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Transport of As in the Soil–Plant System in a Long-term Field Experiment

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Introduction

In a review on the health risks induced by microelements entering the food chain, CHANEY (1980, 1982) introduced the concept of the "soil–plant barrier" and classified microelements into four groups. Group 1 is comprised of the elements Ag, Cr, Sn, Y and Zr, which cause little risks, owing to their low solubility in soil and consequently negligible uptake and translocation by plants. Elevated concentrations of these elements in foods usually indicate direct contamination by soil or dust. Group 2 includes the elements As, Hg and Pb, which are strongly sorbed by plant roots, and are not readily translocated to edible plant parts and so pose minimal risks to human health. Group 3 is composed of B, Cu, Mn, Ni and Zn elements. The "soil–plant barrier" may protect the food chain from these elements, which are partly readily taken up by plants, but are phytotoxic at concentrations that lead to little risks to human health. Group 4 consists of Cd, Co, Mo and Se, which give rise to human or animal health risks at plant tissue contents that generally are not phytotoxic.

Of the elements listed above, those commonly raising health concerns of food safety are the heavy metals Cd, Hg and Pb, jointly with anionic metalloids As and Se (REILLY, 1991; MCLAUGHLIN et al., 1999). The historically widespread use of As compounds as insecticides, herbicides and defolians for agricultural production has brought about the elevated As content of soils. These include inorganic salts and organic compounds of both arsenite As(III) and arsenate As(V). High levels of As occur naturally in some sedimentary rocks and in geothermically active areas. Feed additives for poultry and swine production can result manures with high As pool. Arsenic originating from phosphate fertilizers, fossil fuel combustion and municipal sewage sludge are considered as significant sources of soil As (OVERCASH & PAL, 1979; LISK, 1972; CSATHÓ, 1994; KÁDÁR, 1995).

When metals are added as soluble salts, they generally cause greater plant uptake and toxicity than when applied in forms like sewage sludge or metal oxides. Metals are not insoluble forms in sludge, but are fixed with organic matter binding

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sites or are occluded in $CaCO_3$ or in other minerals. Sludge organic matter adds metal sorption capacity to the soil and raises the soil CEC and often the pH. So, the source and form of element applied may strongly affect the result. This is the so-called "salt vs. sludge" error in experimentation for evaluating metal responses.

The 2nd error is called the "greenhouse vs. field" error. Greenhouse studies offer better manageability and reproducibility, and cost less than field studies. However, the measured element contents may be 2-5 fold higher than field measurements of the same soil and crop. In pots plant roots grow in a small volume of treated soil, plants require abnormal watering. The smaller the pots, the greater the error. Plant response differences may originate from the root distribution in the soil with depth, and can only be found right in field studies (MCLAUGHLIN et al., 1999; BUJTÁS et al., 2003; CHANEY, 1980).

Field experiments represent the reality for a given soil–plant system. They are indispensable for following the transport of elements not only into the various crops, but also further into the food chain, their downward migration, and for assessing the long-term fate and effects of elements under natural circumstances. Field experiments can serve with sufficient plant material for feeding experiments with domestic animals. Repeated sampling of the same plant stand on the same plots several times during the growing period provides more reliable data. The sampling procedures are standardized when plant analysis data are used to establish the nutrient requirement of the crops or to obtain comparable results on the toxic element content of the plants for risk assessment.

The purpose of the present research was to evaluate the long-term effect and movement of some microelement contaminants in the soil–plant–animal food chain with the following topics for investigations:

– Behaviour of these elements in soil (fixation, availability, leaching etc.).

– Effect of these elements on crops (yield, quality, accumulation of element in different plant organs, interactions with environmental stress factors, like pests attack, weediness, water shortage).

– Effect of contaminants entering the plants on domestic animals in feeding experiments (health state, accumulation of elements in different animal organs, retention, excretion, growth performances, biochemical values).

– Effect of contaminants on soil life (soil biological activity, recording of macro- and microorganisms like AM fungi, nematode assemblage etc).

The details and main findings of the long-term experiment were published earlier: the summarized experimental data were published by crops yearly (KÁDÁR, 1995, 2001; KÖVES-PÉCHY et al., 1994; KÁDÁR & NÉMETH, 2003; NÉMETH & KÁDÁR, 2005).

Material and Methods

The trial was set up at the Experimental Station of RISSAC in Nagyhörcsök (Mezőföld region, Hungary) on a calcareous chernozem soil formed on loess, containing 5% $CaCO_3$ and 3% humus in average in the ploughed layer. The soil has a loamy texture, with 20% clay (consisting of illite (~ 50%), chlorite (30%) and smectite) and 40% fine fraction. Soil characteristics of the ploughed layer are: pH(KCl): 7.3, AL-P_2O_5: 80–100, AL-K_2O: 140–160, KCl-Mg: 150–180, KCl+ EDTA soluble Mn, Cu and Zn: 80–150, 2–3 and 1–2 mg/kg, respectively. Based on the methods and limit values developed by the Hungarian Extension Service (MÉM NAK, 1979) the soil is supplied well with Mn, sufficiently with Mg and Cu, moderately with N and K, and weakly with P and Zn. The water table is at a depth of 13–15 m, which practically excludes its contamination by leaching. The climate is dry, the area is drought sensitive with an annual precipitation of 500–550 mm and a negative water balance.

The applied treatments simulate soil contamination conditions that may occur nowadays or in the future in the polluted environment of industrial areas, near highways, settlements and in city gardens. The 4 load levels (0, 90, 270 and 810 kg/ha per element) were applied once at initiation in spring 1991 under maize in the form of $AlCl_3$, $NaAsO_2$, $BaCl_2$, $CdSO_4$, K_2CrO_4, $CuSO_4$, $HgCl_2$, $(NH_4)_6Mo_7O_{24}$, $NiSO_4$, $Pb(NO_3)_2$, Na_2SeO_3, $SrSO_4$, $ZnSO_4$. Fertilization was done yearly with 100–100–100 kg/ha N, P_2O_5 and K_2O active agents, in the form of ammonium nitrate, superphosphate and potash fertilizers. The 13×4 = 52 treatments with 2 replications were arranged in split-plot design, in altogether 104 plots. The size of the plots was 6×6 m = 36 m^2.

Soil samples were taken several times during the last 10 years. Each composite sample consisted of 20 cores drawn from the 0–20 cm plough layer of each plot. Plant samples – 20 plants or 40–50 plant parts per plot – were collected every year randomly. Plant and soil samples were dried at 40 ºC, milled and digested with the mixture of cc. HNO_3 + cc. H_2O_2. The soil samples were also extracted by NH_4-acetate + EDTA according to LAKANEN and ERVIÖ (1971). The macro- and microelement contents of the samples were measured using ICP technique in all cases.

The crop sequence of the long-term experiment was as follows: maize (1991), carrots (1992), potatoes (1993), peas (1994), beetroot (1995), spinach (1996), winter wheat (1997), sunflower (1998), garden sorrel (1999) and winter barley (2000).

Results and Discussion

Table 1 shows the crops cultivated in the first 10 years and their harvested main yields. Within the 10-year period only two years (1998 and 1999) had higher rainfall than the many-year average. Previously, the cultivated crops usually had dried out the upper 1 m soil layer, up to the harvest of sunflower in 1998. In 1998 the area received 682 mm, while in 1999 830 mm precipitation. The garden sorrel in 1999 utilized around 300–400 mm water during its short growing season, so in this year it is probable that 400–500 mm surplus moisture accumulated in the soil.

The first grown crop (maize) in 1991 and sunflower in 1998 revealed no yield restrictions. At the same time, carrot roots in 1992, potato tubers in 1993, peas grain

Table 1
Effect of As loads on the yield of harvested crops (t/ha), 1991–2000
(Long-term field experiment, Nagyhörcsök)

Year	Crop sorts and crop parts	As (kg/ha) applied in spring 1991				$LSD_{5\%}$
		0	90	270	810	
1991	maize grain*	7.6	8.6	7.9	6.9	2.5
1992	carrot root	17.6	15.1	19.0	13.3	4.8
1993	potato tuber	12.1	14.4	11.1	10.2	3.5
1994	peas grain*	2.4	2.6	2.3	0.4	0.8
1995	red beet root	14.7	17.1	17.6	12.3	8.7
1996	spinach leaves	18.2	16.0	13.3	10.2	6.5
1997	winter wheat grain*	7.0	7.2	6.8	4.4	1.0
1998	sunflower seed	2.5	2.3	2.8	2.5	0.6
1999	garden sorrel top	42.7	37.7	44.0	31.0	9.9
2000	winter barley grain*	5.4	4.2	4.3	2.1	1.3

*Air-dried

in 1994, spinach leaves in 1996, winter wheat grain in 1997, garden sorrel top in 1999 and winter barley grain in 2000 were significantly depressed in the maximum As treatment (810 kg/ha). Peas, winter wheat and winter barley were the most sensitive to As loads. In the 4th year of the experiment, the peas died out in the case of the highest contamination level (Table 1).

VLADIMIROV (1945, cit. in: OVERCASH & PAL, 1979) proposed that shallow root species should be avoided in favour of deep rooting species to increase tolerance to applied As. On this calcareous chernozem loamy soil, deep rooting crops like maize and sunflower were not affected by As loads. These crops seemed to be very tolerant and quickly grew out of the contaminated upper soil layer. In the case of shallow rooting ones (potato, red beet, garden sorrel) yield loss was only observed in the maximun As treatment (810 kg As/ha).

Much research has been conducted on the effect of As in agricultural plant–soil systems with specific attention to As(III) and As(V). Long-term data on its effects on a variety of crops are available, often where soil sterilization has occurred. JONES and HATCH (1945) showed that after 20–25 years of spraying with lead arsenate there was a 20–30-fold increase in the As content of some Oregon soils, reaching 40–115 mg/kg. COOPER et al. (1931) – studying the effect of Ca-arsenate on the productivity of certain soils in South Carolina – found that sandy soils were seriously affected by 25–100 pounds per acre applications, while fine-textured soils were not. Yields of wheat grown in good soil, however, were greater following 500, 750 and 1000 pounds/acre Ca-arsenate applications than in the untreated check plots. In the Nagyhörcsök long-term experiment somewhat higher maize grain, potato tuber and red beet root yields were obtained on the moderate As levels, but these yield surpluses were not proven statistically.

Table 2 represents the effect of As loads on the As content of the soil's ploughed layer. In the uncontaminated control soil, the NH_4-acetate + EDTA soluble fraction was below the 0.05 mg/kg detection limit. There were large unrevealed fluctuations during the sampling time. 2.5 months after mixing the pollutants into the soil only 20–25% of the applied As could be detected in this form. In the next month the values were 12–23%, in 1992 20–34%, in 1994 13–30%, in 1997 10–16%, and in 2000 10–13%, resp. The "total" As digested with cc. HNO_3 + cc. H_2O_2 was 70–82% of the amount added four years earlier. The soil profiles were sampled in the 10th year of the trial. The mixed samples consisted of 5 cores/plot and were taken every 30 cm down to 290 cm, and the NH_4-acetate + EDTA-soluble fractions were determined. As displayed no significant vertical movement (KÁDÁR & NÉMETH, 2003; NÉMETH & KÁDÁR, 2005).

Table 2

Effect of As loads on the As content of the soil's ploughed layer
(Long-term field experiment, Nagyhörcsök)

Sampling time		As (kg/ha) applied in spring 1991				$LSD_{5\%}$
month	year	0	90	270	810	
		NH_4-acetate + EDTA soluble, mg/kg				
July	1991	–	7	18	66	14
August	1991	–	7	15	32	13
November	1992	–	6	31	93	17
April	1994	–	4	21	80	9
July	1997	–	3	11	42	3
September	2000	–	3	12	37	3
		cc. HNO_3 + cc. H_2O_2 "total" content, mg/kg				
April	1994	7	28	81	210	34

Remark: –: below the detection limit of 0.05 mg/kg

The chemistry of As resembles that of P in soil. It appears that As remains in the surface zone. It is known that only a few percent of the total As is extractable with diluted acids in the case of soils not receiving As doses earlier. Plant toxicity/response is related to the available, easily extractable or solution phase As concentration. In a well aerated calcareous soil it is expected that the Ca-arsenate As(V) form predominates. Arsenate is absorbed by plant roots via the transport system of P, and so phosphate competitively inhibits As uptake.

Unlike phosphate, the translocation of As to shoots is generally low and the roots are the primary site of injury when As reaches phytotoxic levels. Consequently, aerial parts of crops typically contain little As, usually less than 2 mg/kg D.M. Crop failure is generally expected before tissue As levels are in concern of human health. Elevated As intake is most likely to arise from As-containing drinking water rather than from ingestion of food. Sometimes, this is the case in Hungary, where the geothermic activity is high and groundwater is often rich in As.

As it can be seen in Table 3, maize roots accumulated larger quantities of As than shoots at 6-leaf stage, while the concentration of As in the leaves at flowering as well as in the straw and grain at harvest was below the 0.1 mg/kg dry matter detection limit. In 1992 the foliage of carrot grown in the soil treated with the highest As load had an As content of 1.3–3.6 mg/kg dry matter. Carrot roots were not contaminated at all. Likewise, in 1993 potato tops accumulated 0.5–3.0 mg As/kg dry matter, but tubers were not contaminated. In 1994 the straw of green peas absorbed As up to 2.4 and 7.2 mg/kg dry matter, while its pods and grain were left uncontaminated. In 1995, however, the tops and roots of red beet grown in the soil treated with higher As loads were highly contaminated.

Peas are legume crops sensitive to As contamination. As shown earlier (Table 1), on the control soil 2.5 t/ha air-dry grain yield was achieved, which decreased to 0.4 t/ha with the maximum As load. As accumulation was only significant in the vegetative organs of the pea plants and not in the seed, despite the extreme pollution. Higher As loads practically eliminated nodule formation and thus the atmospheric N fixation on the roots. The endomycorrhizal symbiosis also suffered damage in polluted soils, where the emergence and early development of both peas and weeds were inhibited. In the course of the vegetation period, as toxicity increased, the plant roots were unable to "grow out" of the polluted soil (KÁDÁR, 1995, 2001; KÖVES-PÉCHY et al., 1994).

High amounts of As (reaching 9–13 mg As/kg dry matter) accumulated in red beet tops and roots in the treatment with maximum As load. On this contaminated soil the total As uptake (root+top together at harvest) amounted to 30–40 g As/ha. On the basis of these data it can be concluded that the phytoremediation of highly contaminated soils does not seem to be the real answer to the problem of As pollution, using this crop. Theoretically, it would take several thousands of years to clean the soil in such environment. It can also be stated that maize grain, carrot roots, potato tubers and green peas grain remained clean and suitable for human or animal consumption, even on the soil highly contaminated with As. Only the red beet top and root should be discarded.

It is specified that the permissible As contamination levels are as follows (Decree 9/2003):

– for grain flour up to 0.1 mg As/kg dry matter;
– for sunflower seed to 0.2 mg As/kg D. M.;
– for fresh fruits and vegetables to 0.2 mg As/kg F. M.;
– for dry pulses to 0.5 mg As/kg D. M.,
– for dried fruits and vegetables to 2.0 mg As/kg, and
– for animal basic feeds to 2.0 mg As/kg D. M.

As it can be seen in Table 3, the spinach crop in 1996 accumulated 3–4 mg As/kg dry matter in its vegetative organs in the maximum As treatment, which was above the permissible limits. The same is true for winter wheat, which had higher As contents in the green shoot than in the straw, and lower, but still unacceptable, content in the grain.

The concentration of As declined in sunflower plants from the young leaves to the straw–head–seed direction. The grain As content was around the acceptable

Table 3

Effect of As loads on the As content of crops, mg As/kg air-dry matter
(Long-term field experiment, Nagyhörcsök)

Date of sampling	Crop parts	As (kg/ha) applied in spring 1991				$\text{LSD}_{5\%}$
		0	90	270	810	
Maize (Zea mays L.) in 1991						
8 July	Root[1]	<0.1	6.8	8.2	23.0	8.9
8 July	Shoot[1]	<0.1	0.8	1.1	1.3	0.3
8 August	Leaves[3]	<0.1	<0.1	<0.1	<0.1	–
25 November	Straw[5]	<0.1	<0.1	<0.1	<0.1	–
25 November	Grain[5]	<0.1	<0.1	<0.1	<0.1	–
Carrot (Daucus carota L.) in 1992						
29 June	Top[2]	<0.1	<0.1	<0.1	1.3	0.4
7 October	Top[5]	<0.1	<0.1	0.9	3.6	0.4
7 October	Root[5]	<0.1	<0.1	<0.1	<0.1	–
Potato (Solanum tuberosum L.) in 1993						
14 June	Leaves[3]	<0.1	<0.1	0.1	3.0	0.5
12 July	Leaves[4]	<0.1	<0.1	0.4	0.5	0.3
7 September	Tuber[5]	<0.1	<0.1	<0.1	<0.1	–
Green peas (Pisum sativum L.) in 1994						
26 May	Top[3]	<0.1	<0.1	<0.1	<0.1	–
14 June	Straw[5]	<0.1	<0.1	2.4	7.2	0.4
14 June	Pods[5]	<0.1	<0.1	<0.1	<0.1	–
14 June	Grain[5]	<0.1	<0.1	<0.1	<0.1	–
Red beet (Beta vulgaris L. ssp. conditiva ALEF.) in 1995						
21 June	Top[2]	<0.1	<0.1	2.0	13.4	1.0
7 September	Top[5]	<0.1	<0.1	5.6	12.2	1.5
11 September	Root[5]	<0.1	<0.1	<0.1	9.2	1.2
Spinach (Spinacea oleracea L.) in 1996						
3 June	Leaves[7]	<0.1	<0.1	0.9	3.9	0.2
23 July	Straw[5]	<0.1	0.4	1.7	3.6	0.1
23 July	Grain[5]	<0.1	<0.1	0.3	0.6	0.1
Winter wheat (Triticum vulgare L.) in 1997						
15 May	Shoot[6]	<0.1	0.7	1.2	2.1	0.4
24 July	Straw[5]	<0.1	1.3	1.5	1.9	0.4
24 July	Grain[5]	<0.1	<0.1	0.2	0.4	0.2
Sunflower (Helianthus annus L.) in 1998						
6 July	Leaves[3]	<0.1	0.3	0.8	3.2	0.3
23 September	Straw[5]	<0.1	<0.1	<0.1	0.5	0.5
23 September	Head[5]	<0.1	<0.1	<0.1	0.7	0.3
23 September	Grain[5]	<0.1	<0.1	<0.1	0.2	0.2
Garden sorrel (Rumex rugosus L.) in 1999						
9 July	Top[5]	<0.1	<0.1	1.1	3.6	1.0

Table 3 cont.

Date of sampling	Crop parts	As (kg/ha) applied in spring 1991				$LSD_{5\%}$
		0	90	270	810	
Winter barley (Hordeum vulgare L.) in 2000						
4 May	Flag leaves[3]	<0.1	0.5	1.7	3.6	0.4
20 June	Straw[5]	<0.1	1.5	4.5	6.4	0.6
15 June	Grain[5]	<0.1	0.2	0.4	0.4	0.2

Remarks: [1]: 6-leaf stage, [2]: before root formation, [3]: before flowering, [4]: after flowering, [5]: at harvest; [6]: end of tillering; [7]: middle of vegetation period. <: below the detection limit of 0.1 mg/kg dry matter

value (0.2 mg/kg D. M.) even on the soil receiving the maximum load. Garden sorrel tops were contaminated significantly only in the maximum As treatment, exceeding here the permissible limit values for human use. The As content of winter barley was already significant at the 90 kg As/ha load. In the higher As treatment both grain and straw were unsuitable for animal or human consumption (Table 3).

Summarizing the above, it can be stated that the mobility of As within the soil–plant system is inhibited at this site. Even on the heavily loaded soil, the amount of As in the whole above-ground biomass remained negligible, usually below 5–10 mg As/kg dry matter. Under such conditions, the remediation of the 810 kg/ha As load would theoretically take many thousands of years via plant uptake. Phytoremediation might serve as a solution only on a low diffusion site, if satisfactory hyperaccumulator plant species and high production technology are available. The transfer coefficient, expressing the total straw/soil As concentration by barley had a value of 0.02. So, As does not seem to be a dangerous contaminant either for soil, plants or groundwater. The extreme load, however, caused the phytotoxicity of some crops and in some cases resulted in products unfit for animal or human consumption. The effect of pollution on root-symbiotic microorganisms like Rhizobium and AM fungi was also harmful.

Summary

The first grown deep-rooting crop, maize in 1991 and sunflower in 1998 did not reveal any yield loss on the contaminated soil. The crops responding most sensitively to As were peas, winter wheat and winter barley. In the 4th year of the trial the peas practically died out on the highest As level.

The “total” As (digested with cc. HNO_3 + cc. H_2O_2) amounted to 70–80% of the As added to the ploughed layer 4 years earlier. The NH_4-acetate + EDTA soluble As fractions revealed great fluctuations during the sampling time. In the first 4 years the ratio of As detectable in the ploughed layer in this form ranged between 12 and 30%, while in 2000 between 10 and 13%. On the basis of deep profile sampling, it was established that As displayed no significant vertical movement after 10 years, using the HN_4-acetate + EDTA method (LAKENEN & ERVIÖ, 1974).

The concentration of As, as a rule of thumb, declined in the direction from root–shoot–leaves–straw–grain in grain crops. The mobility of As is limited within the soil–plant system at this site. Even on the heavily loaded soil, the amount of As in the whole above-ground biomass remained negligible, usually below 5–10 mg As/kg dry matter. Under such conditions, the remediation of soil contaminated with higher loads would theoretically take thousands of years in the case of As via plant uptake.

The transfer coefficient, expressing the total straw/soil As concentration by barley, had a value of 0.02. As does not seem to be a very dangerous contaminant either to soil, plants or groundwater. Extreme loads, however, caused phytotoxicity in some crops and resulted in products unfit for animal or human consumption. Soil life was also damaged. Higher As loads decreased nodule formation by green peas, and the endomycorrhizal symbiosis was hindered as well.

Key words: long-term field experiment, As loads, transport of As, As content of soil, As content of crops

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