Use of dredged sediments on agricultural soils from viewpoint of potentially toxic substances

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ABSTRACT

A set of 29 pond sediments samples was collected. The sediments were separated into three groups, field, village, and forest pond sediments. The sediment samples were taken from pond bottoms and sediment heaps. The sediment characteristics (pH, CEC, Al-exchangeable, C_{ox}, humus substances), the content of potentially toxic elements and persistent organic pollutants were analysed in the sediments and compared between sediment groups. The comparison of sediment contamination with Czech legislation for sediment use in agriculture was conducted. The village sediments were more loaded by potentially toxic elements and persistent organic pollutants than the others. The pH value of sediments was considered as the properties regulating the retention of potentially toxic elements and their mobility (Al, Cd, Zn). Polycyclic aromatic hydrocarbons (PAHs) were the most problematic POPs group in the sediments. The correlation showed poor dependency of POPs (especially PAHs) content on the content and quality of sediment organic matter.

Keywords: dredged pond sediments; soil hygiene; risky elements; persistent organic pollutants

The volumes of dredged river and pond sediments reach huge amounts because of the periodical maintenance necessity of river channels and water reservoirs. The existence of 97 million m^3 of ponds sediments and 5 million m^3 of river and irrigation channel sediments was reported in the Czech Republic by Gergel (1995). The problem of the disposal or suitable use of dredged sediments of these amounts is evident. There are no united legislative regulations of sediment use and application on agricultural soils in EU member countries available. Many activities are done in SedNet (Sediment European Network) and the implementation of legislative norms is one of the tasks of this working group (www.sednet.org). The Guidance on Chemical Monitoring of Sediment and Biota under the Water Framework Directive 2008/105/EC can be presented as the issue of the working group. The process of soil water erosion influencing sediment amounts was observed on 50% of soil found in the Czech Republic (Toman 1996). The accumulation of nutrients and organic matter especially in pond and downstream sediments belongs to the positives of sediment application. Additionally it must be accepted that eroded soil particles are under the influence of many factors in water environment resulting in the changeover of their quality especially from the viewpoint of elements and substances sorption (Fuentes et al. 2008). The sediments are known as the 'chemical time bomb' thanks to their function of final deposits of pollutants in the river basins (Hilschnerová et al. 2007). The sediment load by potentially toxic substances is connected with the presence of polluting sources like industrial or urban zones or waste outputs from mining activities. The entrainment of potentially toxic elements into sediments from geochemical anomalous substrates or from other natural sources, respectively, can play an important role (Liu et al. 2008). The increased load of potentially toxic substances leads to the difficulties of sediment use in the same way as the

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use of sewage sludge and other organically rich materials (Hanč et al. 2007).

The potential contamination of the sediments by a wide spectrum of hazardous substances cannot be eliminated. The fluvisols developed on alluvial sediments in river fluvial zones belong to the most loaded soils (Podlešáková et al. 1994, Holoubek et al. 1998, Vácha et al. 2003, Urbaniak et al. 2009). German researchers observed increasing water quality in Labe River after the collapse of the communist regime in central Europe thanks to an increasing number of wastewater plants and other modern pollution-controlling technologies (Netzband et al. 2002). Despite this fact; the concentrations of several contaminants are still remaining in sediments of Labe River and their use for agriculture is questionable (Heininger et al. 2003). The most problematic are the contents of Cd, Hg, As, Zn, hexachlor benzene (HCB), polychlorinated biphenyls (PCBs), and polychlorinated dibenzo-p-dioxins and furans (PCDDs/Fs) in the sediments (Heise et al. 2005).

On the other hand, monitoring of pond sediments load by potentially toxic elements in the Czech Republic confirmed a relatively low contamination level (Benešová and Gergel 2003). The database of sediment load by potentially toxic elements and some persistent organic pollutants (POPs) is available in the Central Institute for Supervising of Testing of Czech Republic (Čermák et al. 2009). The results of this monitoring show only sporadically increased values of potentially toxic elements (Cd and Zn usually) in the sediments but these load levels can reach extremely increased values namely in village ponds (1660 mg/kg for Cd or 1630 mg/kg for Zn in some cases).

Long-term prepared legislative regulation (Directive No. 257/2009 Sb.) for sediment application on agricultural soils is valid in the Czech Republic since 2009. The Directive regulates selected characteristics and conditions for the application of dredged sediments. The directive limits contents of potentially toxic elements (As, Be, Cd, Co, Cr, Cu, Hg, Ni, Pb, V, and Zn) and persistent organic pollutants (BTEX, sum of PAHs, PCB_7, sum of DDT and C_{10}–C_{40} hydrocarbons) in the sediment and soil. The limits in the Directive are defined for pseudo total contents (extract of aqua regia) of potentially toxic elements only.

The paper shows the results of the research into the quantitative analysis of potentially toxic substances in a set of sediment samples collected in 2008. The results are compared with the sediment characteristics depending on the sediment origin and the method of sediment processing.

MATERIAL AND METHODS

Sediment sampling. The pond sediment samples from 29 locations were collected in 2008. The samples from pond bottoms and from sediment heaps were used. Field ponds, village ponds and forest ponds were observed. Probe poles with a length of 50 cm for the sampling of bottom sediments and 100 cm for the sampling of heap sediments were used. The individual samples consist of 10 partial samples. The samples were stored in plastic bags and closed jars (for POPs analysis). Closed jars were stored in a deep-freeze conditions before chemical analysis. The summary of collected samples is presented (Table 1).

Sediment analysis. The following characteristics were analysed in sediment samples by the Research Institute for Soil and Water Conservation (RISWC):

- Dry matter content (%)
- Organic matter content (%) – 550°, ČSN EN 12879, 2001
- pH (H_2O), pH (KCl) – ČSN ISO 10390, 1996
- Indicators of the cation exchange capacity CEC (DIN ISO 13536), BS – the rate of complex saturation adsorption (%)
- Al-exchangeable – titration method (Hraško et al. 1962)

The content and quality of primary organic matter and humus substances were analysed in RISWC using the following approach:

C_{org} – organic carbon indicative of the carbon content in primary soil organic matter (SOM). The determination procedure is based on the chromic acid oxidation of organic carbon under the abundance of sulphuric acid and at elevated temperature. Unexpended chromic acid is determined by the iodometric method. This method is a modification of ČSN ISO 14235. The assay of loosely and tightly bound humus materials include the determination of the humic acid carbon (C-HA), fulvic acid carbon (C-FA), humus matter carbon (C-FA + C-HA) and the assessment of the colour coefficient (Q4/6) indicating the humus quality. The determination procedure is based on the sample extraction method using a mixed solution of so-

Table 1. The numbers and types of sediment samples

	Field ponds	Forest ponds	Village ponds	Total
Bottom	6	4	3	13
Heap	7	7	2	16
Total	13	11	5	29

dium diphosphate and sodium hydroxide (Zbíral et al. 2004). Carbon contents (C-FA, C-HA) are determined by titration and the coefficient Q4/6 results from the photometry.

C_{ws} – water-soluble carbon, indicating the quality of primary SOM (bioavailable carbon for soil microorganism). Laboratory determination consists of an hour sample extraction using 0.01 mol/L $CaCl_2$ solution (1:5 w/v) and the determination of oxidizable carbon in the filtrate evaporation residue by heating the filtrate with chromium sulphuric acid and subsequent titration with Mohr's salt.

C_{hws} – hot water-soluble carbon, being similar for the assessment purpose to water-soluble carbon. After the soil sample was boiled for 1 h in 0.01 mol/L $CaCl_2$ solution (1:5 w/v), the oxidizable carbon in the filtrate evaporation residue through the heating of filtrate with chromium sulphuric acid and subsequent titration with Mohr's salt was determined.

The contents of potentially toxic elements were analysed in sediment samples in RISWC:

As, Cd, Co, Cr, Cu, Hg, Ni, Pb, and Zn in the extract of Aqua regia (ČSN EN 13346), Hg was analysed by AMA 254 method (Advanced mercury analyser, total content).

As, Cd, Cu, Pb, and Zn in the extract of 1 mol/L NH_4NO_3 (mobile contents). The samples were prepared according to ISO 11464.

The analysis of the elements in the samples were conducted by the AAS method (AAS Varian), flame and hydride technique.

Persistent organic pollutants were analysed in commercial accredited laboratories Aquatest a.s.:

BTEX (benzene, toluene, e-benzene, and xylene), gas chromatography with mass spectrometry (GS/MS), EPA Method 8260 B.

PAHs – polycyclic aromatic hydrocarbons, the contents of 16 substances following EPA, liquid chromatography with fluorescence detector (HPLC), methodology TNV 75 8055.

PCB_7 – polychlorinated biphenyls, seven indicator congeners (28, 52, 101, 118, 138, 153, 180), gas chromatography with ECD detector (GC/ECD), EPA Method 8082.

DDT sum – sum of DDT, DDE, and DDD, gas chromatography with ECD detector (GC/ECD), EPA Method 8082.

C_{10}–C_{40} hydrocarbons, gas chromatography with flame-ionisation detector (GC/FID), ČSN EN 14039.

The evaluation of sediment characteristics and the contents of potentially toxic elements and persistent organic pollutants in the sediments separating on the basis of their origin and type were done by the use of elementary statistics where median, maximum, minimum, average, standard deviation are presented (Excel). The correlations (Pearson correlation coefficients) between selected sediment properties (pH, CEC, content and quality of soil organic matter) significant at the 0.01 and 0.05 level were processed (SPSS Statistics 17.0).

RESULTS AND DISCUSSION

The basic physico-chemical properties of dredged sediments are presented in Table 2. The wide range of values of observed parameters is clearly visible. The differences between individual sediment groups can be observed when the separation of sediments with respect to their origin (the sediments of field, forest and village pounds) is carried out. The differences between sediment pH were detected primarily. Forest sediments are characterised by higher acidity than the others. The lower values of the BS – the rate of complex saturation adsorption (%) consecutively display an increase in sediment acidity.

The sediments were separated based on the sediment storage method (bottom, heap) due to the tendency to increase acidity during storage,

Table 2. Sediment characteristics in the set of 29 samples

	Dry matter	Organic matter	pH_{H_2O}	pH_{KCl}	Exchangeable H^+	CEC	BS (%)
	(%)				$(mmol_{(+)}/kg)$		
Field (medians)	85.73	8.40	5.22	5.04	80	200	52.5
Village (medians)	98.06	7.07	5.42	5.23	65	189	63.5
Forest (medians)	80.08	8.44	4.09	3.82	160	208	48
Whole set (medians)	81.24	8.68	5.13	4.91	110	209	57.0
Maximum	99.06	22.5	7.1	6.95	420	411	100
Minimum	46.05	2.73	2.86	2.84	< 5	88	28

CEC – cation exchange capacity; BS – the rate of complex saturation adsorption

Table 3. The medians of sediment characteristics separated into sediment groups based on sediment type and storage method

	pH_{H2O}	pH_{KCl}	Exchangeable H^+	CEC	BS (%)
			(mmol$_{(+)}$/kg)		
Field-bottom	5.26	5.12	55	180	55.5
Field-heap	5.21	4.94	95	222	52.5
Forest-bottom	6.28	6.13	100	180	71
Forest-heap	3.81	3.63	265	252	31

CEC – cation exchange capacity; BS – the rate of complex saturation adsorption

and the comparison of the acidity of separated sediments and adsorption characteristics were observed. The prevailing separate sources (field, village and forest) were also accepted but village sediments were not calculated using this procedure due to missing data (only 1 sample of heap sediment was from a village pond). The results are presented in Table 3.

The results confirm the trend of sediment acidification during sediment storage in the category of both sediment groups (field, forest). The forest sediments show sharper differences between the reaction of bottom and heap sediments. It was surprising to see, however, that the bottom forest sediments reached the highest pH value. The results demonstrate that decreasing pH value influences the values of adsorption characteristics markedly (S and V values).

The pH value influences the content of Al-exchangeable in sediments (Figure 1). The dependency trend has exponential character and content of Al-exchangeable increases sharply under pH 4 value. The symptoms of phytotoxicity (Borůvka et al. 2009) were observed in the stored forest sediment with maximal Al-exchangeable content (72.8 mmol$_{(+)}$/kg, equivalent 654.6 mg/kg) where plant cover was missing. The acidity control of dredged sediments and eventual liming for application on agricultural soils seems to be very important.

The values of content and quality of sediment organic matter are presented in Table 4.

The wide range of observed organic matter content in the set of sediment samples was described. The quality of primary organic matter when compared by water-soluble and hot-water soluble carbon contents (C_{ws} and C_{hws} values) reached the highest values in forest pond sediments (199 and 558 mg/kg) followed by field pond sediments. The lowest values in these parameters were observed in village pond sediments (109 and 324 mg/kg). The same order can be observed by the evaluation of

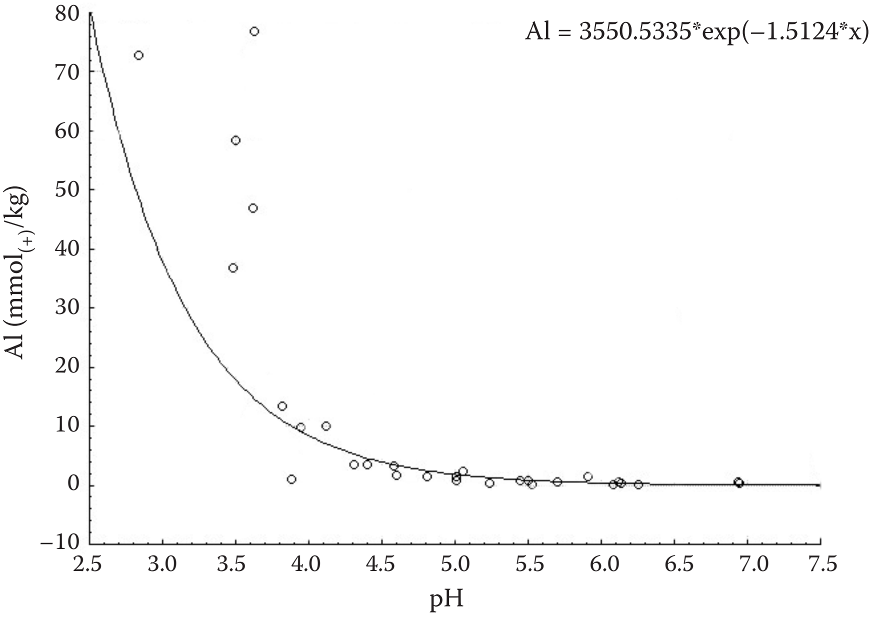

Figure 1. The dependency of Al-exchangeable contents and pH values in the set of sediment samples

Table 4. The contents of risky elements in field, village and forest sediments (mg/kg) and quantities exceeding the limit of the Direction No. 257/2009 Coll

	As	Cd	Co	Cr	Cu	Hg*	Ni	Pb	Zn
Field (maximum)	29.37	0.78	18.22	131	65.6	0.273	46.8	57.2	308.3
Field (minimum)	2.79	0.12	5.45	18.7	8.9	0.021	11.6	9.6	39.2
Field (medians)	9.68	0.22	11.58	42.95	24.4	0.065	28.6	18.0	80.95
Village (maximum)	76.49	0.98	18.22	145.5	50.4	0.131	68	774.5	410.4
Village (minimum)	2.79	0.16	10.71	18.7	31.9	0.025	11.6	< 5.0	46.4
Village (medians)	18.16	0.34	14.86	62.55	39.75	0.057	39.4	24.45	144.9
Forest (maximum)	28.03	0.56	26.08	102.2	54.5	0.436	113.6	78.5	169
Forest (minimum)	7.61	0.1	4.87	14.8	10.8	0.034	10.6	7.1	26.6
Forest (medians)	9.19	0.23	8.92	36.8	24.3	0.093	30.7	32.7	52.2
Limit 257/2009	30	1	30	200	100	0.8	80	100	300
Numbers of excesions Field/Village/Forest	0/1/0	0/0/0	0/0/0	0/0/0	0/0/0	0/0/0	0/0/1	0/1/0	1/1/0

The elements analysed in aqua regia extract; *analysed by AMA method

the content of humus substances where the rise of carbon content of total humus substances in forest pond sediments (0.97%) is distinctly increased in comparison with village sediments (0.47%) The quality of humus substances (compared with the ratio of the carbon of humic and fulvic acids) was higher in the field pond sediments compared with forest pond sediments. The lowest values of humus substances quality were observed in village pond sediments.

The load of sediments by potentially toxic elements (RE) is shown in Table 4 where the maximum values of RE in the extract of aqua regia (pseudo total contents), the limits of RE in the Direction No. 257/2009 Sb. and the numbers exceeding limits are presented.

The limits of Direction No. 257/2009 were exceeded in a total of 5 samples in the case of As, Ni, Pb, and Zn. The level by which the limit was exceeded is relatively high regarding As (2.55×) and Pb (7.75×).

The mobile fraction of As, Cd, Cu, Pb, and Zn in the extract of 1 mol/L NH_4NO_3 was measured and compared with total contents of potentially toxic elements in the aqua regia extract (Table 5). It must be accepted that the application of maximum possible sediment dose (750 t of dry matter/ha following the Direction 257/2009 Sb. for sediment use in agriculture) into the soil when the sediment could reach the weight of one third of soil humus horizon weight can significantly influence the content of potentially toxic element mobile fraction in the soil.

The data confirmed a relatively increased risk of village sediment use where high content of mobile Pb (22 mg/kg) is caused not only by increased

Table 5. Maximum mobile fraction contents of risky elements in the extract of 1 mol/L NH_4NO_3 (mg/kg) and the solubility (%) of risky elements in field, village and forest sediments

		As	Cd	Cu	Pb	Zn
Field	NH_4NO_3	0.044	0.108	0.66	0.75	13.28
	solubility	0.15	13.85	1.01	1.31	4.31
Village	NH_4NO_3	0.054	0.102	0.66	21.99	9.74
	solubility	0.07	10.41	1.31	2.84	2.37
Forest	NH_4NO_3	0.01	0.103	0.55	0.91	23.17
	solubility	0.04	18.4	1.01	1.16	13.71
Soil limit proposal		1	0.1	1	1.5	20

NH_4NO_3 – the content of risky elements in extract of 1 mol/L NH_4NO_3 (DIN ISO 19730); solubility – content in 1 mol/L NH_4NO_3/content in aqua regia × 100 total Pb content (774.5 mg/kg) in the sediment but also by increased Pb solubility (2.84%) which indicates an anthropogenic source of sediment contamination. Relatively increased content of Cd and Zn mobile fraction in forest pond sediment is influenced by decreasing pH (Podlešáková et al. 2001). The contents of mobile Cd slightly exceed the proposal of mobile Cd content limit in agricultural soils of the Czech Republic (Vácha et al. 2010). The As solubility is generally low (Tlustoš et al. 2004) and therefore exceeded total content of As in the sediment only slightly increased As mobile fractions. Nevertheless, the risk is regarding total As contents due to human health risk following from inhalation and dermatological As intake from diffuse dust particles (Saha 2003).

The dependency of total and mobile potentially toxic elements contents on observed sediment properties was calculated by the use of Pearson's correlation coefficients. The data showed that only sediment pH value influenced the contents of mobile fraction of Cd ($r = 0.79$) > Cu ($r = 0.59$), Zn ($r = 0.54$).

The other observed parameters had no relevance to the total and mobile contents of potentially toxic elements on the statistical levels in the set of sediment samples. It could be concluded that sediment pH is the property regulating the retention of potentially toxic elements in the sediments and the process of sediment acidification during sediment storage on the heap can change the contents of potentially toxic elements mobile fractions.

The medians and maximums of POPs contents in field, village and forest sediments are presented in Table 6 where the comparison with the Direction No. 257/2009 Sb. is also available.

The median values of PAHs indicate an increased load of village pond sediments and a similar trend can be found in the case of DDT. The contents of the other POPs are comparable between individual sediment types. The maximum limits of PAHs were exceeded in all three sediment types. PAHs will be the most problematic of the observed POPs group in the sediments. The tendency of the substances to accumulate in the sediments was observed in the order benzo(ghi)perylene > benzo(b)fluoranthene, benzo(k)fluoranthene, pyrene > benzo(a)pyrene, benzo(a)anthracene, fluoranthene and chrysene. Despite of the findings of increased DDT contents in Czech agricultural soils (Vácha et al. 2003, Čupr et al. 2010) no DDT limit exceeding in sediment samples was observed. The existence of the limit for BTEX in the sediments in Direction No. 257/2009 Sb. must be supported with more data collected, especially from river sediments. The limit for C_{10}–C_{40} hydrocarbons will eliminate their increased contents in sediments for agricultural use from local leaks of petroleum hydrocarbons.

The correlation between the contents of observed POPs groups (except of C_{10}–C_{40} hydrocarbons where a dominant number of values were under detection limit) and content and quality of organic matter was assessed. Only sporadic correlation

Table 6. The contents of POPs in field, village and forest sediments (µg/kg) and quantities exceeding the limit of the Direction No. 257/2009 Coll

	PAHs 2n	PAHs 3–4n	PAHs 5–6n	PAHs sum	PCB_7	DDT	BTEX	C_{10}–C_{40}*
Field (maximum)	210	4762	1290	6143	40.8	14.5	71.5	580
Field (minimum)	21	119	22	162	9.57	6.51	14.8	< 100
Field (medians)	48	494	147	694	15.1	9.19	31.2	100
Village (maximum)	210	6842	2133	9052	36.9	32.3	43.2	110
Village (minimum)	62	828	209	1099	10.5	9.35	17.5	< 100
Village (medians)	77	2396	780	3386	14.2	15	30.35	105
Forest (maximum)	228	10347	2961	13536	1010	16.7	96.2	200
Forest (minimum)	21	144	29	203	5	2.84	9.5	< 100
Forest (medians)	41	326	54	517	15.4	8.83	66.3	100
Limit 257/2009	–	–	–	6000	200	100	400	300
Numbers of excesions Field/Village/Forest	–	–	–	1/2/1	0/0/1	0/0/0	0/0/0	1/0/0

*C_{10}–C_{40} content (mg/kg); PAHs 3–4n – the sum of 3 and 4 rings substances; PAHs 2n – the sum of 2 rings substances; PAHs 5–6n – the sum of 5 and 6 rings substances

was observed. The trend of PCB and BTEX accumulation in the dependency on content and quality of humus substances was confirmed. The PAHs groups did not show surprisingly any trend (Cave et al. 2010) of accumulation regarding their properties and affinity to organic carbon ($r < 0.24$).

It could be assumed that the sources of the contamination by POPs determined in most POPs groups, except for BTEX, influenced the sediments load stronger than the selected sediment properties in an observed set of sediment samples.

It could be concluded, that the sediments of village ponds belong to the most contaminated in comparison with forest and field ponds. The storage of sediments on the heaps leads to an increase of potentially toxic elements mobility and content of Al-exchangeable via decrease of sediment pH.

Sediment properties (pH, content and quality of organic matter) influence the mobility of potentially toxic elements (mobile elements like Cd and Zn, especially) and content of persistent organic pollutants partially. The load of sediments is determined by sources of contamination primarily.

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