Immobilisation of As, Cd, Pb and Zn in agricultural soils by the use of organic and inorganic additives

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ABSTRACT

The efficiency of the application of organic and inorganic additives on the reduction of mobility and transfer of As, Cd, Pb and Zn from the soil into the plants was observed. The dung, compost, acid peat and muck presented organic additives. Synthetic zeolite – type P_c of cubic structure, loamy shale and dolomite limestone presented inorganic additives. Five soil types were used during the testing (arenic regosol, typic cambisol, dystric cambisol, typic chernozem and typic fluvisol). The changeover of the mobility of As, Cd, Pb a Zn in the soil (the ratio of mobile and total contents, sequential analysis) and the transfer of the elements from the soil into the plants (the vegetables, cereals and fodder plants) were investigated. The results showed the primary importance of the soil pH value on the behaviour of potentially toxic elements and their intake by the plants. The efficiency of the use of organic additives strongly depended on the quality of the organic matter. Inorganic additives on the base of the sorbents worked in the case of mobile hazardous elements (Cd, Zn). The efficiency of the soil additives was strongly influenced by the soil type.

Keywords: hazardous elements; soil remediation; immobilization; soil additives; the dung; the compost; acid peat; the muck; synthetic zeolite; loamy shale; dolomite limestone

The contamination of the environment by hazardous substances is under intensive investigation in the present time. The soil is a medium, which could absorb the loads of other components of the environment and which can influence these components conversely.

In the framework of soil contamination the most important risk components were characterised in the Czech Republic (MŽP ČR 1994). The basic research is directed to the determining of the limit values of the contaminants in the soils (Podlešáková et al. 1996). The other approaches deal with the behaviour of contaminants in the pedosphere and with the transfer of the contaminants into the other components of the environment (Hornburg and Brümmer 1990).

The next topic is to evolve the methods, which can reduce the negative effects of soil contamination. The development of remediations is successful in the field of organic pollutants. Many techniques of decontamination are used (Svoma 1993). The remediation of the soils contaminated by hazardous elements is more difficult still. The potentially toxic elements cannot be broken down in the soil and cannot be easily taken up (Rulkens et al. 1993, Thöming and Calmano 1995). This is the reason for development of the remediation techniques in agriculturally used soil. These techniques should reduce the negative impact of hazardous elements in the soil (Vangronsveld et al. 1996). The immobilisation of hazardous elements in the soil by application of the sorbents is one of the possibilities (Mench et al. 1993, Kolář et al. 1998, Sun and Doner 1998, Lepp et al. 2000). Some of the immobilisation techniques are the theme of this article. The objective is to investigate the possibility of the use of inorganic and organic sorbents for the immobilisation of As, Cd, Pb and Zn in the soils with increased values of these elements.

MATERIAL AND METHODS

The testing of organic and inorganic soil additives, which were used for immobilisation of hazardous elements in the soil, was done in pot trials, field trial and laboratory experiment. The organic and inorganic soil additives represent available and economically realistic materials. The soils contaminated in the terrain (anthropogenic loads by immission fall-outs and floodwater) were used. Artificial contamination by the salts of hazardous elements has not been used.

Soil additives tested in pot trials, field trial and laboratory experiment are presented in Table 1. The synthetic zeolite (pH 10.05) – type P_c of cubic structure (Kovanda et al. 1996), loamy shale (pH 6.45) and dolomite limestone (pH 8.2) were used as inorganic additives (finely milled). The dung (pH 7.6, C_{ox} 21.9%), the compost (pH 7.2, C_{ox} 35.5%), acid peat (pH 3.8, C_{ox} 30.1) and muck-material of sapric histosols (pH 7.4, C_{ox} 7.2%) were used as organic additives. The additives were incorporated into the soil and incubated for one year.

The content of hazardous elements, pH value, C_{ox} (organic additives), soil texture and sorption characteristics in the soil additives were measured. The mass of organic additives was derived from the dose of organic fertilisa-

Table 1. The used soil additives

	Organic sorbents	Inorganic sorbents
Pot trial	the dung	loamy shale
	the compost	synthetic zeolite
	the muck	
	acid peat	
	green manure (fresh clover)	
Field trial	the muck	loamy shale
	acid peat	synthetic zeolite
		dolomite limestone
Laboratory experiment	the muck	loamy shale
	green manure (fresh clover)	synthetic zeolite
		dolomite limestone

tion with the manure (40 $t.ha^{-1}$ of fresh matter every 3 years). The comparison with the content of organic carbon was performed. This mass of organic additives was increased six times in the trial.

The dose of inorganic additives was used as 1% (synthetic zeolite, the limestone) and 3% (loamy shale) of the weight of soil dry matter.

The nomenclature of soil types was updated by classification of Němeček et al. (2001).

Pot trial

The trial was set up in 1995 and the testing was done in Mitscherlich pots filled with 6 kg of the soil (the sieve 5-mm). Following soil types were used in the trial:

- CAd – dystric Cambisol, the locality Mikulov v Krušných horách
- CAm – modal Cambisol, the locality Příbram
- CEm – modal Chernozem, the locality Slaný
- RGa – arenic Regosol, the locality Boletice nad Labem
- FLm – modal Fluvisol, locality Mladá Boleslav

The variants were used in four replications. Pot trial was placed into the greenhouse and sowed by spinach (1996), radish (1997), mustard (1998), carrot (1999) and corn (2000). Following parameters were determined:

- pH value (0.2 M KCl)
- C_{ox} content (Tjurin method)
- the content of strongly bound fulvo and humic acids
- the content of weakly bound fulvo and humic acids
- quotient $Q_{4/6}$

The total content (extraction by mixture of the acids $HNO_3 + HClO_4 + HF$) of hazardous elements (As, Cd, Pb, Zn) in the soils were measured by AAS method (Podlešáková et al. 1996). The mobile (0.01M $CaCl_2$ and 1M NH_4NO_3) and potentially mobilizable forms (0.025M EDTA) of hazardous elements in special cases were measured, too (Podlešáková et al. 2001). The sequential analysis (Zeien and Brümmer 1989) for determining of binding forms of hazardous elements in the soil was used.

The content (extraction by $HNO_3 + HClO_4$) of hazardous elements (As, Cd, Pb, Zn) in the leaves of barley (the stage of 5th leaf), spinach and maize (green maturity, 60 cm high) and in the root of carrot and radish were measured by AAS method (Podlešáková et al. 2001). The laboratory analysis was done in the central lab of the Research Institute for Soil and Water Conservation.

Field trial, locality Litavka

The field experiment on the contaminated fluvisol in flood area of Litavka River was started in 1998. Heavy contamination by Cd, Pb and Zn was detected in this locality (Borůvka et al. 1996).

The variants were used in four repetitions. The area of the plot was 1 m × 1 m. The experiment was sowed with

Table 2. Total content of hazardous elements in used soils ($mg.kg^{-1}$)

Locality	Soil type	pH	C_{ox}	As	Cd	Zn	Pb
Boletice n. L.	RGa	6.80	2.83	23.93	1.37	362	57.1
Mikulov	CAd	4.50	4.26	2830	1.89	580	108
Slaný	CEm	6.80	2.43	42.40	3.75	948	257
Příbram	CAm	4.80	2.07	66.30	7.32	524	1378
Mladá Boleslav	FLm	4.45	2.59	18.80	1.41	619	146
Litavka	FLm	5.30	4.21	24.60	15	1900	1200

Table 3. Quality of organic matter in used soils (pot trial)

		pH	C_{ox} (%)	C/N	Humic acid (%)	Fulvic acid (%)	Ha:Fa	$Q_{4/6}$	Ha_s:Ha_l
RGa	control	6.62	3.13	13.6	0.35	0.26	1.35	6.96	1.09
	dung 6×	6.62	3.33	12.8	0.54	0.28	1.93	6.42	1.45
	compost 6×	6.73	3.67	13.1	0.41	0.27	1.52	6.47	1.02
	muck 6×	6.85	3.53	12.6	0.51	0.34	1.50	5.42	1.54
CAd	control	5.37	4.62	12.2	0.73	0.82	0.89	6.12	0.78
	dung 6×	5.81	4.93	11.2	0.75	0.83	0.91	6.35	0.86
	compost 6×	5.79	5.11	11.6	0.67	0.81	0.83	6.67	0.64
	muck 6×	6.04	4.86	11.5	0.72	0.83	0.86	5.88	0.86
CEm	control	7.15	2.75	13.1	0.36	0.23	1.57	4.33	4.00
	dung 6×	7.09	3.38	12.1	0.48	0.28	1.71	4.60	1.40
	compost 6×	7.13	3.52	12.4	0.49	0.29	1.69	4.75	2.73
	muck 6×	7.11	3.36	12.0	0.66	0.36	1.83	4.70	3.30

mustard (1998), rye (1999) and mustard and oat (2000). The mustard has been harvested in the flowering stage, the rye in the stage of full maturity and the oat in the 5^{th} leaf stage (1^{st} half of the yield) and in the stage of full maturity (2^{nd} half of the yield). The yield of the plants (leaves of the mustard and oat, grain of the rye and oat) was determined. The laboratory analysis was identical with pot experiments.

Laboratory experiment

The efficiency of soil additives on the sorption of hazardous elements and the stability of the sorption in different soil pH was determined in laboratory experiment. The soil (CAm, locality Příbram) was sieved (2 mm), stirred with materials and incubated (20°C) in variable water conditions for 6 month. The volumes filled with soil were extracted by solutions (dilute solution of HNO_3) at four pH levels (3, 4, 5, 6). Every volume was extracted (6 hours) four times by each solution. The conductivity, pH value and content of hazardous elements (As, Cd, Pb, and Zn) were measured. The methodological approach was based on the experiences with sorption-desorption experiments (Kozák and Jehlička 1991). The content of observed hazardous elements in used soils is presented in Table 2.

RESULTS AND DISCUSSION

Pot experiments

The results of pot experiment with organic additives inform about the efficiency of organic additives on the immobilisation of hazardous elements in different soil types (Podlešáková et al. 1998). For the explanation, the differences between pH values and quantity and quality of organic matter were observed. The results are presented in Table 3.

The influence of the application of organic materials on the pH is noticeable in dystric cambisol only. The influence of incorporation of organic materials on pH values in fluvisol and chernozem was marginal.

The content of C_{ox} was slightly increased after application of muck into the dystric cambisol and compost into the arenic regosol. The reduction of the ratio C/N in the soils after incorporation of organic materials was detected. The decreasing of $Q_{4/6}$ after application of the muck into the dystric cambisol and arenic regosol shows positive changeover of the quality of organic matter.

The increasing of pH value was detectable after incorporation of synthetic zeolite and loamy shale into the modal cambisol in pot experiment with inorganic and organic materials. The application of acid peat and green manure slightly decreased the pH value of soil.

The results of the CEC, base saturation (BS), content and quality of organic matter measurement are presented in Table 4. The incorporation of loamy shale slightly increased cation exchange capacity (CEC). This tendency was not noticeable in the case of synthetic zeolite, unexpectedly. After incorporation of acid peat the content of C_{ox} was increased.

Sequential analyse (Zeien and Brümmer 1989) of dystric cambisol (locality Mikulov) and typic cambisol (locality Příbram) for the understanding of changeover of existing binding forms of hazardous elements in the soil was measured. The results inform about the high efficiency of the incorporation of the materials into soil on the decreasing of Cd and Zn mobility. The decreasing of water-soluble and exchangeable fraction of Cd in the soil after application of the muck into dystric cambisol was detected. The content of organic and residual bound Cd in the soil increased, too.

The incorporation of synthetic zeolite into the modal cambisol decreased the content of Cd water-soluble and

Table 4. Sorption characteristics and quality of organic matter in used soils (pot trial)

Soil	Sorbent	pH	CEC	S	BS (%)	C_{ox} (%)	Chk (%)	Cfk (%)	Chk:Cfk	$Q_{4/6}$
					Pot trial II					
FLm	control	6.76	106.2	126.3	92.50	2.04	0.35	0.27	1.30	6.10
	synthetic zeolite	6.84	124.8	81	64.00	2.20	0.33	0.26	1.27	5.62
	loamy shale	6.81	152.5	126.3	82.80	2.32	0.34	0.27	1.26	5.70
	peat	6.65	150.0	113.8	75.80	2.57	0.41	0.31	1.32	5.60
	green manure	6.69	129.8	118.5	91.25	2.15	0.36	0.30	1.20	6.35
CEm	control	7.25	211	190.8	89.25	2.16	0.40	0.28	1.43	4.15
	synthetic zeolite	7.25	192.8	192.8	100.0	2.04	0.31	0.28	1.10	4.30
	loamy shale	7.24	227	225.8	99.50	2.15	0.35	0.25	1.40	3.90
	peat	7.12	236	236	100.0	2.66	0.47	0.32	1.47	4.55
	green manure	7.11	212	212	100.0	2.18	0.42	0.30	1.40	4.40
CAd	control	5.01	116	33	34.70	1.85	0.39	0.41	0.96	5.80
	synthetic zeolite	5.67	116.3	21.8	23.60	1.80	0.36	0.37	0.97	5.90
	loamy shale	4.98	137	57	41.50	2.08	0.35	0.37	0.95	6.10
	peat	4.59	135	32.5	23.10	2.59	0.48	0.47	1.02	5.80
	green manure	5.06	137	42	30.53	1.99	0.43	0.43	1.00	5.90

CEC – $mmol^+.kg^{-1}$, S – $mmol^+.kg^{-1}$

exchangeable fraction in the soil but easily mobilizable fraction increased. Water-soluble and exchangeable fraction of Cd after application of loamy shale was decreased in a lesser extent. The increasing of residual fraction of Cd and Pb, in lesser extent of As, was detected after the use of loamy shale. The incorporation

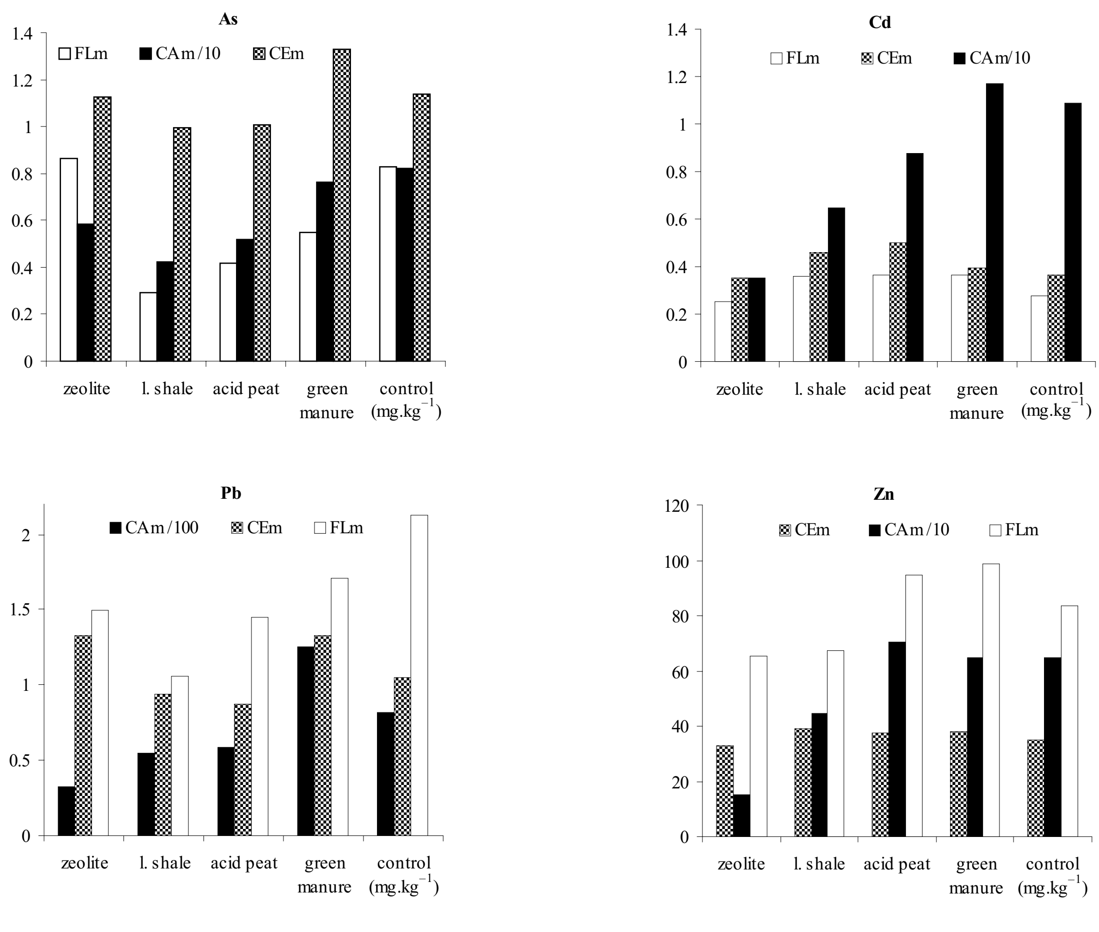

Figure 1. The content of As, Cd, Pb, and Zn in the root (radish)

Table 5. Correlation ratios between C_{ox} and pH of the soils and between content of mobile forms of hazardous elements in the soils and content of hazardous elements in the plants (field trial)

Content	Element	pH	C_{ox}
Extract NH_4NO_3	As	–0.42	0.16
	Cd	**–0.54**	–0.13
	Pb	**–0.87**	0.30
	Zn	**–0.97**	0.30
Extract $CaCl_2$	As	**–0.58**	0.29
	Cd	**–0.86**	0.22
	Pb	**–0.83**	0.26
	Zn	**–0.93**	0.32
Extract EDTA	As	–0.14	–0.37
	Cd	–0.25	–0.04
	Pb	–0.23	–0.35
	Zn	**–0.57**	0.04
Rye-grain	As	**–0.51**	0.03
	Cd	**0.71**	–0.39
	Pb	–0.09	0.01
	Zn	**–0.63**	–0.03
Oat-grain	As	–0.38	0.08
	Cd	**–0.50**	–0.06
	Pb	0.26	–0.25
	Zn	–0.41	0.30
Green oat	As	–0.21	–0.13
	Cd	**–0.65**	0.24
	Pb	–0.14	0.05
	Zn	**–0.81**	0.33
Mustard 98	As	**–0.55**	0.29
	Cd	**–0.90**	0.21
	Pb	**–0.51**	0.10
	Zn	**–0.89**	0.24
Mustard 00	As	**–0.50**	0.21
	Cd	**–0.79**	0.24
	Pb	**–0.69**	0.26
	Zn	**–0.91**	0.28

of acid peat and green manure increased water-soluble and exchangeable fraction of Cd and Zn in the soil. Organic bound fraction of Cd and Zn were not increased in acid conditions.

In the first pot experiment the incorporation of muck into dystric cambisol reduced the transfer of Cd and Zn into the root of carrot and radish. The slight reduction of Pb transfer was detected, too. The efficiency of compost on the reduction of transfer of Cd and Zn was lower. The application of the dung increased the transfer of Cd and As in some cases. The efficiency of organic materials is strongly influenced by the quality of organic matter.

Synthetic zeolite decreased the content of Cd and Zn, in lesser extent Pb in the roots of radish in second pot trial (Figure 1). Synthetic zeolite increased transfer of As. Loamy shale reduced the content of Cd and Zn in the roots of radish in lesser extent, but slightly decreased the content of As. Positive influence on the decreasing of Pb content was detected. The efficiency of the materials on the transfer of hazardous elements into shoots of rye and corn was low.

Field trial

The influence of the incorporation of materials on the behaviour of hazardous elements and quantity and quality of crops was observed. The behaviour of hazardous substances was observed by the use of sequential analysis (Zeien and Brümmer 1989). The influence of the incorporation of materials on the mobility of As in the soil was marginal. Cd shows different trends and Cd water-soluble and exchangeable fractions in the soil were reduced after the use of muck, dolomite limestone, synthetic zeolite > loamy shale. The efficiency of these materials on the decreasing of potentially mobilizable fraction was low. The best efficiency showed the muck, because of increasing of organic and residual bound fraction of Cd in the soil. Organic bound fraction of Pb was detected. The high dependence of Zn > Cd on the changeover of pH values after incorporation of materials into the soil was confirmed by the use of the correlation analysis (Table 5).

The transfer of the hazardous elements was strongly influenced by the crop. The content of Cd, Zn > Pb in the shoot of mustard was reduced after the incorporation of the muck, dolomite limestone, synthetic zeolite and loamy shale. Acid peat increased the transfer of Cd, Zn and Pb into the mustard. The differences between variants were reduced in the case of shoot of the oat. The differences in the accumulation of As, Cd, Zn and Pb in the rye (grain) and oat (grain) between the variants were marginal.

The positive influence of the materials (except acid peat) on the yield of mustard, rye and oat was determined (Figure 2).

Laboratory experiment

The use of extraction of the soil with incorporated materials contributed to explain the influence of some conditions on the mobility of hazardous elements in the soil. The pH and conductivity of the extracts were measured in the first step. The pH of solution and number of the volume influenced the pH value of the extracts slightly. The pH was strongly influenced by the incorporation of the material. The neutralisation tendency of the soil additives was detected in order synthetic zeolite, muck, dolomitic limestone, and loamy shale. Green manure caused slight acidification tendency (first volume only). The conductivity was strongly increased after the use of green manure (in first volume) and synthetic zeolite (content of Na ions). Green manure was decomposed in the soil and extracted from the soil profile in the first volume quickly (increased conductivity in the first volume and

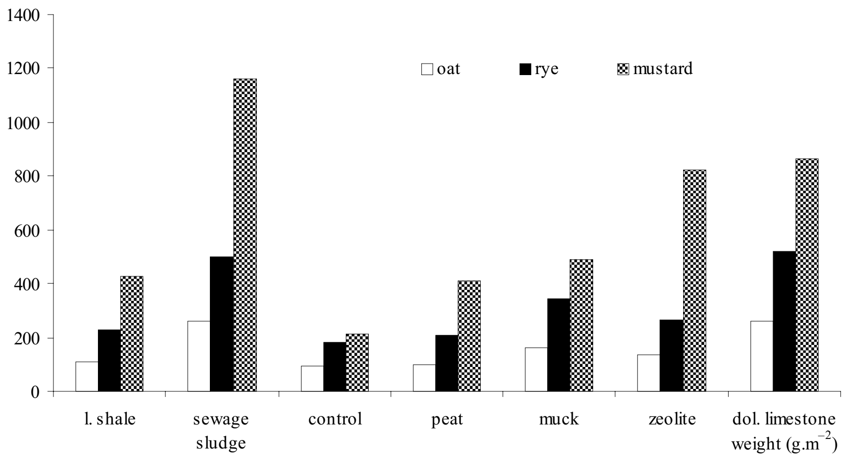

Figure 2. The yield of the crops (plant tissue) after the use of soil additives (field trial)

quick decrease of the conductivity in the next volumes). This decomposition influenced the extractability of Cd, Zn, Pb > As in the soil, but in the first volumes, only. The reduction of the extractability of Cd, Zn and Pb after incorporation of muck, dolomite limestone and loamy shale is in good agreement with the results of pot and field trials. Loamy shale reduced the extractability of Pb to a greater extent. The extractability of Cd and Zn was strongly decreased after incorporation of synthetic zeolite but the extractability of As and Pb was strongly increased (high concentration of Na and exchange reactions on the surface of synthetic zeolite). Because the content of Pb in the plants was not increased in pot and field trials, the bioavailability of created Pb compounds is low.

On the base of the experiments we concluded, that high effect on the decreasing of the mobility and transfer of Cd and Zn from the soil into the plants was reached after incorporation of materials with neutralisation properties (synthetic zeolite, dolomite limestone, the muck) into acid soils. The pH value was determined as the most important factor, which influenced the mobility of Cd and Zn in the soil. The dependence of Cd and Zn mobility on soil acidity is confirmed by many authors (Gavi et al. 1997, Lebourg et al. 1998, Podlešáková et al. 2001).

High efficiency of the muck (model material) on the decreasing of mobility and bioavailability of Cd, Zn > Pb was determined. The muck influenced the behaviour of hazardous elements not only due to neutralisation ability, but also due to binding of elements on stabile organic substances (Li and Shuman 1996) in the muck.

The application of high doses of the dung and green manure increased the mobility of hazardous elements in the soil. The binding of the potentially risk elements on unstable and mobile organic compounds (fulvoacids) is presented in Krishnamurti et al. (1997) or in McBride et al. (1997).

The use of acid peat in pot and field trial increased the mobility and transfer of Cd, Zn > Pb into plants. The binding of hazardous elements on the organic matter in the peat was not increased in acid properties.

The quality of used organic amendments was of primary importance for the remediation. The importance of the quality of organic matter is emphasised for example by Kolář et al. (1998).

Dolomite limestone and synthetic zeolite immobilised Cd and Zn in the soil and reduced their transfer into plants. Dolomite limestone decreased mobility of Pb, too. Synthetic zeolite increased the mobility and transfer of As into plants. This finding is in good agreement with the results of Lepp et al. (2000). High extractability of As and Pb after application of synthetic zeolite was observed in laboratory experiment.

Loamy shale decreased the mobility of Cd and Zn in lesser extent in comparison with dolomite limestone and synthetic zeolite but positively influenced the decreasing of the mobility of Pb and slightly As.

The efficiency of hazardous element immobilisation in the soil on their plant uptake reduction depends strongly on the crop (Hornburg and Brümmer 1990). The content of hazardous elements in the root of plants and in the shoot of sensitive plants was influenced by the application of the materials. The decreasing of the mobility of hazardous elements in the soil influenced their transfer into generative organs of the plants (grain) to a very low extent.

The use of materials (except acid peat and synthetic zeolite in chernozem) increased the yield of the crop.

The gentle remediations based on the immobilisation of hazardous elements in the soil could be recommended on the soils with low pH and low value of the CEC. The application of the materials had marginal effect in the case of soils with high buffer capacity. The efficiency of soil additives was strongly influenced by the soil type. Tlustoš et al. (2001) informs about different Zn and Pb uptake by the crops planted on different soils treated by sewage sludge. Zn and Pb uptake by the plants was influenced after the incorporation of organic matter into the cambisol predominantly. This finding is in good agreement with our results.

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ABSTRAKT

Imobilizace As, Cd, Pb a Zn v zemědělských půdách aplikací organických a anorganických pomocných půdních látek

Nádobovým, polním mikroparcelkovým a laboratorním extrakčním pokusem byla testována možnost použití anorganických a organických pomocných půdních látek pro účely imobilizace rizikových prvků As, Cd, Pb a Zn v půdě. Organické pomocné půdní látky byly zastoupeny chlévským hnojem (hovězí), statkovým kompostem, slatinnou rašelinou a kyselou rašelinou, anorganické pak syntetickým zeolitem typu P_c s kubickou strukturou, cyprisovými jílovci a dolomitickým vápencem. V pokusech bylo použito pět půdních představitelů (regozem arenická, kambizem modální, kambizem dystrická, černozem modální a fluvizem modální). Byla sledována změna mobility rizikových prvků v půdě (poměr jejich celkových a mobilních obsahů, použití sekvenční analýzy) a transfer rizikových prvků z půdy do rostlin (zelenina, obilniny, pícniny).

Z šetření vyplynul primární význam změny půdní reakce na změnu mobility rizikových prvků a jejich transferu z půdy do rostlin. Účinnost zapravení organických pomocných půdních látek výrazně závisela na kvalitě dodané organické hmoty. Anorganické pomocné látky na bázi sorbentů se osvědčily při imobilizaci mobilních rizikových prvků (Cd, Zn). Účinnost zvolených pomocných půdních látek byla výrazně ovlivněna faktorem půdního typu.

Klíčová slova: rizikové prvky; remediace půd; imobilizace; pomocné půdní látky; chlévský hnůj; zemědělský kompost; kyselá rašelina; slatinná rašelina; syntetický zeolit; cyprisové jílovce; dolomitický vápenec

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