

Environmental study of gravitation dust sediment and soil

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Abstract: In presented work are results of the complex study of significant solid environmental samples: gravitation dust sediments (industrial pollutants, potential source of risk elements input to soil) collected in typical industrial region and as urban dust, and soil (component of the environment, potential source of risk elements input to food web) collected in typical agricultural region. In the first step of the study, we are focused on the study significant chemical properties of such as phase composition, content of organic and inorganic carbon of the dust and soil samples. In the next step of study, fractionation analysis was used on the evaluation of the mobility of chosen risk elements (Cu, Pb, Zn, Ni) in the studied samples. The single-step extractions were carried out by reagents: 1 mol dm⁻³ NH₄NO₃ for isolation of mobile forms (water-soluble, exchangeable), 0.05 mol dm⁻³ EDTA and 0.43 mol dm⁻³ CH₃COOH for isolation of mobilizable forms (water-soluble, ion-exchangeable, organic, carbonate), and 2 mol dm⁻³ HNO₃ for isolation of all releasable forms. The content of elements in extracts of single-step extractions was determined by FAAS. The results obtained in this study confirmed differences among studied samples, it is possible to state that different origins and positions of solid environmental samples in the environment reflect in different chemical properties of their matrix. The different properties of the sample matrix result in different mobilities of risk elements in the these kinds of samples. The fractionation analysis with single-step extractions is the method most suitable for easy checking of environmental pollution and for evaluation of risk elements cycle in the environment.

Keywords: Environmental pollution, Mobility of elements, Fractionation analysis, Single-step extractions, Gravitation dust sediment, Soil

INTRODUCTION

The solid environmental samples are not only solid components of the environment but also unacceptable solid products of industrial activities, which may be potential sources of the environmental pollution [1, 2]. In regard to the different origins and positions of the solid environmental samples in the environment it is possible to assume their different properties and behaviour in the environment. The mineralogical composition and content of organic and inorganic matter are very significant properties of these solid environmental samples, whose matrix consists of inorganic and organic materials. The behaviour (bioavailability, mobility, toxicity and distribution) of

risk elements in the solid environmental samples depends not only on their total content, but also on their chemical forms or type of the binding in samples, upon which phases the elements occur in the samples, and which chemical and physical processes these phases are subjected to [3]. The sediments and soils are very often monitored components of the environment because accumulation of risk elements in them causes a potential risk to human health due to transfer of these elements in aquatic media, their uptake by plants and subsequent input to food web [1]. The contamination of soils by risk elements is therefore a momentous environmental problem and has significant connection with human health. Elements from anthropogenic sources tend to be more mobile in soils than those from natural (pedogenic or lithogenic) sources [4].

The solid industrial wastes (ash, fly ash, emissions of solid pollutants) very often contain variable content of toxic or potential toxic elements (risk elements), which may be dangerous for the environment. The emitted solid pollutants become part of the atmospheric dust particles. The atmospheric dustiness from the ecological standpoint can be divided into two groups: the gravitation dust sediment and the fly ash. The gravitation dust sediment is created by particles with high sedimentation ability [5]. The diameter of these particles is more than 10 μm . From an aerodynamic point of view, pollutants which are carried by gravitation dust sediment may cause near-source pollution resulting from dust particles which are easily deposited. Soils are the frequent place of settling of dust particles and these risk element forms, which are released by leaching procedures from dust particles under given soil-ecological conditions, may come into the soil solution [6].

The fractionation analysis is a method of analytical chemistry, which enables classification (i.e. isolation and quantification) of different element forms according to their various physical or chemical properties [7]. The isolation of element forms by extraction is based on the different solubilities of these forms in individual-defined extraction reagents, which may be applied in the single-step or several sequential ordering steps [8, 9, 10, 11, and 12]. The sequential extraction procedures are based on extraction of the sample by the extraction reagents with increasing strength, which cause the release of element from sorption sites with decreasing reactivity [13]. The main disadvantages of sequential extraction procedures are that they are time- (several days) and material-consuming although the long operating time is not a significant disadvantage from point of view of obtained results reliability.

Single-step extractions by various extraction reagents (no-buffered solutions of inorganic salts, organic complexing reagents or other organic reagents, inorganic acid) were originally proposed for fractionation of trace elements forms which may be mobile in the soil under defined soil-ecological conditions [9]. Single-step extractions are less time-consuming than sequential extractions [8]. Solutions of inorganic salts as CaCl_2 , NaNO_3 or NH_4NO_3 are very often used for separation of the “mobile” or “actual bioavailable” fraction of elements, which may be movable in soils or available for plants during of the normal (without growing season or combined utilization of agrochemicals and agrotechnics, respectively) soil conditions, respectively [9]. The 1-h extraction by the complexing agent, ethylenediaminetetraacetic acid (EDTA), and the 16-h extraction by the acid reagent, acetic acid (CH_3COOH), enable separation of the “mobilizable” or “bioavailable” fraction of elements, which consists of the “mobile” fraction and “potential mobile” or “potential bioavailable” fraction, respectively [14]. The “potential mobile/potential bioavailable” fraction may be movable and bioavailable under soil conditions changed by application of agrochemicals and agricultural techniques. EDTA isolates the fraction of the element forms predominantly associated with organic matter but also forms adsorbed on or occluded in the oxides or hydroxides of Fe and, partially, it can dissolve carbonates on defined experimental conditions as well. CH_3COOH releases most of the fraction of elements associated with calcium carbonate, kaolinite, potassium-feldspar, and ferrihydride. From the point of view of the mobilization process of elements, the EDTA releases fraction mobilised by the complexation processes and CH_3COOH fraction remobilised by an acidification process [3]. The HNO_3 have the strongest extraction effect and enables isolation of the “maximal potential mobilizable” fraction or so-called “environmental risk” fraction of elements. This

fraction consists of the “mobilizable” and “potential mobilizable” fraction and includes all releasable element forms. The “potential mobilizable” fraction represents risk forms of elements, which may be movable under extreme changes of soil conditions caused by unpredictable external actions (e.g. ecological damage or natural disaster).). With respect to the fact that gravitation dust sediment becomes part of a soil system, it is possible to use these extraction procedures for evaluation of risk elements entrance from atmospheric dusts to soils as well [15].

The aims of this study were: (1) to assess and compare some significant chemical properties of gravitation dust sediment and soils (2) to optimise the single-step extraction for the gravitation dust samples (3) to compare extraction effect of EDTA and CH₃COOH, and (4) to evaluate risk elements (Cu, Pb, Ni, Zn) input from gravitation dust sediments to soils and their output from soils to biological web. Two samples of gravitation dust sediments and four samples of agricultural soils collected near a metallurgical industrial complex were used for this study.

MATERIALS AND METHOD

Site description, soils and gravitation dust sediments sampling and pre-treatment

Košice residential city agglomeration in East Slovakia is the most important producer of solid waste materials with variable content of elements, which may be dangerous for the environment, is the big metallurgical plant U.S. Steel. In the past, this agglomeration was known for mining and treatment of magnesite. On the basis of results of soil science research the soils in this locality belong to the soils with high or very high soil potential for transforming of inorganic pollutants and moderate risk of heavy metal contamination of a plant production.

In the present study, two areas were chosen for sample collection. In the first sampling area (Fig. 1a), located near U.S. Steel, four agricultural soil samples (P1–P4) and the gravitation dust sediment sample (GDS2), which represents pollutants of metallurgical industry, were collected. The second area for sampling of the gravitation dust sediment, which represents an urban dust (GDS1), is near the Košice city centre (Fig. 1b).

The samples of the gravitation dust sediment (GDS2) have been collected in 12 glass-settling vessels replete by 250 cm³ distilled water for a period of 3 months (summer time 2002). This sample is part of sampling plan for monitoring of risk element input to soils near the big metallurgical plant. The sample of gravitation dust sediment (GDS1) was sampled for a period of 3 years (summer 2000–summer 2002) because this sample was used for extensive analytical study, which includes optimization of various extraction procedures and determination of chemical properties of matrix, too. After expiration of exposition time, the content of vessels was quantitatively flushed by distilled water into the Pt-vessels and then evaporated to dryness on the water bath. After drying the sample was homogenised by crushing and grinding in the agate mill.

The surface soil samples from horizon A (depth = 0–20 cm) were collected from four agricultural sites and subsequently taken into the clean polyethylene bags. About 5.0 kg of the representative soil sample was collected from each sampling area (approximately 10,000 m²). The soils were air-dried, crushed and sieved to 0.2 mm prior to analysis.

Characterization of matrix

The mineralogical composition of the gravitation dust sediment (GDS1) and soils samples were determined by X-ray diffraction (XRD) analysis. Full automatic diffractometer URD-6 (Rich Seifert-FPM, Germany, radiation—CoK α /Ni filter, voltage—40 kV, current intensity—35 mA, step 0.05 2 θ)

was used for the XRD analysis of the sample. The internal standard (cca 5 weight % chemically pure ZnO—zincite) was added to the sample for purpose of quantification of the amorphous material. The content of total organic (TOC) and total inorganic carbon (TIC) was determined by TOC and TIC analysis (equipment: Analytic Jena-multi N/C 3100, with thermocatalytic oxidation and MC- NDIR detection for TOC analysis). The total matrix elements content by WD XRF analysis (equipment: Spectroscan MAKC-GV, Russia) was determined. The samples were dried at 105°C and milled on the size fraction under 63 µm prior to analysis. The standards GBW07309, GBW07310, GBW07311, and GBW07312 were used for calibration.



Fig. 1 Sampling area for soils samples and the gravitation dust sediment sample (GDS2) representing metallurgical pollutants (a), location of sampling areas for the gravitation dust sediment sample GDS2 and sample GDS1 representing urban dust (b) (www.mapy.sk)

Single-step extractions

The single-step extractions were realized as follows: the “mobile/bioavailable” element fraction (water-soluble and ion-exchangeable forms) was isolated by a 1-h extraction with 1 mol dm⁻³ NH₄NO₃ (no-buffered), the “mobilizable” fraction of elements (“mobile” and “potential mobile/potential bioavailable” forms i.e. organic or carbonates bonding forms) was isolated by a 1-hour extraction with 0.05 mol dm⁻³ EDTA (pH = 7, treated with NH₄OH) and by a 16-h extraction with 0.43 mol dm⁻³ CH₃COOH, and for isolation of the “maximal potential mobilizable” fraction (“mobilizable” forms and “potential mobilizable” i.e. bound to the Fe/Mn oxides or sulphides), a 6-h extraction with 2 mol dm⁻³ HNO₃ was used. The ratio value of the soil sample mass to the volume of the extraction reagent (g/cm³) 5/50 or 5/200 for CH₃COOH was constant for all experiments. The (mass/volume) ratio value (g/cm³) for the dust sediment sample was modified on the basis of optimisation experiments realised with the GDS1 sample. The single-step extraction procedures were carried out by mechanical shaking (equipment: laboratory shaker MRC TS—400 D, M.R.C. LTD., Israel) of the suspension of the sample and the extraction reagent in 100 cm³ polyethylene extraction vessel at laboratory temperature. After finishing the extractions, the suspensions were passed through the filter paper with narrow pores (blue ribbon) and paper diameter = 18.5 cm. Obtained extracts were analysed by the FAAS method (equipment: PERKIN ELMER 3030, equipped with a deuterium lamp for background correction, Germany). The FAAS method was used also on the determination of the total content of chosen risk elements (Cu, Ni, Pb, Zn) after pressure digestion of samples by acid mixture HNO₃: HCl (3:1). All vessels were cleaned by washing in 4 mol dm⁻³ HNO₃, thereafter by an extraction reagent, and rinsing with distilled water [16].

RESULTS AND DISCUSSION

On the basis of the XRD analysis results of the soils samples (Table 1) and the gravitation dust sediment sample (Table 1) it is possible to state, that the matrix of both types of samples is very different. The matrix of the gravitation dust sediment sample (GDS1) is predominantly created by amorphous material (approx. 80%). From mineral forms was in the sample detected: quartz, aluminosilicates (chlorite, muscovite), Fe oxides (magnetite, hematite), and carbonates (calcite, dolomite). Obtained minerals are typical for solid pollutants in the agglomeration with metallurgical industrial activities and previous mining and treatment of the magnesite. The content of an amorphous material in the matrix of soil samples is considerably lower and vary between 2–22% approximately. The mineralogical composition of soil matrix is moderately dependent on the sampling area and predominant minerals are quartz and aluminosilicates. The total percentage contents of matrix elements, inorganic (TIC) and organic carbon (TOC) in the studied samples are listed in the Table 2.

Table 1: Percentage content of minerals and amorphous material in studied samples

Results of the mineralogical composition study of both types of the solid environmental samples confirmed differences between both samples resulting from their different origins and positions in the environment.

Higher content of amorphous material in the gravitation dust sediment sample relates to the higher content of TOC. The content of TIC in the dust sample is lesser than the content of TOC, but compared with soil samples is considerably higher. Higher contents of Ca and Mg and TIC in the dust sample GDS1 than in the soil samples correspond to the minerals calcite and dolomite obtained in this sample. Higher content of Fe, together with the high content of amorphous material, suggest the presence of amorphous Fe-bond forms. Contrariwise, higher contents of Si and Al in the soil samples show evidence of dominant presence of quartz and aluminosilicates in the soils.

The total content of chosen elements in the soil samples, decreases in order: Zn, Ni, Pb, Cu, in the gravitation dust sample GDS1 decreases in the order: Cu, Zn, Pb, Ni; in the GDS2, in the order: Zn, Pb, Cu; and (Table 3). The total content of risk elements in the gravitation dust sediment samples is much higher than in the soil samples but the amount of the dust in the environment is much lower than soils. The total content of risk elements in the dust sample GDS2, which represents metallurgical pollutants, is lower than in the sample GDS1.

Phase	Sample					Content/weight %
	Soil P1	Soil P2	Soil P3	Soil P4	Dust GDS 1	
Amorphous	1.40±10.80	16.80±5.40	21.90±4.80	7.50±7.50	78.17±2.10	
Quartz	45.05±2.58	46.94±2.73	48.56±2.76	44.60±2.79	7.51±0.51	
Magnetite	-	-	-	-	3.47±0.60	
Hematite	-	-	-	-	3.41±0.60	
Chlorite IIb-2	12.70±8.10	3.88±1.38	4.96±1.68	10.20±4.80	2.94±1.32	
Muscovite_2M1	23.30±3.60	13.62±2.88	9.91±1.53	23.10±3.30	2.61±1.02	
Calcite	-	-	-	-	1.21±0.48	
Dolomite	-	-	-	-	0.68±0.26	
Orthoclase	5.74±2.07	3.69±1.56	4.69±1.56	4.70±1.14	-	
Plagioclase albite	11.84±1.65	15.06±2.55	9.97±1.59	9.95±1.59	-	

Table 2 Total content (weight %) of matrix elements, TIC, and TOC in studied samples

Sample	Element										
	Si	Al	Fe	K	Mg	Ca	Ti	P	S	TIC	TOC
Soil P1	31.54	7.77	2.71	2.57	0.51	0.55	0.51	0.079	0.050	0.32	1.29
Soil P2	33.39	6.24	2.99	2.08	0.60	0.46	0.51	0.088	0.052	0.31	1.35
Soil P3	34.13	6.09	2.86	1.85	0.51	0.61	0.52	0.078	0.058	0.23	1.98
Soil P4	32.61	7.58	3.48	2.42	0.61	0.45	0.49	0.059	0.026	0.19	0.97
Dust GDS1	7.12	2.25	7.69	0.46	1.56	1.63	0.20	0.103	1.077	5.81	34.93

Table 3: Total risk element content (mg kg⁻¹) in the dust sample

Sample	Element			
	Zn	Ni	Pb	Cu
Soil P1	85.45	28.25	15.27	12.59
Soil P2	84.20	29.84	13.97	13.17
Soil P3	84.30	32.57	19.99	9.19
Soil P4	90.10	29.63	19.86	13.28
Dust GDS1	2498.90	84.90	278.20	6908.30
Dust GDS2	792.05	10.00	224.00	89.00

The second phase of this study was focused on the risk element mobility in the both studied environmental samples or the risk element mobility from dust to the soils and from soils to the plants, respectively. The extraction ratio (g/cm³) for single-step extractions of the gravitation dust sediment (GDS1) was modified on the basis of optimisation experiments results. The influence of extraction ratios 5/50 (validated for soil extractions), 0.5/25, 0.5/50, 0.5/75, 0.5/100 (proposed for gravitation dust extractions) on the recoveries of chosen risk elements in the extracts of EDTA and HNO₃ were investigated. The extraction ratios for the dust extraction were proposed so that the collected dust samples and proportion between total quantity of gravitation dust sediment and soil in the environment corresponded. Two parallel extractions and ten repeated measurements of elements content in the extracts for each extraction ratio and extraction reagent were realised.

The obtained recoveries of element together with the three time standard deviation values (± 3 s) are given in Fig. 2a for EDTA extraction and in Fig. 2b for HNO₃ extraction. The highest recoveries and the lowest ± 3 s ranges were mostly determined at the extraction ratio 0.5/75 (g/cm³). This ratio conformed to the amount of collected dust samples as well as to the dust/soil ratio in the environment.

The third part of this study was focused on the comparison of the extraction effect of EDTA and CH₃COOH on the recoveries of selected elements in the extracts of soil samples and the gravitation dust sample (GDS1). The results of these experiments are listed in the diagrammatic registrations in Fig. 3.

The low extraction effect of the CH₃COOH on the soil samples is connected with absence of carbonates in these samples. The content of chosen elements was detected in EDTA-extract of both types of the studied samples. The extraction effect of the CH₃COOH on the gravitation dust sediment sample was slightly greater than the effect of EDTA and therefore it is possible to state that the risk elements in this dust sample will predominantly associate with carbonates, although their content is lower than the content of organic matter, amorphous phase and Fe.

The fourth part of this study was focused on the determination of the element forms distribution in the studied samples. The percentage recoveries of elements in extracts of 1 mol dm⁻³ NH₄NO₃ represented their percentage content in the “mobile/available” fraction. For the isolation of “mobilizable” fraction of elements, the extraction with 0.05 mol dm⁻³ EDTA was used. The difference between EDTA and

NH_4NO_3 extractable content corresponds to the element content in the “potential mobile/available” fraction or element forms associated with organic matter, respectively. The recoveries of elements in 2 mol dm^{-3} HNO_3 extracts represented their content in all releasable forms. The difference between HNO_3 and EDTA extractable content corresponds to the element content in the “potential mobilizable” fraction or element forms associated with Fe/Mn oxides and sulphides, respectively. The difference between total element content in the samples and their content in HNO_3 extractable forms represented the percentage element content in the “residual” fraction. The residual fraction includes element forms associated with quartz and aluminosilicates and these forms are not movable in the environment.

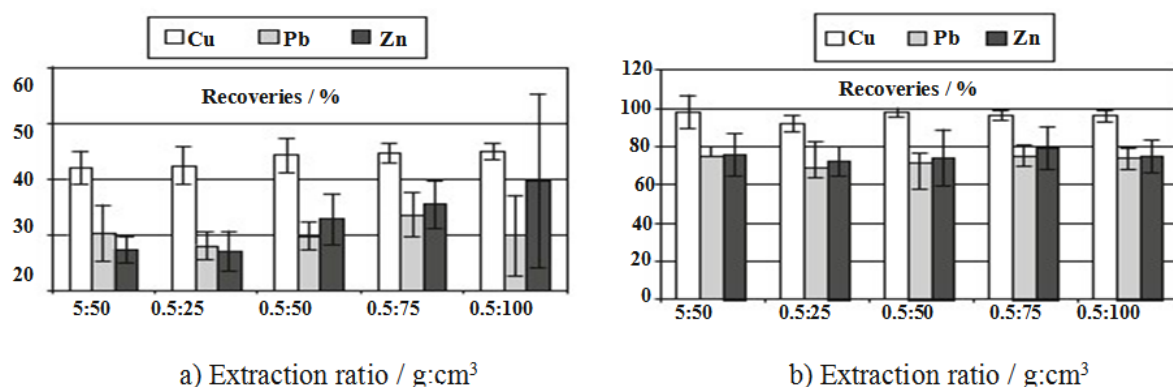


Fig. 2 Influence of extraction ratio values on the percentage recoveries of Cu, Pb, and Zn in: a) EDTA extracts b) HNO_3 extracts

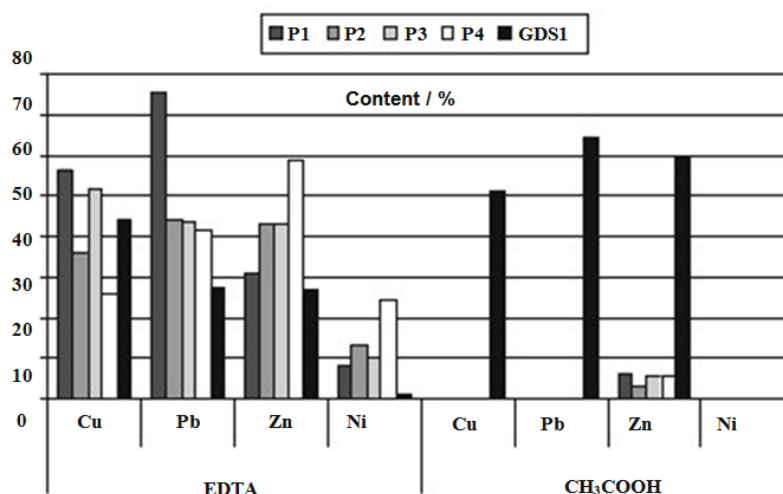


Fig. 3 Percentage content of elements in the EDTA and CH_3COOH extractable fractions

The results of the fractionation analysis confirmed different mobility or bioavailability of the chosen risk elements in both types of the solid environmental samples as well as dependence of element mobility on the sampling area. The percentage contents of Cu, Pb and Zn in EDTA “mobilizable” fraction in the soils vary between 26–57% for Cu, 42–75% for Pb and 31–59% for Zn. These element contents may be transported by the soil solution to the food web under exchanged soil conditions

caused by application of agrochemicals and agricultural technics. The content of these elements in the “potential mobilizable” fraction in the soils vary between 18–36% for Cu, 23–52% for Pb and 6–50% for Zn.

The mobility of elements from dusts to soils depends on the sampling area or on the source of the dust particles. Gravitation dust sediments in studied areas may be an important source of Cu, Pb, and Zn input to the soils under soil conditions changed by agricultural activities (fertilization and tillage). The “mobile/bioavailable” Cu and Zn fraction was detected only in the dust sample GDS1 and Cu fraction in the sample GDS2.

Nickel is the element with the lowest mobility in the dusts and soils. The percentage content of Ni in the residual fraction (non-releasable fraction), in the range from 70% to 90% for soil samples and from 95% to 100% for dust samples, was obtained. On the basis of these results, we can deduce that gravitation dust sediment is not source of Ni input to the soils.

CONCLUSION

On the basis of the results obtained by this study, we can state that:

- The chemical composition of soils and dusts matrix is different and this fact results in different mobilities of elements in these environmental samples. These differences result from different origins and positions of both samples in the environment.
- The total risk element content in dust sediments from industrial areas is much higher than in soils but the amount of dusts in the environment is lower than amount of soils.
- The single-step extractions, in contrast of sequential extraction procedures, have other advantages: shorter operating time and smaller amount of needed chemicals and equipments.
- The extraction ratio of single-step extractions of gravitation dust sediments needs to be optimised prior to their application.
- The optimal extraction ratio for the fractionation extractions of gravitation dust sediment samples is 0.5 g of the sample and 75 cm³ of the extraction reagents.
- For environmental samples with low content of carbonates, it is preferable, for isolation of the mobilizable content of risk elements, to use a 1-h extraction with EDTA than a 16-h extraction with CH₃COOH.
- The fractionation analysis realised by single-step extractions is a very suitable method for evaluation of risk element mobility from gravitation dust sediments to soils and from soils to biological chain, under soil-ecological conditions defined by the used extraction reagent. With this method, it is also possible to identify other sources of risk element input into soils (various types of solid wastes of industrial activities).

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