Particular aspects of environmental impact of potentially risk elements from airborne particulate matter

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ABSTRACT

Two simple experiments were carried out to demonstrate two possible ways of environmental impact of element contents in airborne particulate matter. In the first part of the experiment, the response of three rural dust samples applied into the soil were assessed in pot experiment to observe uptake of As, Cd, Pb, and Zn by aboveground biomass of oat (*Avena sativa* L.). Although the element contents in dust samples exceeded significantly total element contents in soil, the element content in plants was not affected by single-rate soil amendment with rural dust sample. Soil sorption capacity and neutral pH of the experimental soil (7.0) was sufficient for immobilization of the elements. However, potentially mobilizable portions (0.005 mol/l DTPA extractable) of elements significantly increased (Cd by 116%, Pb by 39%, Zn by 50%) in the treated soil, which suggests a possible release of these elements in long-term horizon. On the contrary, high percentages of potentially toxic elements (Cd, Zn, Ni) in the most mobile (exchangeable) fractions were determined in the second part of investigation in two urban dust samples collected in Prague Letná automobile tunnel, and Prague subway station Museum. These results suggest possible direct impact of mobile, and thus potentially bio-available, element fractions on human environment. The results of both particular experiments cannot give complete information concerning behavior of harmful pollutants in airborne particulate matter and their influence on human health. They can however indicate two of possible ways of environmental pollution with this material. Yet, it would require a more detailed investigation in future.

Keywords: airborne particulate matter; potentially risk elements; soil; oat; plant-availability; bioavailability; sequential extraction

An exposure of the population to harmful pollutants occurring in an internal environment – i.e. an environment with no direct connection to the external atmosphere, occupied permanently or temporarily by people – is a matter of global concern (Anonymous 1992, Byrne et al. 1993). The subway embodies a highly unique combination of different sources of contamination with harmful pollutants occurring in an internal environment. In the Czech Republic a large-scale project *System of Monitoring the Environmental Impact on Population Health* has been pursued for evaluation of outdoor air quality with regard to an adverse effect of elevated concentrations of toxic components on human health (Anonymous 1994). Monitored pollutants include a group of analytes like arsenic, cadmium, chromium, manganese, nickel, lead, and zinc. More than 30 laboratories participated in that project because the system of quality control of chosen elements had to be developed. Test sample preparations for proficiency testing were already described (Sysalová et al. 2002). The monitoring project does not explain the analyte forms and their mobility and thus the system of quality control was developed for the determination of total analyte concentrations. Now, there is a new research program in development directed at harmful pollutants occurring in an internal urban environment or in public transportation, with respect to the assessment of the mobility and potential bioavailability of chosen toxicologically important elements already established in the monitoring program (As, Cd, Cr, Mn, Ni, Pb, and Zn).

The total contents of some potentially toxic elements in urban particulate matter as affected by site, traffic level, seasonal variability, meteorological parameters (air humidity and temperature, wind direction, rainfall), and particle size were examined by using various instrumental analytical techniques (Varrica et al. 2003, Deboudt et al. 2004). Seasonal variations of urban dust precipitation and chemical dust composition were studied by Norra and Stueben (2004). Annual courses of metal depositions correlated positively with the

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dust precipitations and negatively with the trace metal concentrations in dusts. The movement of heavy metals from source to deposit of urban dusts and sediments was investigated by Charlesworth and Lees (1999). They concluded the necessity of an application of sequential leaching of these materials to determine the changes during movement from source to deposit as well as to determine the environmentally harmful metal species and fractions. As for modern medical research, the total content of elements in airborne particles is not so important because it does not specify various adverse health effects, neither does it explain an anthropogenic contribution to sources of air pollutions (Davidson et al. 1998, Dabek-Zlotorzynska et al. 2002). For such studies or monitoring there is the need to determine the reactivity of individual analytes as well as the necessity to quantify specific analyte forms (species) and to estimate an environmental mobility and a possible bioavailability of investigated elements in air particulate matter. The mobility of an element is usually related, among other factors, to its chemical properties and the toxicity mainly to its oxidation state. Generally, the most frequently used method to assess the distribution of elements in environment of solid samples (soil, sediment, sewage sludge, urban dust, atmospheric deposit, coal fly ash, etc.) is the chemical fractionation of elements. This is a process performed by a sequence of different leaching or selective extraction procedures that determine element forms as an important factor in controlling the health impact. The basic method of sequential leaching was developed by Tessier et al. (1979) for sediments; at present the BCR (now the Institute for Reference Materials and Measurements) scheme is usually applied to different types of solid samples (Quevauviller et al. 1993).

In addition to the direct environmental impact of airborne particulate matter on human environment, an interdisciplinary approach is required to assess the influence of pollutants contained in this matter on topsoils and vegetation (Varrica et al. 2003, Norra and Stueben 2004). Viard et al. (2004) investigated the induction of contamination in localities with heavy traffic in the vicinity of highways, where the levels of metals in surface soil and plants decreased with increasing distance from the highway with maximum levels between 5 and 20 m.

The long-term effect of soil acidification due to the atmospheric deposition of metals on plant-availability was described as well (Blake and Goulding 2002). The investigation of relationships between airborne particulate matter and metal concentrations in leaves of common plants (*Taraxacum officinale*, *Rumex acetosa*) in field conditions showed weak correlations because of different concentrations of elements in individual soils, different soil properties and plant-availability of individual elements (Keane et al. 2001, Wang et al. 2003).

In our research, two experiments were carried out to demonstrate two possible environmental impacts of element content in airborne particulate matter: i) possible contamination of human food chain via soil pollution followed by plant uptake of these elements; in this case the influence of the addition of three rural dust samples into the soil on element uptake by oat biomass (*Avena sativa* L.) was investigated in pot experiment and ii) mobility of elements in two samples of urban dust samples by sequential leaching procedure was studied to assess their possible direct health impact.

MATERIAL AND METHODS

Sampling of airborne particulate matter

Rural dust samples were collected at three field locations: Červený Újezd (small village in Central Bohemia), Přestanov (village in Northern Bohemia with elevated level of air pollution), and Suchdol (Prague suburb). Samples were collected from the surface of 4 m^2 into plastic jars positioned 1 m above the ground surface to eliminate pollution from ground caused by wind. To obtain a sufficient quantity of dust for the pot experiment, samples were collected over the period of three years, air dried, and homogenized before analyses. Samples from individual sites were mixed in ratio 1:1:1 and applied into the soil.

Urban dust samples were collected in sedimentation chambers of ventilation systems of Prague Letná automobile tunnel and Prague subway station Museum and processed as follows: materials were irradiated with photons of ^{60}Co at 25 kGy for sterilization and sieved to obtain fractions with particle size < 50 meshes. These fractions were jet-milled twice and finally homogenized in V-shape blender for 48 h. Details of the whole procedure were already published by Kučera et al. (2000) and Sysalová et al.(2002). Particulates in final fractions were smaller than 400 meshes. Homogeneity tests and elemental characterization of urban dust samples were carried out using instrumental neutron activation analysis (INAA), proton induced *X*-ray emission (PIXE) and atomic absorption spectrometry (AAS); for comparison some analyses were performed by inductively coupled plasma optical emission spectrometry and mass spectrometry (ICP-OES and ICP-MS), respectively, in selected expert laboratories. The homogeneity of dust samples was tested by determining selected elements and by using different amounts of approximately 100, 50, 20, 10 and 0.5 mg (estimated from the size of beam and energy of protons). Calculated relative standard deviations caused by inhomogeneity were low for both samples (less than 8%) even by PIXE, in which only a small mass of sample was analyzed.

Pot experiment

Luvisol characterized by pH_{KCl} 7.0, cation exchange capacity 280 mmol/kg, and content of oxidizable carbon 1.35%, was investigated in the pot experiment. Soil was treated by N, P, K in the amount representing 1.5 g N, 0.44 g P and 1.1 g K per pot. Nutrient status of the soil was as follows: available P 331 mg/kg, available K 449 mg/kg, available Mg 163 mg/kg, and available Ca 2903 mg/kg. Available nutrient contents were determined by Mehlich II soil extraction procedure (Zbíral 2000). Oat var. Pan was cultivated in pots filled with 5 kg of air-dried soil, for which three replications of each treatment were used. Thirty five grams of mixed rural dust sample was applied per pot and thoroughly mixed with the experimental soil. The plants were watered by deionized water and soil moisture was kept at 60% of its maximal water holding capacity. Plants were harvested in wax grain maturity and after harvest the above ground biomass was checked for fresh and dry biomass (dried at 60°C), ground and analyzed for As, Cd, Pb, and Zn contents. Soil samples were taken from the bulk of the oat plants immediately after the harvest, then air-dried at ambient temperature, ground in a mortar, passed through a 2-mm plastic sieve, and analyzed.

Analytical methods

To determine the total analyte content in urban dust samples by ICP-OES and GF AAS methods, samples of 20 mg were digested in a mixture of high purity concentrated nitric acid, hydrofluoric acid and hydrogen peroxide (5 + 1 + 1) in a microwave oven for 10 min at maximum power. The resulting clear solution was evaporated almost to dryness at 170°C on a hot plate coated with PTFE layer. The residue was dissolved and stored in 5% (v/v) HNO_3 in 10 ml plastic tubes at +4°C until measurement. Operations were carried out in a clean room, class 100 working environment. Total element concentrations in rural dust samples and soils were determined in digests obtained by two-step decomposition as follows: 0.5 g of sample was decomposed by dry ashing in a mixture of oxidizing gases ($O_2 + O_3 + NO_x$) in an Apion Dry Mode Mineralizer (Tessek, Czech Republic) at 400°C for 10 h; the ash was then decomposed in a mixture of HNO_3 + HF, evaporated to dryness at 160°C and dissolved in diluted *aqua regia* (Száková et al. 1999). The plant samples were decomposed by dry ashing procedure as follows: An aliquot (~1 g) of dried, thoroughly grounded whole aboveground biomass was weighed to 1 mg into a borosilicate glass test-tube and decomposed in a mixture of oxidizing gases ($O_2 + O_3 + NO_x$) at 400°C for 10 hours in Dry Mode Mineralizer Apion (Tessek, Czech Republic). The ash was dissolved in 20 ml of 1.5% HNO_3 (electronic grade purity, Analytika Ltd., Czech Republic) and kept in glass tubes at ambient temperature until measurement (Miholová et al. 1993).

For determination of plant-available portions of elements in soil samples, aliquots of fresh soil samples were extracted with 0.01 mol/l aqueous $CaCl_2$ solutions in ratio 1:10 (w/v) for 6 hours (Novozamsky et al. 1993). Potentially mobilizable portion of elements in soils were released by extraction with 0.005 mol/l solutions of dietylentriaminopentaacetic acid (DTPA) (+ 0.1 mol/l trietanolamine + 0.01 mol/l $CaCl_2$) in ratio 1:2 (w/v) for 2 hours (Lindsay and Norwell 1978). Sequential extraction procedure was done according to the BCR recommendations (Quevauviller et al. 1993) modified by Dabek-Zlotorzynska et al. (2003). Samples (100 mg) were weighed into centrifuge tubes and sequentially extracted by following agents: i) exchangeable fraction by 0.11 mol/l CH_3COOH in ratio 1:50 (w/v) for 16 h; ii) fraction bound to Fe/Mn oxides and carbonates by 0.1 mol/l $NH_2OH.HCl$, in ratio 1:50 (w/v), pH = 2 for 16 h; iii) fraction bound to organic matter and sulfides by 8.8 mol/l H_2O_2, in ratio 1:20 (w/v) evaporated to moist residue at 80°C followed by 1 mol/l CH_3COONH_4, in ratio 1:50 (w/v), pH = 2 for 16 h; iv) residual fraction was calculated as the difference between the total element concentration in samples and the sum of the three fractions above. The supernatant phase of each step was obtained after centrifugation at 2500 rpm (e.g. 325 × g) for 15 min, solutions were acidified by 0.1 ml 65% HNO_3, completed to 10 ml by water and stored in a refrigerator (+4°C) until measurements. At the end of each step the washing procedure of residual sample was carried out by addition of 10 ml of ultra pure water, shook 15 min and centrifuged. Supernatant was decanted and discarded. Procedural blanks were prepared with each batch of extractions.

The elements Cd, Cr, Ni, Pb, Mn, and Zn in urban dust samples were determined by ICP-OES, Varian VistaPro equipped with autosampler SPS-5 (Australia). For the determination of As in these samples a Perkin-Elmer AAS, model 4000 (Germany), equipped with a graphite furnace HGA 500 and an autosampler AS-40 was used. The element contents in the digests and extracts of rural dust samples, soils, and plant biomass were determined by AAS in flame (Zn, Mn), flameless (Cd, Pb, Cr, Ni), and hydride generation (As) measurement modes by using Varian SpectrAA-300 equipped with autosampler SPS-5, graphite furnace GTA-96, and continuous hydride generator VGA-76. For quality assurance of analytical data certified reference materials RM 12-02-03 Lucerne, BCR-144 Sewage sludge, and NIST 1648 Urban particulate matter were analyzed. Kruskal – Wallis test (Statgraphics plus 5.1) was applied for statistical evaluation of the data ($\alpha = 0.05$).

RESULTS AND DISCUSSION

The total element contents in experimental soil, rural dust samples (Červený Újezd, Přestanov, Suchdol), and urban dust samples (Letná and subway) are summarized in Table 1. Expectably, element contents in airborne particulate matter exceeded significantly the soil element contents for As (except for rural dust from Červený Újezd), Cd, Cr, Pb, and Zn. The results did not show any significant differences between a rural dust from polluted area (Přestanov) compared to an unpolluted one (Červený Újezd). Moreover, as for Cd, Pb, and Zn, the contents in urban dust samples were much higher than in the rural dust samples. Concerning chromium the differences among all the dust samples were relatively low whereas soil Cr content was 4-5-fold lower than in the dusts. For manganese, high contents of this element were observed in urban dusts while its contents in rural dusts were comparable to the soil. As opposed to that, the highest contents of nickel were found in rural dusts and urban dusts were comparable to the soil. Contrary to these results Romic and Romic (2003) reported five times higher nickel contents in urban soils near Zagreb (Croatia) compared to agricultural ones, whereas for Cd, Pb, and Zn the highest levels of these elements corresponded to our research.

The results of the pot experiment are summarized in Table 2. Illustrated by Figure 1 as well, the single-rate soil amendment with rural dust sample did not lead to a significant change of element contents in the oat biomass. The plant-available (0.01 mol/l $CaCl_2$ extractable) element contents in soil were not significantly affected by soil amendment either. Relatively high soil sorption capacity and high pH level of the soil (7.0) were sufficient for immobilization of elements at the treated pots for the whole vegetation period. Although not significant, plant-available arsenic contents in soil tended to increase in treated pots. The arsenic mobility in soil is affected by complex of soil physicochemical parameters where soil organic matter plays an important role. The organic matter contained in the dust samples could therefore provoke the changes in organic matter composition in soil. As for the adsorption sites, competitive adsorption of dissolved organic matter and arsenate was observed by Lin et al. (2002). However, Carbonell-Barrachina et al. (1999) found

Table 1. Total element contents in urban dust samples (Letná and subway), rural dust samples (Č. Újezd, Přestanov, Suchdol), and experimental soil (mg/kg)

Sample		As (mg/kg)	Cd (mg/kg)	Cr (mg/kg)	Mn (mg/kg)	Ni (mg/kg)	Pb (mg/kg)	Zn (mg/kg)
Soil	mean	18.4	0.191	40.5	367	67.3	23.3	98.9
	RSD (%)	32.8	19.0	14.4	10.1	11.9	1.6	14.0
Č. Újezd	mean	16.7	0.471	161	321	449	76.4	287
	RSD (%)	24.0	15.7	15.8	15.4	2.0	13.9	3.9
Přestanov	mean	47.1	1.17	126	165	188	475	694
	RSD (%)	8.4	16.8	17.3	13.2	4.5	20.1	10.5
Suchdol	mean	63.9	0.270	149	235	476	369	624
	RSD (%)	11.8	21.4	13.4	10.4	4.8	18.1	6.2
Letná	mean	25.4	2.00	182	1005	64.5	1762	1284
	RSD (%)	2.9	2.78	4.57	4.68	10.4	3.00	4.46
Subway	mean	71.5	9.59	204	1415	111	749	1724
	RSD (%)	3	2.21	0.65	0.01	3.62	1.88	0.24

RSD = relative standard deviation (%)

Table 2. Element contents in oat biomass, in $CaCl_2$ extractable portion in soil, and in DTPA extractable portion in soil at the soil amended by rural airborne particulate matter

Oat biomass	As (mg/kg)		Cd (mg/kg)		Pb (mg/kg)		Zn (mg/kg)	
	mean	*SD*	mean	*SD*	mean	*SD*	mean	*SD*
Control	0.277	0.032	0.217	0.042	0.614	0.156	44.2	7.1
Treated	0.261	0.033	0.222	0.080	0.500	0.072	44.5	5.9
$CaCl_2$	As (mg/kg)		Cd (mg/kg)		Pb (mg/kg)		Zn (mg/kg)	
	mean	*SD*	mean	*SD*	mean	*SD*	mean	*SD*
Control	0.017	0.004	0.005	0.002	< 0.007	–	0.057	0.012
Treated	0.035	0.014	0.008	0.004	< 0.007	–	0.057	0.015
DTPA	As (mg/kg)		Cd (mg/kg)		Pb (mg/kg)		Zn (mg/kg)	
	mean	*SD*	mean	*SD*	mean	*SD*	mean	*SD*
Control	0.010	0.004	0.042	0.016	1.49	0.04	2.23	0.17
Treated	0.013	0.002	0.084*	0.006	2.07*	0.24	3.35*	0.08

*significantly different element content in treated variant as compared to control ($\alpha = 0.05$)
SD = standard deviation, $n = 3$

that an organic amendment rich in sulfur compounds can reduce the risk of As pollution under anoxic conditions by precipitation of this element as insoluble sulfides. Evidently, the behavior of arsenic affected by soil organic matter depends on individual experimental conditions and cannot be unambiguously predicted. The soil extraction by 0.005 mol/l DTPA can be applied for assessment of potentially mobilizable portions of elements. The results (Table 2) showed a significant increase of element contents (Cd by 116%, Pb by 39%, Zn by 50%) at treated soil. These observations indicate that the possibility of release of these elements into soil solutions cannot be excluded in next vegetation periods. In the case of arsenic, DTPA extracts seems to be ineffective for release of mobilizable portion of this element (Száková et al., 2000). The effects of soil properties on element mobility in soils polluted by atmospheric deposition were investigated by Blake and Goulding (2002). Because the long-term atmospheric deposition can lead to soil acidification they assessed predominantly the effect of soil pH. Their long-term observations suggested that the increase in exchangeable lead resulted more from increased total Pb in soil via repeated atmospheric deposition than from Pb mobilization by changing soil pH. Cadmium and zinc mobility was more closely connected to the soil acidity. Although in our case the single-rate soil amendment did not indicate a direct contamination of crop production, long-term atmospheric deposition to the soil followed by changing soil characteristics can release potentially mobilizable portions of elements into the soil solution.

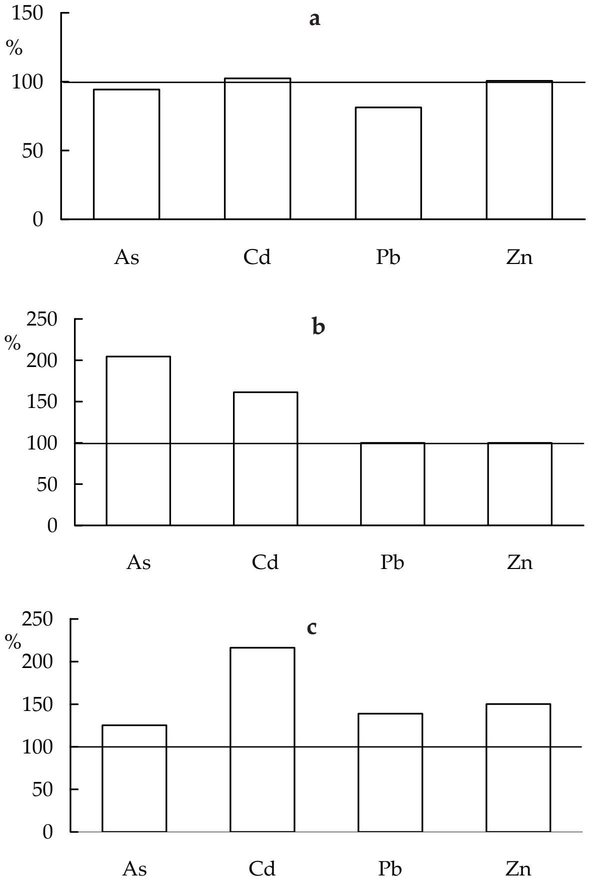

Figure 1. Relative changes of element contents in oat biomass (a), in $CaCl_2$ extractable portion in soil (b), and in DTPA extractable portion in soil (c) at the soil amended by rural airborne particulate matter (100 % represents the control treatment)

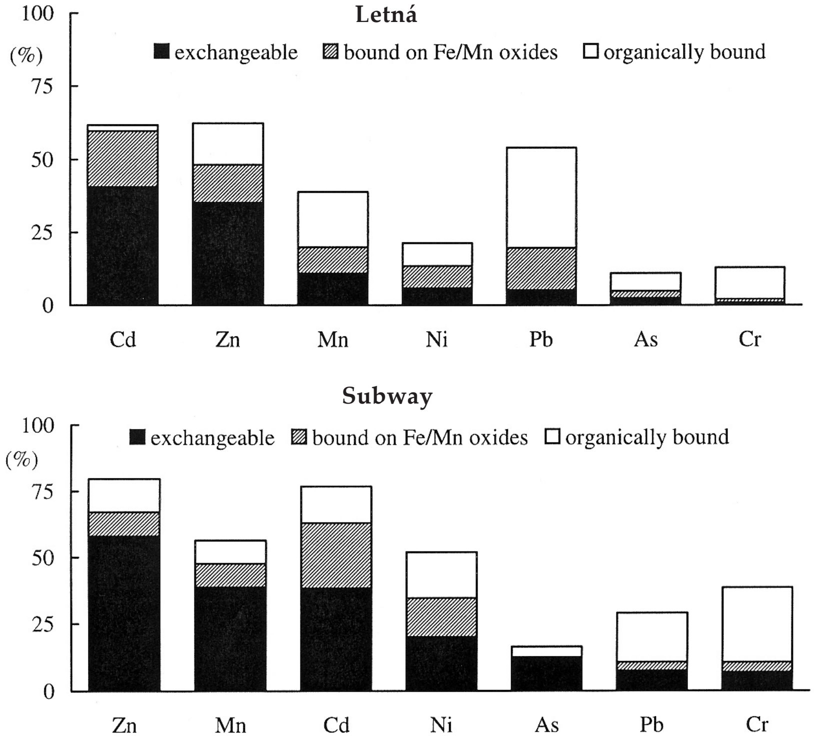

Figure 2. Relative element contents in extractable fractions of urban dust samples according to the sequential extraction procedure (% of total element contents in these samples)

Mobility of elements in urban dust samples determined by sequential extraction procedure differed according to sample sources and individual elements. Differences between both analyzed samples are illustrated in Figure 2, presenting relative values for the chemical distribution of each analyte based on the sequential leaching procedure. In exchangeable fraction more than 50% of Zn, 38% of Cd, and about 20% of Ni were found in the subway sample. In the road tunnel sample Letná even 40% of Cd, and 35% of Zn were found in the exchangeable fraction. 24% of cadmium was found in the subway sample in fraction associated with Fe/Mn oxides, whereas in the road tunnel dust it was only 19% in this fraction. The other element associated with environmentally mobile fractions is lead, mostly bound to organic matter and sulfides (34% in the road tunnel dust). Using similar sequential extraction procedure of urban dust samples from Sevilla (Spain), Fernández-Espinosa and Ternero-Rodriguez (2004) determined 31% of Ni, 21% of Cd, and 36% of Zn in exchangeable fraction and almost 50% of Pb bound to fraction that represents elements bound to organic matter and sulfides. These results suggested, regardless of the differences between extraction procedures and sampling site, similar character of distribution of risk elements in urban dust samples. On the other hand, elements like As, Cr, Ni are mostly accumulated in the environmentally immobile fraction – more than 55% of each of them are probably incorporated into individual stable compounds (oxides, phosphates, silicates, etc.) and hence not bio-available. Rather high levels of some toxically important elements were found in the subway particulate matter sample. Their occurrence in environmentally mobile fractions, potentially bio-available, could represent certain health risk for passengers and employees, staying in Prague subway for longer time. It has to be considered that dust deposited in ventilation chambers is mixed by piston effect caused by movement of trains; the secondary dust is generated and respirable particles containing toxic elements can penetrate into a human body. Depending on the particle size, toxic forms of analytes can be transported as far as to individual organs.

Whereas a single-rate application of rural dust sample into the soil did not lead to the pollution of oat biomass by potentially toxic elements, there still exists a risk of contamination of crop production by long-term atmospheric deposition. The problem is more serious in the case of soils with lower sorption capacity and pH level. On the contrary, the high percentages of mobile and potentially bio-available fractions of toxic elements in urban particulate matter from both external (automobile tunnel) and internal (subway) environment make this material highly dangerous for human health. The experiments presented in this paper cannot lead to a complex characterization of environmental impact of airborne particulate matter but suggests some of possible aspects of this problem and indicates possible approaches for evaluation of this source of harmful pollutants.

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ABSTRAKT

Vybrané aspekty dopadu potenciálně rizikových prvků obsažených v pevném spadu na životní prostředí

Byly provedeny dva jednoduché experimenty s cílem demonstrovat dva možné způsoby dopadu prvků obsažených v pevném spadu na životní prostředí. Nejprve byl v nádobovém pokusu sledován vliv aplikace do půdy tří vzorků pevných spadů odebraných v zemědělské krajině na příjem As, Cd, Pb a Zn nadzemní biomasou senážního ovsa (*Avena sativa* L.). Obsah sledovaných prvků ve vzorcích pevných spadů významně převýšil jejich obsah v půdě, ale příjem prvků rostlinou nebyl po této jednorázové aplikaci pevných spadů významně ovlivněn. Sorpční kapacita pokusné půdy a její neutrální hodnota pH (7,0) byly dostačující pro imobilizaci těchto prvků. Je však třeba zdůraznit, že potenciálně mobilizovatelné obsahy prvků v půdě (tj. extrahovatelné 0,005 mol/l DTPA) se po aplikaci pevných spadů významně zvýšily (v případě Cd o 116 %, Pb o 39 %, Zn o 50 %). Lze tedy očekávat, že tyto prvky by se mohly mobilizovat v dlouhodobém časovém horizontu. Odlišná situace byla zaznamenána u vzorků městského prachu odebraného v pražském letenském tunelu a ve stanici pražského metra Muzeum. V těchto vzorcích byly stanoveny vysoké podíly některých prvků (Cd, Zn, Ni) v nejmobilnějších frakcích. Existuje tedy možnost přímého dopadu těchto mobilních (a tedy biopřístupných) frakcí rizikových prvků na životní prostředí člověka. Oba pokusy provedené v rámci této studie nemohou poskytnout vyčerpávající informaci o chování polutantů v pevném spadu a jejich vlivu na lidské zdraví. Naznačují pouze dva možné způsoby kontaminace životního prostředí tímto spadem a potvrzují, že tato problematika zasluhuje podrobný a systematický výzkum.

Klíčová slova: pevný spad; potenciálně rizikové prvky; půda; oves; přijatelnost rostlinami; biopřístupnost; postupná extrakce

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