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Geochemical factors controlling the migration and immobilization of  
heavy metals as reflected by the study of soil profiles  
from the Cserhát Mts.

PhD Theses

with 10 figures and 4 tables

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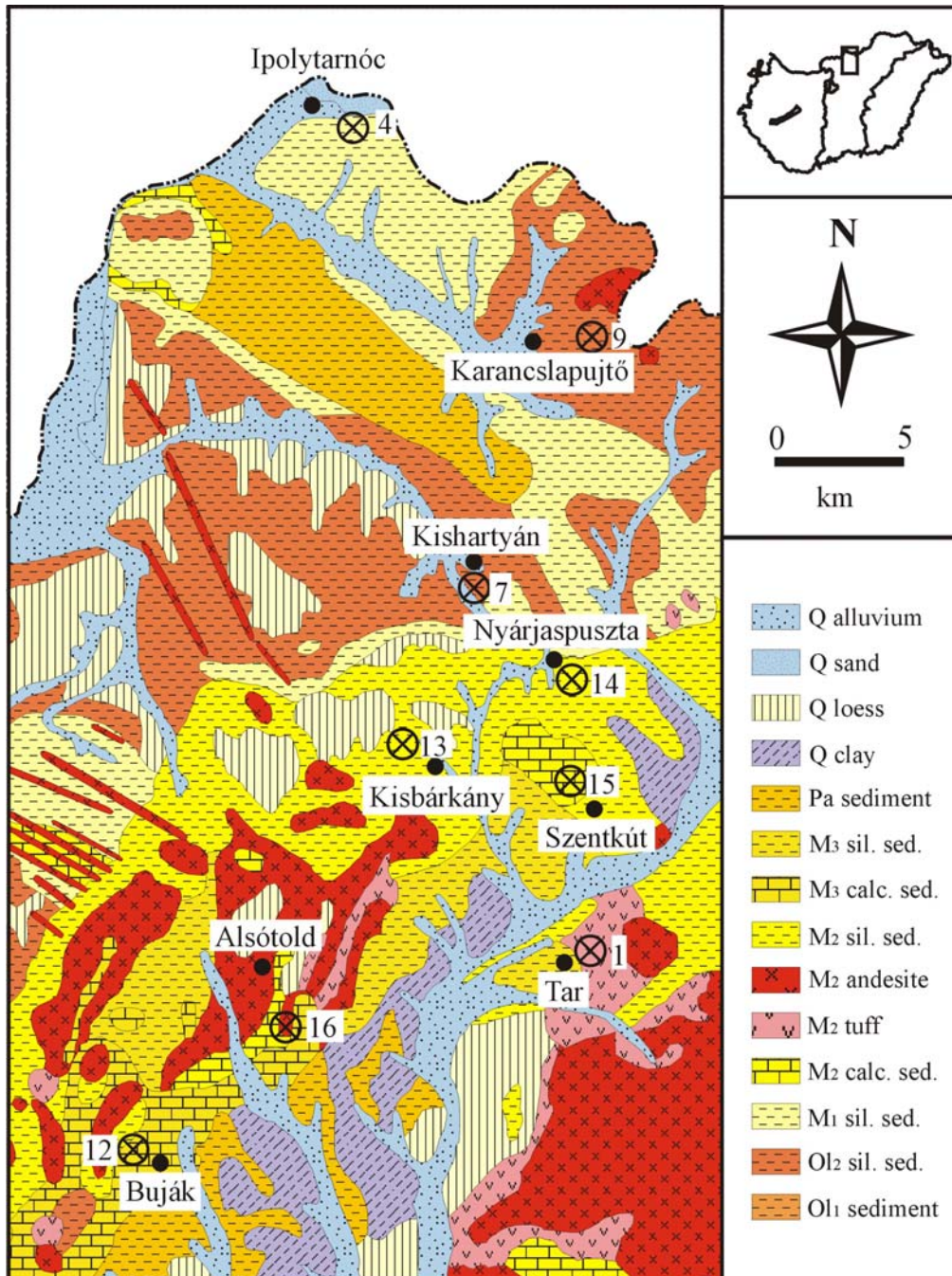
## **Introduction**

The natural balance of the biogeochemical cycles has been considerably changed by the human activity causing enhanced mobilization of chemical elements into the environment as compared to the natural processes. Therefore the essential processes under natural conditions may become harmful. The soil is able to store and accumulate trace metals to some extent due to its adsorption capacity. Significant migration may be started by changes in the conditions controlling the mobility of adsorbed metals. This may result in “explosion of chemical time bombs” (Stigliani, 1991). This process can begin due to both natural and anthropogenic factors, and a long time can pass between the contamination and its effect. The study of factors controlling the migration, adsorption and accumulation of trace elements in soils and sediments is one of the basic tasks of environmental geochemistry.

My purpose was to study the effect of various geochemical factors on the distribution, migration and immobilization of heavy metals in soil profiles characterized by similar pedogenic processes but different soil parent materials. The studied profiles were characterized from environmental geochemical point of view. Their buffering capacities were studied by laboratory experiments using samples chosen according to their characteristics affecting the behavior of heavy metals in soils. The geochemical factors affecting the distribution of heavy metals in soils in the case of concentrations close to or considerably higher than the natural background level were compared, primarily taking into consideration the parent material. Finally, conclusions were drawn on the environmental geochemical conditions of the study area, as well as on the environmental geochemistry of brown forest soils.

To this aim a study area characterized by varied geology and similar soil forming factors within a small distance was needed. Such areas are expected to be covered by similar soil types. My goal was to study samples being free of direct anthropogenic effects, admitting that the atmospheric deposition of heavy metals can not be ignored anywhere. Accordingly, hilly woodland areas in regions characterized by low industrial activity are most convenient to carry out the study. An additional advantage is that these areas are mostly covered by brown forest soil types, which belong to the most widespread soil types of Hungary (Stefanovits, 1971). On this

basis, the eastern Cserhát Mts., the Karancs area and the western slopes of the Mátra Mts. were chosen as the sampling area (Figure 1).



**Figure 1.** Schematic geological map of the study area (after the map of the Hungarian Geological Institute in Láng, 1967) showing the sampling sites

*sil. sed.* =siliciclastic sediments (gravel, sand, clay etc.), *calc. sed.* = calcareous sediments (limestone, calcareous sandstone, marl etc.)

## Methods

Out of the 16 sampled profiles 9 were studied with full particulars (5 brown forest soil with clay illuviation, 1 Ramann brown forest soil, 2 humous sandy soil and 1 stony soil profiles). The soil parent materials represent well the geology of the study area (Table 1). Mineralogical (XRD), major element (XRF) and organic matter (Rock-Eval) analyses, as well as soil mechanical tests and pH measurements were carried out to characterize the profiles.

**Table 1.** *The studied soil types, their parent material, as well as the sampling sites*

No.	Soil type	Parent material	Sampling site
1.	Stony soil	Dacite tuff / loess	Tar
4.	Humous sandy soil	Sandstone	Ipolytarnóc
7.	Humous sandy soil	„Glauconite” sandstone	Kishartyán
9.	Brown forest soil with clay illuviation	Calcareous siltstone	Karancslapujtó
12.	Brown forest soil with clay illuviation	Limestone	Buják
13.	Brown forest soil with clay illuviation	Siltstone	Kisbárkány
14.	Brown earth	Sandstone	Nyárjaspuszta
15.	Brown forest soil with clay illuviation	Calcareous sandstone	Szentkút
16.	Brown forest soil with clay illuviation	Piroxene andesite	Alsótold

The total and the bioavailable contents of chromium, cobalt, nickel, copper, zinc and lead in the studied soil samples were determined. The samples were digested according to the Hungarian standard methods (MSZ 21470-50), the analyses of solutions were performed by AAS. Sequential extraction studies after Li et al. (1995) were used to determine the potential mobility of studied heavy metals in four brown forest soil profiles with clay illuviation developed on different parent materials. The concentrations of chemical elements were established by ICP-AES.

Nickel, copper, zinc and lead adsorption and desorption studies in single element and competitive situations were performed on samples chosen according to their characteristics affecting the behavior of heavy metals in soils. The laboratory experiments were carried out in the 20-2000 mg/l range and in the concentration interval of potentially mobile fractions of

sewage sludge in the case of single element and competitive situations, respectively. After the 48 hours long reaction time the determination of heavy metal contents of the equilibrium solution was performed by AAS. Desorption of adsorbed metals in competitive situation was studied by distilled water, magnesium-chloride and sodium-acetate dissolution. The dissolved metal concentrations in solution were analyzed by ICP-AES. Determination of lead adsorption capacity of individual mineral grains was carried out by TEM-EDS.

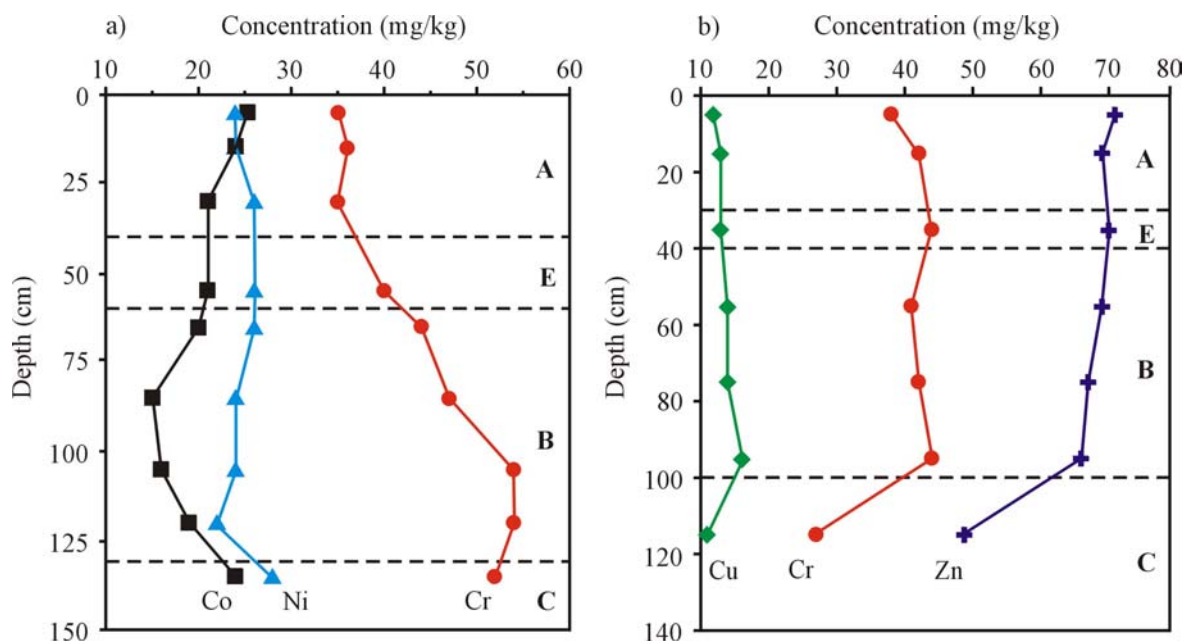
## Results

1. I have found that the concentrations of heavy metals in the studied profiles are at or below the natural geochemical background in Hungary (Table 2).

**Table 2.** Heavy metal concentrations in the studied soil profiles as compared to the values measured at the sites of the Soil Information and Monitoring System (TIM), as well as the background concentrations („A” value) given in the 10/2000 (VI. 2.) governmental decree

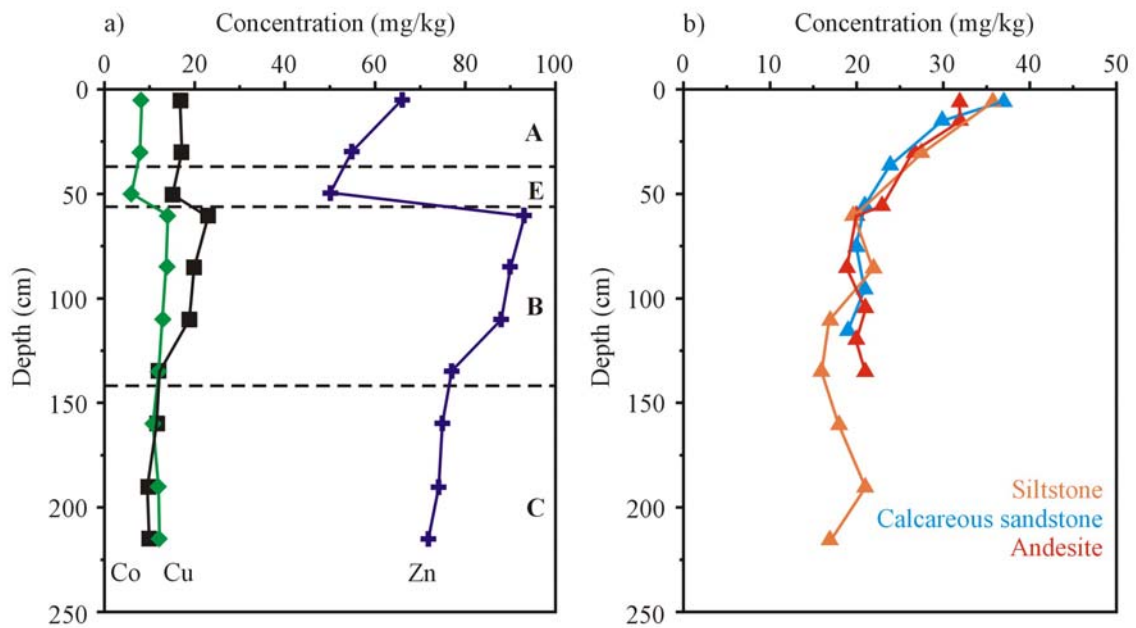
Metals mg/kg	Studied profiles		TIM sites		„A” values
	range	average	range	average	average
Co	0-25	9	3-24	12	15
Cr	9-54	30	4-59	25	30
Cu	2-29	12	3-23	13	30
Ni	9-42	25	4-38	21	25
Pb	3-37	18	2-27	14	25
Zn	28-94	59	14-71	47	100

The brown forest soils with clay illuviation are characterized by the highest heavy metal contents, while the humous sandy soils by the lowest one. I have shown the significant effect of soil parent material on the heavy metal content of soils. The soil developed on andesite contains the highest, the soils formed on sandstone the lowest heavy metal amounts, while there is a significant relative enrichment of heavy metals in soils developed on calcareous bedrock because of the rapid weathering of carbonate minerals (Figure 2).



**Figure 2.** The effect of soil parent material on the distribution of heavy metals in soil profiles  
a) The enrichment of metals in the C horizon of the brown forest soil developed on piroxene andesite was found, which can be also characteristic in some cases (e. g. chromium) in the lower part of B horizon. b) There is a significant relative enrichment of heavy metals in the upper horizons of the brown forest soil developed on calcareous sandstone because of the rapid weathering of carbonate minerals

2. According to my studies the distribution of heavy metals in soils is usually uniform, independently of the soil type. However, lead content is the highest at the surface horizons and it decreases with depth (Figure 3). This phenomenon suggests a relation between the lead and the soil organic matter. Moreover it refers to the concentration of lead from atmospheric deposition, which is also shown by the lack of lead enrichment in the upper horizon of the eroded profile.
3. The amounts of bioavailable metals in the studied profiles decrease as follows:  $Pb > Co > Ni > Cu > Zn > Cr$ . I have found no direct relation between the total and the bioavailable metal contents of soils. The relative availability for cobalt, lead and nickel is always high or medium, for copper and zinc is low, and for chromium is very low, independently of the pedogenic properties. In the ratio of bioavailability the highest proportions of heavy metals are available from the horizons characterized by the highest organic matter content, but the availability does not decrease with orders of magnitude in the deeper horizons (Figure 4a).

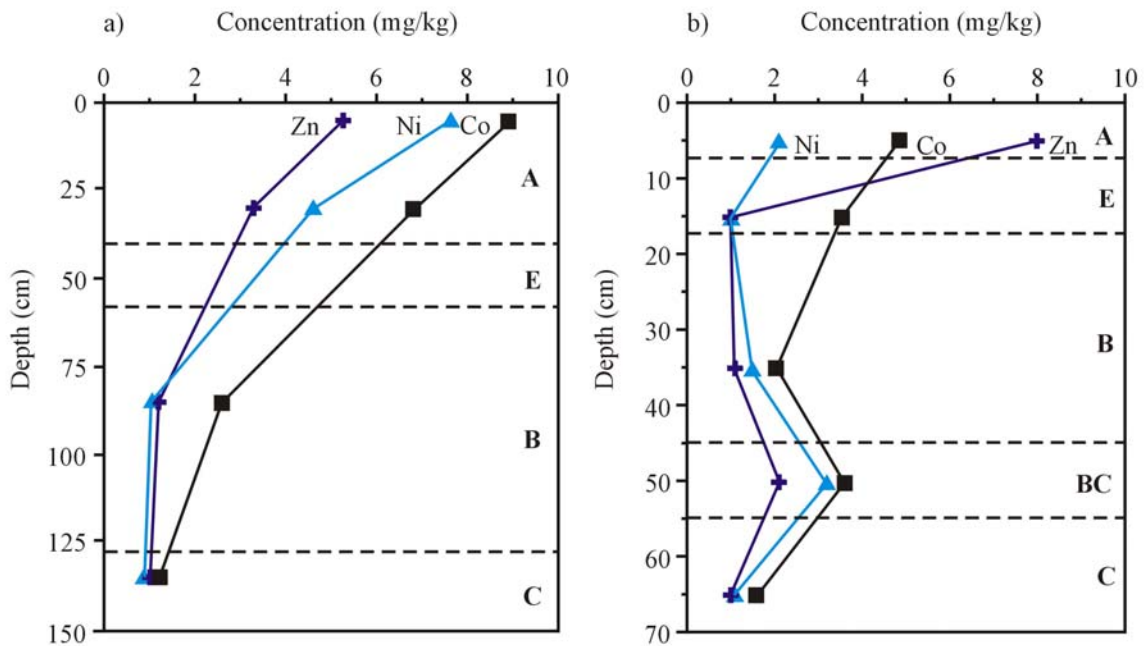


**Figure 3.** The effect of clay illuviation (a), as well as the anthropogenic activity (b) on the distribution of heavy metals in soils

a) Some metals show a relative enrichment in the B horizon of the brown forest soil developed on siltstone as compared to the upper horizons due to the downward leaching. b) The concentration of lead decreases with depth in all of the studied profiles suggesting the presence of an anthropogenic source (atmospheric deposition)

I have shown that the bioavailability of metals is mostly affected by the organic matter content of soil and by the specific controlling ability of plants. The bioavailability of some metals (Co, Ni, Cu, Zn) decreases in horizon B of the soils with clay illuviation due to their enhanced adsorption capacity (Figure 4b). The availability of metals also decreases in the calcareous soil horizons because of the enhanced buffering capacity of these horizons.

4. According to my studies the  $Pb > Co > Ni > Cu > Zn > Cr$  relative mobility sequence was established. 10% and 50-70% of chromium and lead amounts are potentially mobile in the studied profiles, respectively (Figure 5). These results show the higher mobility of lead as compared to the other studies. This may be due to the anthropogenic origin of some part of the lead content. The mobility of certain metals is very similar to each other in the studied soil profiles corresponding to the similar weathering-pedogenic processes in the profiles.

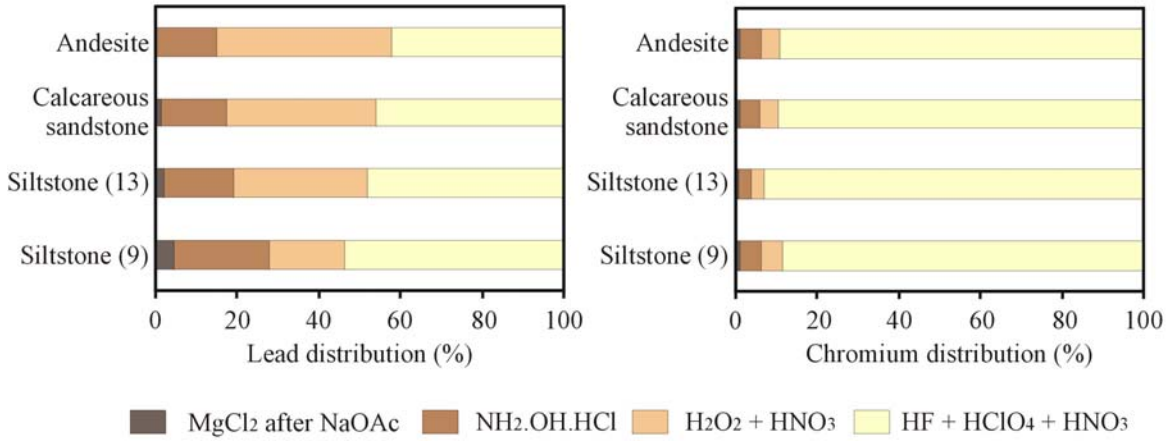


**Figure 4.** Distribution types of bioavailable heavy metal concentrations

The amounts of the bioavailable cobalt, nickel and zinc decrease with depth in the brown forest soil formed on piroxene andesite (a) suggesting their close relation to the organic matter content of the soil. In the other hand, in the brown forest soil developed on siltstone the bioavailable amount of these metals smaller than in the A or BC horizons (b), which is because of the enhanced adsorption capacity of this horizon due to the clay illuviation

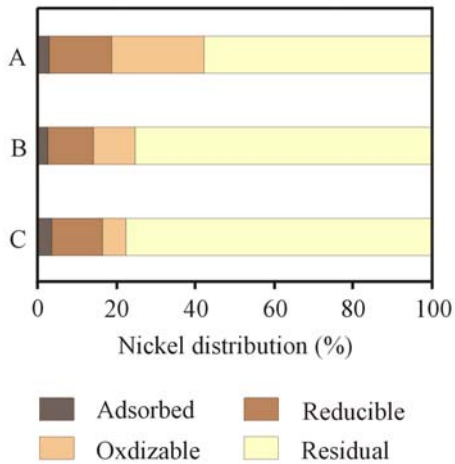
- I have shown that the highest proportion of metals can not be mobilized, or only with much difficulty. This proportion increases with depth, which means that the effect of bedrock on the mobility of heavy metals increases downwards (Figure 6). I have found that among the mobile fractions the fraction bound to organic matter plays the most important role in the case of lead, copper and nickel, while a considerable part of cobalt and zinc, but also of lead and nickel can be bound to Fe-Mn oxides. I have found that the role of the easily mobilizable heavy metal fraction in these soils is ancillary.
- I have established that both the single element and the competitive one can be correctly described by the Langmuir-isotherm, but the fitting fails in the case of those elements (Ni, Zn), where the competition has a strong effect on the adsorption. In the case of single element adsorption the shape of the curves suggests the high affinity of metals to the studied samples

and in some cases the competition and surface precipitation, i.e. it is the combined result of several processes.



**Figure 5.** Distribution of lead and chromium in the different sequential extraction steps in the case of some soil profiles

Among the studied metals the lead is the most, and chromium is the less mobile. The mobility of a given metal in the different brown forest soils is very similar independent of the parent material, showing the similarity of soil forming processes in these profiles

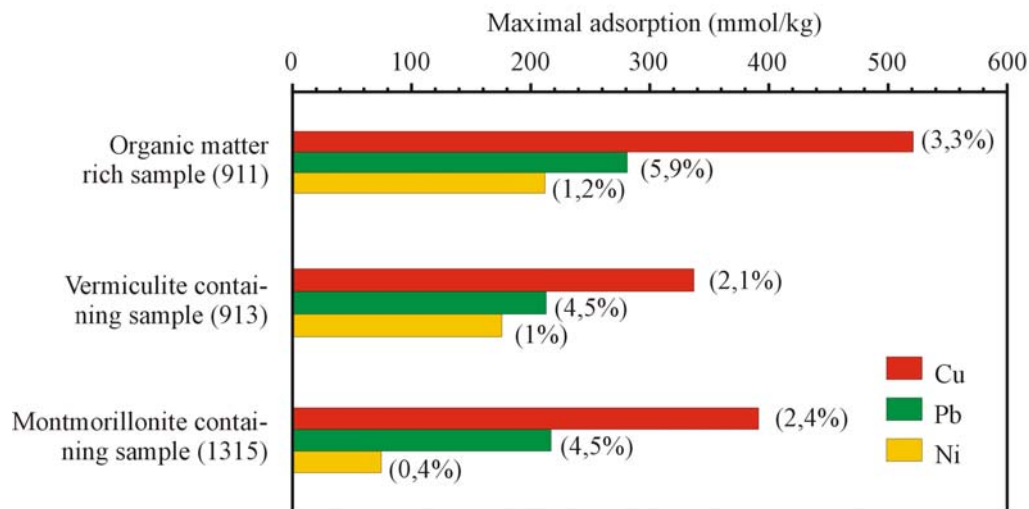


**Figure 6.** Distribution of nickel in the different sequential extraction steps in the case of the different soil horizons

The mobility of metals decreases with depth showing the increasing effect of soil parent material and decreasing effect of soil forming processes

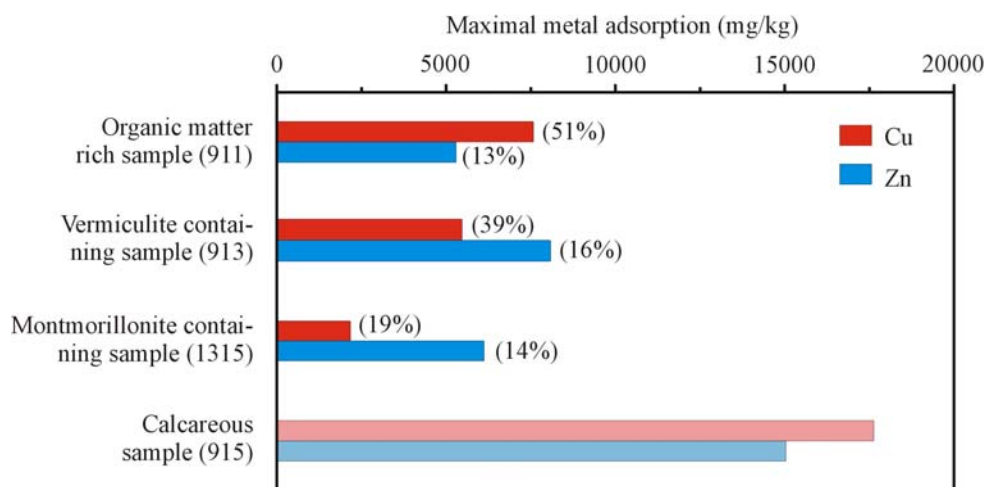
Adsorbed = MgCl<sub>2</sub> and after NaOAc, Reducible = NH<sub>2</sub>.OH.HCl, Oxidizable = H<sub>2</sub>O<sub>2</sub> + HNO<sub>3</sub>, Residual = HF + HClO<sub>4</sub> + HNO<sub>3</sub>

7. According to the laboratory experiments the affinity of the studied metals to soils decreases as follows, independently of soil composition: Cu > Pb > Zn > Ni (Figure 7).



**Figure 7.** Maximal adsorption of copper, lead and nickel on several soil samples

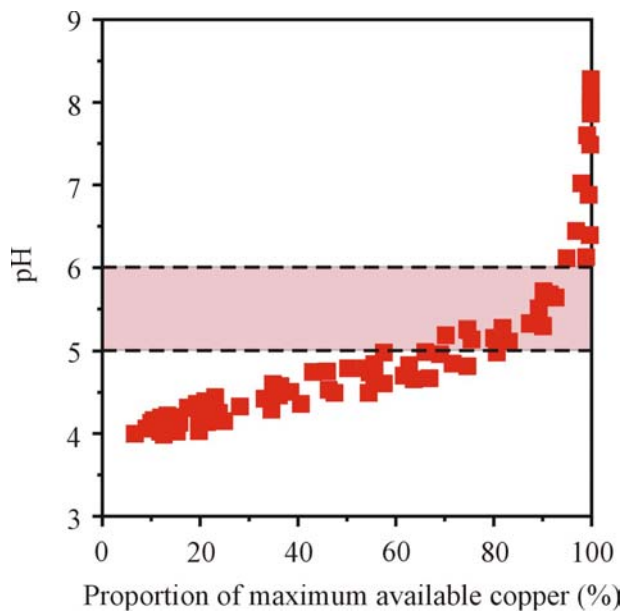
The figure shows that the affinity of different metals to soils is similar independent of soil composition. The values in brackets are the adsorbed metal concentrations in the proportion of available metal amounts. In latter case the affinity sequence will change as follows:  $Pb > Cu > Ni$



**Figure 8.** Competitive adsorption of copper and zinc on soil samples with different composition

The initial concentration of Zn (1200 mg/l) is six times higher than that of Cu (200 mg/l), but the affinity of Cu is higher independent of soil composition, because the adsorption of Cu inhibits the Zn adsorption. In carbonate containing samples the role of precipitation exceeds the adsorption's resulting higher immobilization. The precipitation of different metals takes place at different pH, so the extent of immobilization depends not on the concentration but on the pH

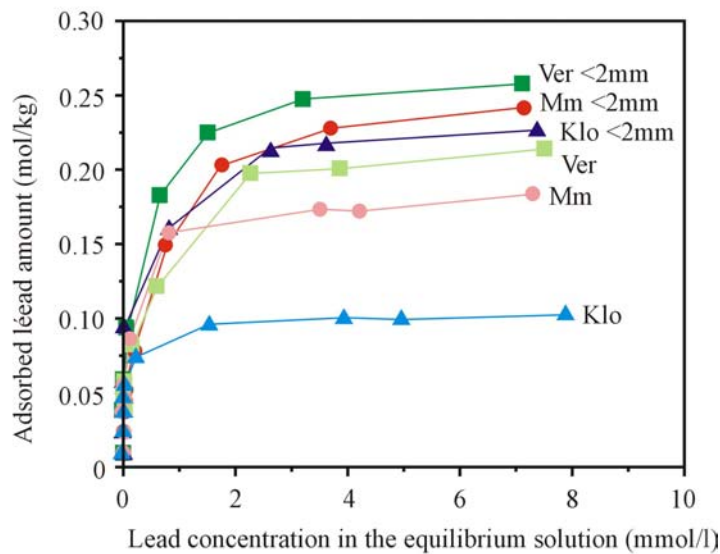
My studies confirmed that the presence of copper and lead has a significant effect on nickel and zinc adsorption, while the inverse is not true (Figure 8). I have shown that the pH is the most important factor affecting adsorption. I have observed that the rapid decreasing of adsorption begins at different pH for each metal, at pH 5,5-6,5 by lead, 5-6,5 by copper, 6-7 by zinc and 6,5-7,5 by nickel (Figure 9).



**Figure 9.** *The relative copper adsorption in the function of pH*

*The relative adsorption of different metals decreases in different pH range, so below a given pH significant immobilization can take place, which is a metal specific process*

8. I have found that the maximum adsorption of studied metals varies similarly in the case of different samples suggesting that the same soil properties control the adsorption of heavy metals. Calcareous soil samples immobilize the highest metal amounts, more than the sample characterized by high organic matter content. Significant decrease in lead adsorption capacity was found after removal of the organic matter content of the samples. The lowest metal amounts were adsorbed by the samples characterized by clay illuviation, but the selectivity of adsorption was the most conspicuous in these cases. I have shown that clay fractions adsorb by 30-40% more heavy metal as compared to whole soil samples do. The affinity of lead to soil samples decreases with the dominant clay mineral species in the sequence of vermiculite > montmorillonite > chlorite, while samples characterized by montmorillonite adsorb more copper and less zinc than the vermiculite dominated ones (Figure 10).



**Figure 10.** The lead adsorption curves of soil samples and soil clays characterized by different clay mineralogy

The soil clay fractions adsorb significantly more metal than whole soil samples. This difference is the highest in the case of chloritic and the less in the case of vermiculitic samples  
*Ver = vermiculite, MM = montmorillonite, Klo = chlorite*

9. The lead adsorption capacity of individual mineral grains was studied by adopting a novel method (TEM-EDS). I have shown that the individual mineral species adsorb metals inhomogeneously (Table 3). Among the mineral constituents of soil clay minerals are the most important lead adsorbents, but also the crystalline Fe-oxides can play an important role in adsorption, while calcite does not adsorb lead at all. The lead adsorption capacity of individual clay mineral phases varies within a broad interval (0-15%), so it is difficult to distinguish them according to their metal adsorbing capacity.

**Table 3.** Lead amounts adsorbed by individual mineral phases

Phase	Adsorbed lead (%)	
Vermiculite	1.92-6.72	* = illite/smectite, illite/vermiculite,
Smectite	5.44-9.85	chlorite/vermiculite,
Illite	0.68-11.03	** = chlorite/vermiculite + illite,
Mixed structures*	1.80-9.12	illite/smectite + illite,
Mixed phases**	2.85-6.07	*** = crystalline Fe-oxide
Fe-oxide***	3.22-4.10	

10. I have found that desorption is also a selective process similarly to adsorption, and the mobility of adsorbed metals increases as follows: Cu < Pb << Zn (< Ni). According to the laboratory experiments the ion exchange is of importance primarily in the samples of high clay mineral content. This process is significant in the case of copper and zinc, as well as of

lead in samples characterized by montmorillonite, and vermiculite, respectively. The ion exchange of zinc can be also considerable in the case of samples rich in organic matter, because the competition has a strong effect on the adsorption of this metal. I have shown that specific adsorption plays an important role in the case of samples having high organic matter content. Metals characterized by high affinity to soils (Pb, Cu) adsorb mainly at these sites. In the case of calcareous samples the precipitation is the primary immobilizing process, and the highest heavy metal amount can be mobilized from these samples (Table 4).

**Table 4.** Extractability of adsorbed metals from the soil samples characterized by different composition

*Different metals is adsorbed at different sites depending on soil sample composition, and the change of the chemical conditions causes mobilisation in different extent in the function of the quality of the metal and of soil composition*

*Exchangeable = MgCl<sub>2</sub>, Specifically adsorbed and precipitated = NaOAc + CH<sub>3</sub>COOH, Easily mobile = distilled water soluble + exchangeable + specifically adsorbed*

	Organic matter rich sample (911)	Carbonate containing sample (915)	Montmorillonite containing sample (1315)	Vermiculite containing sample (913)
Exchangeable	Zn (26%), Pb (13%), Cu (9%)		Zn (42%), Pb (31%), Cu (25%)	Pb (41%), Zn (14%), Cu (6%)
Specifically adsorbed	Pb (39%), Cu (24%), Zn (6%)		Cu (9%), Pb (7%), Zn (4%)	Pb (26%), Cu (19%), Zn (7%)
Precipitated		Ni (80%), Cu (65%), Pb (62%), Zn (44%)		
Easily mobile	Pb (53%), Zn (49%), Cu (35%)	Ni (80%), Cu (65%), Pb (62%), Zn (44%)	Zn (57%), Cu (43%), Pb (39%)	Pb (67%), Zn (35%), Cu (28%)

## Conclusions

1. This study has confirmed the fact that the knowledge of the total heavy metal content of soils is not enough to recognize the effect of pedogenic processes on the behavior of heavy metals exactly. One has also to know the mobility of the heavy metals in soils, which can be effectively studied by sequential extraction experiments. However, these studies can be applied for the examination of heavy metal speciation in soils only under reserve as contrasted with its frequent application. This is supported by the fact that one can not draw any inference from these studies to the role of clay minerals at heavy metal adsorption. One can also get information about the heavy metal mobility in soils from the study of bioavailability of the elements. According to earlier studies plants are able to control the available amounts of heavy metals in soils, so the different plant associations mobilize the chemical elements from the different soil horizons. That is why it is important to study the bioavailability of trace elements in the deeper soil horizons as contrasted with the common practice.
2. Besides the large difference among the mobile metal contents the significant pH difference also renders it more difficult to compare the characteristics under natural and laboratory conditions. I have found that the adsorbed metal amounts in natural soils are negligible as compared to the total heavy metal contents. Competition can not be supposed under such conditions, and no significant amount of heavy metal would be mobilized in the case of an eventual contamination. The laboratory experiments have shown that the main immobilizing process is the precipitation in samples characterized by considerable carbonate content, which is assumed to occur also under natural conditions. The high affinity of lead, copper and nickel to soil organic matter was shown both under natural and laboratory conditions.
3. Soils with high amounts of organic matter and clay minerals, as well as with significant amount of carbonate minerals may be suitable media for the immobilization of an eventual heavy metal contamination. The immobilizing effect of a soil decreases with the increasing number of polluting metals, and a very small pH change (one unit) can result in loss of an important part of this capacity. The advantage of complex soil composition is that soil acts as

a selective buffer for the change of natural conditions. The loss of the buffering capacity of one of the soil compounds does not result in the loss of effect of the other components.

4. The sampled soils from the study area are relatively poor in heavy metals, their concentration is usually below the natural geochemical background in Hungary. This phenomenon is due to the bedrocks, because there are coarse grained and calcareous sedimentary rocks dominating at the surface of the study area, which are poor in trace elements except the volcanogenic formations. According to the studies of Soil Information and Monitoring System (TIM) there are similar heavy metal contents in the soils of agricultural lands of the study area. This means that agricultural activity has not increased the heavy metal content of soils as compared to the forest areas.
5. The total lead content and its distribution are largely determined by the atmospheric deposition, which produced higher lead contents in the upper horizons of soils. The horizons A of the studied soil profiles contain by 50% more lead as compared to the horizons B. The anthropogenic origin of lead is also supported by its unexpected high mobility and availability. The eroded profiles do not show lead enrichment in their upper horizons because of the continuous erosion of the top of the soils.
6. The most widespread soil type of the study area is the forest soil with clay illuviation. This is characterized by average organic matter and varying clay mineral contents, and by the predominance of swelling clay minerals. The spread of soils formed on calcareous bedrock is not important in the study area. That is why the clay mineral content of these soils would play the most important role in the immobilization of a possible heavy metal contamination. Among the studied profiles these soils are characterized by the highest heavy metal contents. These soils are more mature as compared to the other soil types of the study area, so the amount of metal adsorbent phases is also higher. In spite of this fact the most important proportion of metals in these soils is inherited from the parent material. The accumulation horizons of these soils are characterized by the decreasing bioavailability of cobalt, nickel, copper and zinc as compared to the deeper horizons. This is due to the enhanced adsorption capacity of these horizons through the increasing proportion of clay minerals in them. It can be also due to the decreasing plant activity.

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