

Effect of Microelement Loads on the Element Fractions of Soil and Plant Uptake

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Introduction

Our environment, and especially soil, is more and more exposed to contamination by heavy metals and other potentially toxic elements. This concerns mostly the urban environment, having sources of heavy metal contamination, as: settlements (heating, coal/oil combustion, sewage sludge, garbage), industry, traffic, agriculture (fertilizers, chemicals etc.), wastewater (KÁDÁR, 1993; MÉSZÁROS et al., 1993; IZSÁKI & DEBRECZENI, 1987; TAMÁS & FILEP, 1995). High trace element concentrations may harmfully affect soil microorganisms, the quality of yield and groundwater (CSATHÓ, 1994).

Most metals present in soils are fixed. Only traces of them can be found in the soil solution, but this fraction is determinative from the ecological point of view. This fraction affects plant uptake and hence the contamination of the complete food chain and the groundwater. The ratio of mobile, mobilizable and pseudo total fraction depends on soil properties and also on the physico-chemical characteristics of the given element (KÁDÁR, 2005; NÉMETH & KÁDÁR, 2005; SOMOGYI et al., 2004).

Soil extraction with unbuffered salt solutions is an effective method for determining the mobile element fraction (JÁSZBERÉNYI et al., 1994). Many studies have proven that there is a strong correlation between the element fraction extracted with unbuffered salt solution and the plant element content (KERESZTÉNY & MÜLLERNÉ, 1977). The determination objectivity of plant available fractions, however, depends on the given plant, the examined element, the soil, and the extraction method itself. An extraction method may be reliable in the case of a given soil but may give misleading results on another one. That is why no totally trustful method exists. Accordingly, the application of these methods without any preliminary experiment must be avoided. However, plant experiments for calibrating plant available fractions on different soils are still partly missing (FILEP & KÁDÁR, 2003).

An extensive experiment was set up for investigating heavy metal uptake by plants in Hungary, in which the fate of the contaminants was followed along the complex soil-plant-animal-human food chain (KÁDÁR, 1995).

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The report by NÉMETH et al. (1998) is also involved with the fate of heavy metal contaminations, based on data from the Hungarian Soil Information and Monitoring System (TIM). Sorting ammonium-acetate + EDTA soluble (LE) (LAKANEN & ERVIÖ, 1971) element concentrations by soil properties the following relationships were found:

1. There was a 2–3-fold increase in the LE-soluble Cd content in the ploughed layer with the drop of pH and growth of CaCO_3 , organic matter.
2. Concentrations of Cu, Zn and Pb changed differently as a function of soil properties. As the organic matter content grew, all of them showed higher concentrations. Soils with more clay contained more Cu and Pb. But only Cu showed a relationship with pH.

In these surveys the mobile element fractions (unbuffered salt soluble) were not examined. This fraction was represented by the ammonium-acetate + EDTA soluble concentration (KÁDÁR et al., 2000b).

In the present study (as suggested by KÁDÁR (2005)), the mobile fraction is represented by the 1 M NH_4NO_3 extracted element concentration. Mobilizable elements are extracted by ammonium-acetate + EDTA and the pseudo total fraction is characterized by cc. HNO_3 + cc. H_2O_2 digestion. Answers to the following questions are sought for: How does the mobile fraction change after heavy metal loads? What kind of relationships can be found among the different element fractions? How does the plant–soil transfer coefficient change according to the element fraction taken into account?

Materials and Methods

A long-term field microelement experiment was set up in the spring of 1991 on a calcareous chernozem soil at the Experimental Station of the Research Institute for Soil Science and Agricultural Chemistry of the Hungarian Academy of Sciences in Nagyhorcsók. The ploughed layer of the growing site contained around 5% CaCO_3 and 3% humus and was very well supplied with Ca and Mn, satisfactorily supplied with Mg and Cu, moderately well with N and K and poorly with P and Zn. The soil texture is loam with 40% clay + silt fraction. Half of its clay minerals are illite, one third is chlorite and partly smectite. The pH (KCl) of the ploughed layer was 7.3. The groundwater depth was 15 m and the water balance was negative, tending to drought (KÁDÁR, 2000).

Salts of the 13 microelements tested were applied at four levels, once in spring 1991, prior to maize sowing. The $13 \times 4 = 54$ treatments were set up in two replications giving a total of 104 plots (21 m² each) in a split-plot design.

The loads were 0, 90, 270 and 810 kg/ha for each element in the form of AlCl_3 , NaAsO_2 , BaCl_2 , CdSO_4 , K_2CrO_4 , CuSO_4 , HgCl_2 , $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}$, NiSO_4 , $\text{Pb}(\text{NO}_3)_2$, Na_2SeO_3 , SrSO_4 and ZnSO_4 . The extreme high doses are used for modelling the soil contamination levels.

In addition to the micronutrients N-P-K fertilizers were given yearly in the dose of 100–100–100 kg/ha N, P_2O_5 and K_2O .

Representative plant samples were made from at least 20 plants or plant parts, or by serials using 8–8 running meter above-ground plant material, collected from the net plot area. The determination of plant element concentrations was carried out with the ICP-AES method after cc. $\text{HNO}_3 + \text{H}_2\text{O}_2$ digestion.

20–20 soil core samples of each plot were united to get a representative sample. From the samples 1 M NH_4NO_3 extractable element concentrations were examined. The latter method according to the DIN standard (1995) is as follows: 20 g air-dried soil is shaken for 2 hours with 50 cm³ 1 M NH_4NO_3 solution. Then the suspension stands for 15 minutes, after which the supernatant is filtrated through a 0.45 µm filter. The first 5 cm³ of the filtrate must be tip off. Finally, the extract is acidified with one drop of cc. HNO_3 . The determination of element concentrations was conducted with the ICP-AES method. The pseudo total (cc. $\text{HNO}_3 + \text{H}_2\text{O}_2$ digestion) and the 0.5 M ammonium-acetate + 0.02 M EDTA extractable (LAKANEN & ERVIÖ, 1971) element fractions were measured previously (KÁDÁR et al., 2000a,b, 2001).

Results and Discussion

In the investigated soil the concentrations of Pb and Al with 1 M NH_4NO_3 extract did not reach the detection limit on any treatment level. Pb is strongly bound in most soils (ELLIOTT et al., 1986) and Al constitutes stable complexes with soil organic matter (FILEP, 1988). The mobile fraction of other elements increased as a function of treatments. In the case of As this increase was not significant. As compared to the control treatment, there was a 370-, 167-, 95- and 92-fold increase in the mobile fraction of Cr, Se, Hg and Cd, respectively. The mobile fraction of Ba, Cu and Sr showed the lowest increment (Table 1). According to PUEYO et al. (2004) in the case of Cu the extraction effectiveness with NH_4NO_3 is enhanced due to the formation of strong amino complexes.

Element concentrations of the three fractions differ considerably (Table 1) and the sequence among them is as follows: NH_4NO_3 extractable < LE < pseudo total. This is in accordance with the results of previous experiments (MOLNÁROS & GRÁCZOL, 2000a,b). This is a consequence of the different extraction methods and the fact that they characterize different element fractions.

The unbuffered NH_4NO_3 solution mainly measures the elements from the soil solution, while the buffered (pH = 4.6) ammonium-acetate + 0.02 M EDTA solution extracts a significant amount of exchangeable cations. The LE-solution can solve precipitated or other insoluble metal-carbonates due to its low pH. The pseudo total fraction involves cations bounded to the organic matter and the above-mentioned fractions. This concludes that the NH_4NO_3 solution extracts a significantly smaller amount of cations than the buffered LE-solution. For example, as mentioned above, in the case of Al the mobile fraction was not measurable, while the mobilizable fraction reached 60–90 mg/kg in the treatments. The same is true for Pb and partly for Ni and Zn. In general there is a one order of magnitude difference between the NH_4NO_3 soluble and LE-soluble element concentrations.

Table 1
Effects of treatments on the element composition of the ploughed layer, mg/kg
(Calcareous chernozem soil, Nagyhorcsök)

Element	Loads in spring 1991, kg/ha				LSD _{5%}	Average
	0	90	270	810		
<i>Mobile fraction (NH₄NO₃ extraction), mg/kg</i>						
Zn	—	—	0.1	0.4	0.3	0.1
Ni	—	0.1	0.3	0.7	0.1	0.3
Hg	—	0.2	0.9	4.1	0.7	1.3
Cr	—	0.3	1.0	3.8	3.1	1.3
Cu	0.1	0.3	0.5	1.1	0.2	0.5
As	0.1	0.2	0.8	4.4	6.8	1.4
Cd	0.2	0.8	3.1	13.9	4.3	4.5
Se	—	2.1	6.2	46.6	37.9	13.8
Mo	0.4	7.3	11.2	27.8	4.2	11.7
Sr	5.7	10.8	22.5	55.2	26.3	23.5
Ba	13.8	22.1	34.3	94.5	21.4	41.1
<i>Mobilizable fraction (Lakanen-Erviö extraction), mg/kg*</i>						
Hg	—	4	49	189	13	61
Cr	—	2	6	30	5	10
Mo	1	21	27	104	14	38
Se	—	7	23	123	13	38
Zn	2	13	55	153	18	56
As	—	7	18	66	14	23
Ni	3	15	40	74	20	33
Pb	5	29	56	158	32	62
Cu	7	24	49	110	40	47
Cd	—	30	86	228	40	86
Ba	20	29	41	100	16	47
Sr	31	49	67	146	16	73
Al	67	73	87	90	8	79
<i>Pseudo total fraction (cc. HNO₃+H₂O₂ digestion), mg/kg**</i>						
Mo	—	10	20	114	24	36
Pb	10	49	142	264	33	117
Hg	—	26	67	157	33	66
Se	7	29	81	224	22	88
Cu	17	43	85	230	24	120
Cd	0.3	18	50	162	18	62
Cr	18	48	64	121	32	63
As	7	28	81	210	34	86
Ni	28	63	112	248	18	113
Zn	40	71	118	274	26	127
Sr	60	90	158	352	28	165
Ba	80	111	154	398	54	221
Al	10703	12220	11810	11805	1423	11945

Remarks: * In: KÁDÁR et al. (2000b); **In: KÁDÁR et al. (2001) –: below the detection limit

Table 2
Element uptake (g/ha) of maize at harvest on 25 November, 1991
(Calcareous chernozem, Nagyhorcsök) (KÁDÁR et al., 2000a)

Element	Loads in spring 1991, kg/ha				LSD _{5%}	Average
	0	90	270	810		
<i>In the stalk yield</i>						
Al	1007	1543	1039	878	758	1117
As	—	—	—	—	—	—
Ba	26	36	96	269	75	107
Cd	—	18	56	201	6	68
Cr	—	4	4	3	2	3
Cu	42	52	50	42	17	46
Hg	—	0	3	8	3	3
Mo	—	107	144	289	28	135
Ni	2	2	6	8	5	4
Pb	22	17	22	18	12	20
Se	—	23	40	60	4	31
Sr	43	69	62	103	18	69
Zn	36	155	244	273	103	177
<i>In the stalk + grain yield</i>						
Al	1010	1548	1041	888	740	1122
As	—	—	—	—	—	—
Ba	26	36	98	269	72	107
Cd	—	18	56	201	6	68
Cr	—	4	4	3	2	3
Cu	55	64	62	56	18	59
Hg	—	-	3	8	3	3
Mo	—	156	181	352	30	172
Ni	3	9	14	15	6	10
Pb	22	17	22	18	13	20
Se	—	81	106	153	5	85
Sr	44	71	75	16	22	76
Zn	108	483	543	536	110	418

Remark: –: below the detection limit

The pseudo total fractions exceed the LE-soluble element concentrations notably (Al, Ba, Zn) or in some cases hardly.

Treatment effects can be seen in all element fractions, but the most significant increase in concentrations shows up in case of the pseudo total fraction.

For quantifying the relationships between soil element fractions, a regression analysis was made. The pseudo total and mobilizable fractions were plotted as a function of mobile fraction. The assumed relationship between the fractions was linear. The slope of the linear functions shows the mobility of the given element in soil. The higher the slope value the stronger the fixation. Accordingly, the most mobile elements are Ba, Mo, Sr and Se. The most bound elements are Cu, Ni and

Zn. These are fixed mainly on the soil organic matter. The sequence of fixation strength is: Ni > Zn > Cu > Hg ≈ As > Cr > Cd > Sr ≈ Mo ≈ Ba ≈ Se.

The experiment may show the element amount extractable from soil by the above-ground parts of maize. The extracted amounts can be calculated by multiplying the plant element concentrations with the amount of yield (Table 2). The maximum quantities of elements incorporated in the whole above-ground yield of maize were as follows (in g/ha): Al 1548, Zn 543, Mo 352, Ba 269, Cd 201, Se 153, Sr 116, Cu 64, Pb 22, Ni 15, Hg 8, Cr 4 and As <1. Uptake is negligible compared to the degree of pollution, so bioremediation is probable to take up to 4000 years, for example in the case of extreme rates of Cd pollution (KÁDÁR et al., 2000a).

The uptake and accumulation of metals can also be described with the transfer coefficient that is the quotient of plant element concentration and soil element concentration:

$$\text{Transfer coefficient} = \text{plant concentration (mg/kg)} / \text{soil concentration (mg/kg)}$$

Transfer coefficient values were calculated considering maize grain and stem and each soil element fraction, respectively (Tables 3 and 4). The maize element

Table 3
Transfer coefficients between different soil element fractions and maize grain
(Calcareous chernozem soil, Nagyhörsök)

Element	Loads in spring 1991, kg/ha				LSD _{5%}	Average
	0	90	270	810		
<i>Mobile fraction (NH₄NO₃ extraction), mg/kg</i>						
Zn	—	—	0.08	0.36	0.31	0.11
Mo	—	0.61	0.59	0.49	0.09	0.42
Sr	0.03	0.02	0.05	0.03	0.11	0.03
Ni	0.2	8.8	2.7	1.2	0.8	3.2
Cu	10.9	5.2	3.1	1.7	3.5	5.2
Se	23.1	3.5	2.1	0.5	12.9	7.3
<i>Mobilizable fraction (Lakanen-Erviö extraction), mg/kg</i>						
Ni	—	0.06	0.02	0.01	0.01	0.02
Mo	—	0.25	0.25	0.14	0.24	0.16
Al	0.01	0.01	—	0.01	0.02	0.01
Sr	0.01	—	0.02	0.01	0.04	0.01
Cu	0.25	0.06	0.03	0.02	0.17	0.09
Zn	4.5	1.8	0.92	0.20	2.6	1.9
Se	5.2	1.1	0.57	0.19	1.6	1.8
<i>Pseudo total fraction (cc. HNO₃+H₂O₂ digestion), mg/kg</i>						
Sr	—	—	0.01	—	0.02	—
Ni	—	0.01	0.01	—	0.00	0.01
Mo	—	0.49	0.40	0.12	0.40	0.25
Cu	0.08	0.09	0.09	0.11	0.04	0.09
Zn	0.18	0.37	0.34	0.10	0.20	0.25
Se	0.34	0.25	0.14	0.10	0.16	0.21

Remark: –: below the detection limit

concentrations were published previously by KÁDÁR et al. (2000b). In the case of grain the coefficient can be calculated only for a few elements, as most of the contaminants are below the detection limit in this plant organ. Transfer coefficient values concerning the three soil element fractions differ notably. Values are between 0.02–23.1 for NH_4NO_3 , 0.01–5.2 for LE and 0.01–0.25 for pseudo total ele-

Table 4
Transfer coefficients between different soil element fractions and maize stem
(Calcareous chernozem soil, Nagyhörcsök)

Element	Loads in spring 1991, kg/ha				LSD _{5%}	Average
	0	90	270	810		
<i>Mobile fraction (NH₄NO₃ extraction), mg/kg</i>						
Zn	—	—	436	171	308	151
Cr	—	4.2	4.3	1.7	4.1	2.6
Mo	—	4.7	3.4	3.8	2.2	3.0
Ba	0.36	0.34	0.56	0.54	0.67	0.45
Sr	1.56	1.3	0.65	0.38	0.55	0.96
Cd	20.6	5.7	3.7	3.5	9.5	8.4
Se	23.6	2.5	1.9	0.49	32	7.1
Ni	41.9	3.0	3.8	2.3	19.4	12.5
Cu	64.9	41.5	23.7	10.3	63.5	35.1
<i>Mobilizable fraction (Lakanen-Erviö extraction), mg/kg</i>						
Mo	—	1.8	1.4	1.1	0.96	1.1
Cr	—	0.81	0.62	0.22	0.52	0.41
Ni	0.19	0.01	0.03	0.02	0.03	0.06
Cd	0.2	0.08	0.07	0.11	0.09	0.11
Ba	0.25	0.25	0.47	0.51	0.62	0.37
Sr	0.3	0.28	0.21	0.13	0.31	0.23
Pb	0.82	0.14	0.12	0.05	0.3	0.28
Cu	1.5	0.48	0.23	0.1	1.7	0.6
Al	3.6	4.8	2.6	2.0	1.4	3.3
Zn	3.9	3.6	0.65	0.36	5.7	2.1
Se	5.0	0.77	0.5	0.17	5.5	1.6
<i>Pseudo total fraction (cc. HNO₃+H₂O₂ digestion), mg/kg</i>						
Cr	—	0.02	0.06	0.04	0.05	0.03
Mo	—	3.5	2.3	0.9	1.9	1.7
Al	0.02	0.03	0.02	0.01	0.01	0.02
Ba	0.06	0.06	0.13	0.13	0.15	0.09
Ni	0.11	0.09	0.1	0.09	0.02	0.01
Sr	0.16	0.15	0.08	0.04	0.02	0.11
Zn	0.16	0.3	0.26	0.2	0.11	0.23
Cd	0.16	0.26	0.23	0.29	0.34	0.24
Se	0.29	0.19	0.13	0.09	0.17	0.17
Pb	0.35	0.07	0.03	0.02	0.26	0.12
Cu	0.47	0.25	0.13	0.05	0.18	0.23

Remark: –: below the detection limit

ment fractions. The greatest accumulation was found in relation to the mobile fraction and grain Ni, Cu and Se contents, having coefficient values of 3, 5 and 7, respectively. The coefficients calculated from mobilizable and pseudo total soil fractions were smaller than these: for LE extractions Se and Zn (1.8), for pseudo total fractions Se (0.21), Zn (0.25) and Mo (0.25) accumulated in the highest concentrations in the grain. As the function of soil element concentrations the coefficient decreases. Thus maize accumulates relatively less metal at the highest loads. Mo and Ni uptake cannot be established on the control plots, but they showed extreme accumulation at the smallest load level, that decreased at the highest loads.

Maize stem transfer coefficients can be calculated for more elements than in the case of grain (Table 4). Coefficients for stem may exceed those obtained for grain. Values were between 0.34–436 for NH_4NO_3 extractions, 0.01–5.0 for LE and 0.01–3.5 for pseudo total element fractions. The highest values in all three fractions were found for Se, Ni, Zn, Al, Cd and Mo. Similarly to grain, the accumulation in stem also decreases as a function of loads.

Generally it can be stated that the coefficients for NH_4NO_3 extractions show the greatest standard deviation, thus this quotient indicates the heavy metal and toxic element accumulation in plants with the greatest sensitiveness. It is still a question, however, whether this coefficient overestimates the accumulation or not, and which soil element fraction can be considered appropriately in environmental risk assessment.

Summary

The effect of microelement loads was investigated on the mobile (1 M NH_4NO_3 soluble), mobilizable (NH_4 -acetate + EDTA soluble) and pseudo total (cc. HNO_3 + H_2O_2) element concentrations of a calcareous chernozem soil in a long-term field experiment. Salts of 13 microelements were applied on four levels: 0, 90, 270 and 810 kg/ha in the spring of 1991 prior to sowing maize.

The relations between the mobile, mobilizable and pseudo total fractions of a heavy metal contaminated soil were defined and quantified. According to this, Ba, Mo, Sr and Se have the greatest mobility, while Cu, Ni and Zn showed strong fixation on the examined soil.

The maximum quantities of elements incorporated in the whole above-ground yield of maize were as follows (in g/ha): Al 1548, Zn 543, Mo 352, Ba 269, Cd 201, Se 153, Sr 116, Cu 64, Pb 22, Ni 15, Hg 8, Cr 4 and As <1.

Transfer coefficients calculated as a quotient of different soil element fractions and maize grain and stem element contents were compared. In the case of grain the greatest accumulation was found in relation to the Ni, Cu and Se contents of the mobile fraction. The coefficient values were 3, 5 and 7, respectively. Coefficients for stem may exceed those obtained for grain. The accumulation decreases as a function of loads. The coefficients for NH_4NO_3 extractions show the greatest standard deviation, thus this quotient indicates the heavy metal and toxic element accumulation in plants with the greatest sensitiveness.

Key words: element fractions, NH_4NO_3 extractable elements, transfer coefficient

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