material was reported by KAPIČKA *et al.* (2011).

Frequency-dependent magnetic susceptibility. The results of magnetic measurements of anthropogenic magnetic particles from different sources suggest that they are coarse-grained, and are therefore dominated by multidomain (MD) and stable single domain (SSD) grain sizes (PETROVSKÝ & ELLWOOD 1999). Compared to fine superparamagnetic (SP) particles usually of pedogenic origin, the coarser particles, such as MD and SSD, are frequency-independent (DEARING *et al.* 1996b). The average values of $\chi_{fd}\%$ of topsoil and magnetically enhanced layers are 1.93 and 1.92%, respectively. According to DEARING *et al.* (1996b) $\chi_{fd}\%$ below 2–3% indicates a negligible proportion of ultra-fine pedogenic ferrimagnetic minerals in soil samples. It is possible that the rather low values observed may be also affected by paramagnetic and canted antiferromagnetic minerals, which are often present in water-logged soils. Therefore only the samples with χ values exceeding the calculated median for the data set were chosen for $\chi_{fd}\%$ calculation. Although the presence of weakly magnetic Fe oxides in the studied soil samples is apparent (discussed later), their effect on $\chi_{fd}\%$ values is unlikely in the samples with high χ values. In this case, χ values are controlled dominantly by strong magnetic minerals such as magnetite/maghemite. Their presence is supported also by SEM observations, showing coarse spherical iron-rich particles (Figure 5), which are presumably of anthropogenic origin (MAGIERA *et al.* 2011).

Thermo-magnetic analysis of magnetic susceptibility. The presence of anthropogenic magnetite in topsoil samples from polluted areas can be determined by low-temperature k measurement (e.g. MAGIERA *et al.* 2008). Magnetite undergoes Verwey transition at about -150°C . Magnetic concentrates prepared from the raw samples of topsoil and magnetically enhanced layers exhibit a prominent Verwey transition, indicating the presence of coarse MD magnetite, likely of anthropogenic origin (Figure 6a). In comparison, samples from the subsoil exhibited a decrease in χ value with increasing temperature, pointing to a significant presence of paramagnetic minerals in these samples (data not shown).

The heating/cooling runs of magnetic concentrate from topsoil and magnetically enhanced layers

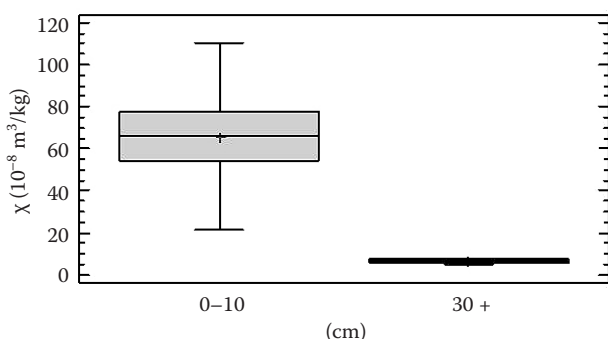


Figure 4. Box-and-whisker plots for magnetic susceptibility of the topsoil and subsoil

Box-whisker plots show minimum, maximum, median, lower quartile, and upper quartile information for groups of data

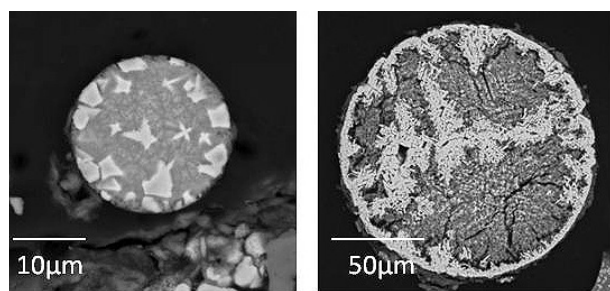


Figure 5. Scanning electron microscopy (SEM) images of Fe-rich spherules found in magnetic concentrates separated from topsoil and magnetically enhanced layer

exhibit a rapid decrease in χ ranging 570–590°C (Figure 6b). The identification of Fe oxides is based on the determination of Curie temperature (T_c) that is characteristic for individual oxides. The T_c of studied samples are close to 585°C (assessed according to PETROVSKÝ & KAPÍČKA 2006) pointing to the presence of magnetite. In some cases, a shift to higher Curie point temperature is likely due to a more significant contribution of maghemite.

Relationships between magnetic susceptibility and risk elements in alluvial soils. Overall the concentrations of studied risk elements (RE) in the whole profiles significantly exceeded the pollution limits set by Czech legislation (Table 1). These results are in agreement with high contamination observed in the Litavka river region (ETTLER *et al.* 2006).

Firstly, the relationship between in-situ measured surface k and the corresponding laboratory measured χ of collected topsoil samples with contents of Cd, Cu, Pb, and Zn was analyzed by correlation analysis (Table 2). In the case of in-situ measurements, statistically significant relationship was found only for Pb and Zn. More precise laboratory

Table 1. Comparison between average risk elements concentration in topsoil (0–10 cm) and pollution limit set by Czech legislation (in mg/kg) (Czech Regulation 13/1994; Ministry of the Environment of the Czech Republic)

| | Cu | Cd | Pb | Zn |
|----------------------|-------|-------|---------|---------|
| topsoil ($n = 53$) | 68.43 | 30.02 | 2321.45 | 2743.15 |
| pollution limit | 50.00 | 1.00 | 70.00 | 100.00 |

measurements, including correction for mass of the samples also reveal a significant relationship between χ and concentration of Cu and Fe. The moderate strength of the observed correlation can be explained by the presence of more than one contamination sources (STRZYSCZ & MAGIERA 1998; HANESCH & SCHOLGER 2002). According to results of field measurements of κ in deep soil profiles, the migration of ferrimagnetic particles can be assumed, which cannot be reflected by measurements of surface κ . Therefore the concentration of RE was determined also in deeper soil layers and their connection with observed magnetic features was studied.

The values of χ at the top of the soil cores correlate in a similar way with Cu, Fe, Pb, and Zn as

Table 2. Correlation coefficients between studied elements and magnetic susceptibility for topsoil samples are significant at the 0.05 significance level

| $n = 44$ | Cd | Cu | Pb | Zn | $\log(\text{Fe})$ |
|-----------------|------|-------|-------|-------|-------------------|
| κ (MS2D) | n.s. | n.s. | 0.452 | 0.455 | n.s. |
| χ (MS2B) | n.s. | 0.465 | 0.454 | 0.465 | 0.419 |

n.s. – nonsignificant correlation; κ (MS2D) and χ (MS2B) refer to in-situ and/or laboratory measurements

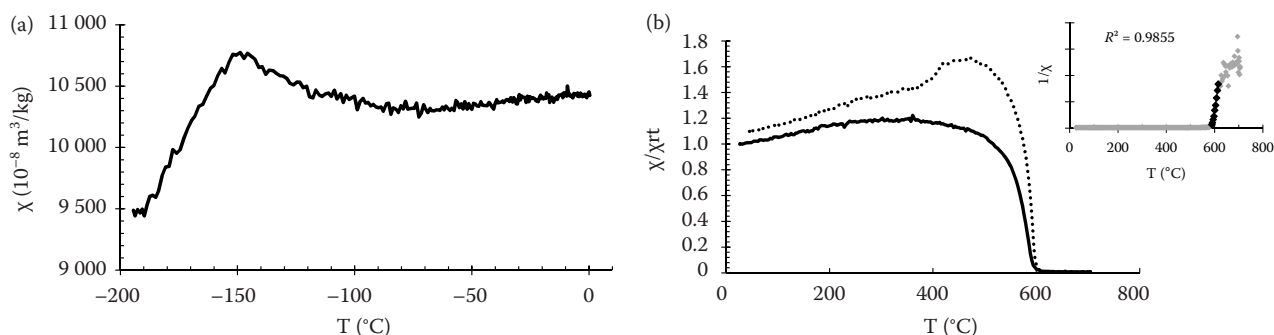


Figure 6. Low-temperature dependence of mass-specific magnetic susceptibility of magnetic concentrate separated from magnetically enhanced layer (a) and temperature dependence of mass-specific magnetic susceptibility of magnetic concentrate from topsoil (b)

MS-T curves are normalized by the initial susceptibility measured at room temperature; the estimation of Curie temperature (T_c) is depicted; full line denotes heating run, dashed line denotes cooling run

above-mentioned samples from topsoil. In the case of subsoil, statistically significant correlation coefficients were found between χ and Cu ($r = 0.675$) and Pb ($r = 0.747$). Correlations for samples from the middle part of the soil profiles were not statistically significant. To test the relationship between the magnetically enhanced layer and the RE content, the data were assessed separately for the individual soil units. Although the relationship was not statistically significant, a trend of increasing RE content at the depth of 15–20 cm in FLg was observed for Cd, Zn, and Fe (Figure 7).

Sorption and co-precipitation of RE with the Fe oxide/oxyhydroxides are considered as one of the most important ways of contaminants binding in alluvial soils (HUDSON-EDWARDS & TAYLOR 2003). The concentration of the studied RE showed the correlation of moderate strength with Fe content in topsoil (Table 3). No correlation was observed in the subsoil. The efficiency of the leaching method to release Fe embedded in Al-Si matrix is reflected in the observed correlation between Fe and RE and

Table 3. Correlation coefficients between risk elements and iron concentration in topsoil samples are significant at the 0.05 significance level

| $n = 48$ | Cd | Cu | Pb | Zn |
|------------|--------|--------|--------|--------|
| Fe (mg/kg) | 0.4043 | 0.4469 | 0.4988 | 0.4881 |

also between Fe and χ . Relatively weak correlations between Fe content and χ in topsoil were reported e.g. by DEARING *et al.* (1996a). According to MAHER (1998) this effect can be caused by a higher amount of weakly magnetic Fe oxides (e.g. goethite, ferrihydrite) compared to a usually low content of strong ferrimagnetic minerals in soils. The contribution of poorly and well crystalline Fe phases in the soil samples are estimated according to the Fe amount extracted by acid-oxalate (Feo) and dithionate-citrate (Fed), respectively. Relatively high average values of Feo/Fed ratio observed along the profiles (0.7 in 0–10 cm; 0.65 in 10–30 cm; 0.5 lower than 30 cm) suggest a significant contribution of poorly crystalline Fe phases like ferrihydrite in the studied soil samples. The observed correlation of χ with RE and iron can thus be lowered due to the occurrence of paramagnetic and/or antiferromagnetic minerals (such as ferrihydrite, goethite) which partially decrease the bulk magnetic signal of soil samples.

CONCLUSIONS

Correlations of moderate strength were observed between mass-specific magnetic susceptibility and concentration of Cu, Pb, and Zn in heavily polluted alluvial soils of the Litavka floodplain. Significantly higher values of magnetic susceptibility observed in topsoil compared to the values of subsoil accompanied by high concentrations of risk elements pointed to an anthropogenic magnetic anomaly. Two types of magnetic susceptibility patterns were observed in profiles of studied Fluvisols and Gleyic Fluvisols, likely to be reflecting the vertical migration of small ferrimagnetic particles under the different pedo-hydrological condition of these profiles. The presence of multi domain and stable single domain magnetic particles of anthropogenic origin was confirmed in magnetically enhanced soil layers. The efficiency of magnetic susceptibility measurements with respect to monitoring the anthropogenic input of risk elements into the Litavka alluvial soils is partially limited due to the specific water regime which influenced the movement of ferrimagnetic particles in soils and also affected the dissolution of strongly magnetic oxides. The temporal effect of the fluctuating ground water table and the accumulation of anthropogenic

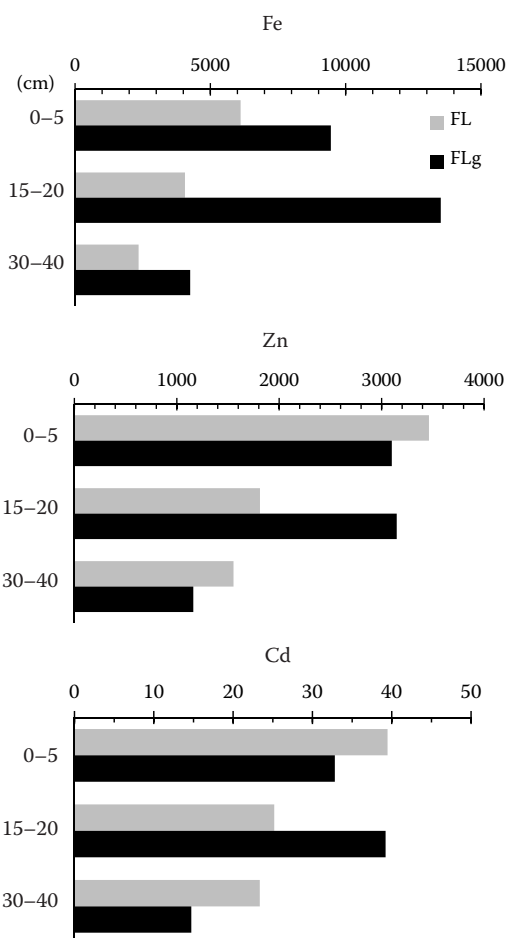


Figure 7. The average concentrations of Cd, Zn, and Fe (in mg/kg) along the soil profiles of Gleyic Fluvisols (black bars) and Fluvisols (grey bars)

fine-grained metal-bearing particles on soil magnetic properties in the area of the alluvial plain should be subjected to detailed study.

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