

HEAVY METALS IN FLUVIAL SEDIMENTS OF THE Odra RIVER FLOOD PLAINS - INTRODUCTORY RESEARCH

ALEKSANDRA IBRAGIMOW, GRAŻYNA GŁOSIŃSKA

Adam Mickiewicz University, Collegium Polonicum Ślubice, Poland

MARCIN SIEPAK

Adam Mickiewicz University, Institute of Geology, Department of Hydrogeology and Water Protection,
Poznań, Poland

BARBARA WALNA

Adam Mickiewicz University, Jeziory Ecological Station, Mosina, Poland

Manuscript received January 4, 2010

Revised version February 15, 2010

IBRAGIMOW A., GŁOSIŃSKA G., SIEPAK M. & WALNA B., 2010. Heavy metals in fluvial sediments of the Odra River flood plains - introductory research. *Questiones Geographicae* 29(1), Adam Mickiewicz University Press, Poznań 2010, pp. 37-47, Figs 6. Tabs 2. ISBN 978-83-232-2136-4. ISSN 0137-477X. DOI: 10.2478/v10117-010-0004-7.

ABSTRACT. The article presents the results of research on concentrations of heavy metals (Cd, Cr, Cu, Ni, Pb and Zn) in fluvial sediment samples collected in the flood plains of the Odra River. The samples were collected from the presently flooded area and from the area which was formerly flooded. The extraction of sediment samples was conducted using aqua regia and four other extractants: 0.01M CaCl₂, 0.1M HCl, 0.005M DTPA and 0.02M EDTA. The analysis of the results revealed different concentration values for particular sample collection sites situated along the course of the Odra River. The differentiation of metal concentrations in the investigated samples depending on the extractants was also observed. The concentrations of metals were determined using the technique of atomic absorption spectrometry with flame atomization (F-AAS).

KEYWORDS: heavy metals, single extraction, fluvial sediments, the Odra River

Aleksandra Ibragimow, Grażyna Głosińska, Adam Mickiewicz University, Collegium Polonicum Ślubice, Kościuszki 1, 69-100 Ślubice, Poland

Marcin Siepak, Adam Mickiewicz University, Institute of Geology, Department of Hydrogeology and Water Protection, Maków Polnych 16, 61-606 Poznań, Poland, e-mail: marcin.siepak@amu.edu.pl

Barbara Walna, Adam Mickiewicz University, Jeziory Ecological Station, PO Box 40, 62-050 Mosina, Poland, e-mail: walna@amu.edu.pl

Introduction

Heavy metals are one of the most important factors which threaten living organisms because, once introduced to the natural environment, they undergo biological accumulation. This concerns especially Cd, Cu, Pb and Zn (Kabata-Pendias & Pendias, 1999). The degree of threat is not determined by the total concentrations of metals but by the forms of metals which may be potentially absorbed by living organisms (Quevauviller, 2002).

Various kinds of pollutants are introduced into the Odra River. This results from dense population and industrialisation of the catchment areas in its upper course. The study of fluvial accumulation of sediments and heavy metals bound with them resulted in emphasizing the significance of flood plains as the place where pollution accumulates (Walling *et al.*, 2003; Owens *et al.*, 1999). The deposited pollutants cause major reduction of the load transported by the river to its mouth (Leece & Pavlowsky, 1997; Miller, 1997; Taylor, 1996; Zhao *et al.*, 1999; Hudson-Edwards *et al.*, 1999). The estimated pollution load for the Odra River amounts to 40-50% (Lewandowski & Przewłocki, 1995).

The mobility of heavy metals compounds depends on their solubility, which is affected by many factors, such as pH, cation exchange capacity of the solid phase, redox potential, grain size composition and moisture. In polluted soils, heavy metals may occur as mobile forms – they are subject to sorption, complexation and coprecipitation with soil particles and, in favourable physico-chemical conditions, they increase the amount of potentially available metals (Lopez-Sanchez *et al.*, 2002; Quevauviller, 2002).

Extraction procedures – single extraction and sequential extraction – are used to determine available forms of heavy metals in sediments and soil. The methods of sequential extraction are based on gradual release of metals occurring in different speciation forms by means of extraction using specific extractants (Tessier *et al.*, 1979; Hulanicki, 1998).

The methods of single extraction involve subjecting a soil sample to one extractant in order to determine forms of metals soluble in extractant solutions selected to reflect the bioavailability of

a given metal for plants or organisms (available forms) and susceptibility of metals to leaching (mainly in highly polluted soil and waste) (Karczewska, 2002). The single extraction is a simple and fast method which provides information concerning a potential environmental hazard. It involves the use of so called ‘soft extractants’, which include non-buffered salt solutions (*e.g.* KCl, CaCl₂, NaNO₃), acid of different concentrations (*e.g.* 0.1M or 1M HCl) and solutions of complexant agents (*e.g.* EDTA, DTPA) (Quevauviller, 2002). In comparison with the sequential extraction, the procedures of single extraction reduce the possibility of mistake at particular stages of extraction (Pijnenburg *et al.*, 2007). Non-buffered salt solutions first and foremost release the cation – exchangeable fraction; diluted acids extract heavy metals of the exchangeable element and carbonate fraction, bound with Fe and Mn oxides and organic matter; complexing agents extract the metals of the exchangeable element fraction and organic matter (Karczewska, 2002).

The study aimed at (1) conducting introductory research on the level of total concentrations of heavy metals (Cd, Cr, Cu, Ni, Pb and Zn) in fluvial sediments of flood plains of the mid – Odra Valley, deposited in the present and former flood area, as well as at (2) determining the easily available forms and (3) defining the extracting effectiveness of the used reagents with reference to the investigated metals. The research is planned to be continued for a bigger number of samples in several cross-sections.

Materials and methods

The sediment samples were collected in April 2009, in the River Odra Valley, at the section from the 564th to 574th km of the river course. They were collected from three cross-sections: Rybocice 1 – R1, Rybocice 2 – R2 and Kunice – K (Fig. 1). In each section, two sediment samples were collected in the distance of about 10 m from the flood bank: from the present flood area (A) and from the former flood area (B). The investigated section of the river belongs to the Nature 2000 Mid-Oder Valley PLB080004 area.

The sediment samples of the Odra flood plains were collected from the surface layer (up

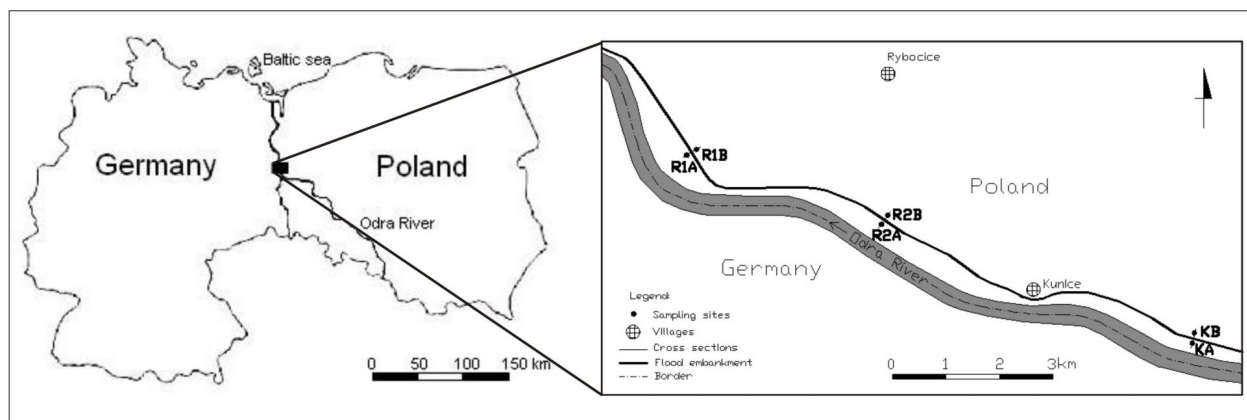


Fig. 1. Study area. R1A, R1B, R2A, R2B, KA, KB – sampling sites

to 20 cm) into polyethylene bags. The samples were dried in the room temperature and sieved with mesh sizes of 2.0 mm.

The samples were then subjected to mineralization with aqua regia according to the Polish Norm (PN-ISO 11466). The single extraction was performed using the following reagents and extraction conditions (Quavaviller, 2002; Karczevska & Kabała, 2008): 0.01M CaCl_2 (1:10 soil/solution, shaking in an end-over shaker for 2h); 0.1M HCl (1:10 soil/solution, shaking in an end-

over shaker for 1h); 0.02M EDTA (1:5 soil/solution, shaking in an end-over shaker for 0.5 h); 0.005M DTPA (1:2 soil/solution, shaking in an end-over shaker for 2h).

Determinations of the total content and available forms of heavy metals were conducted using the technique of atomic absorption spectrometry with acetylene-air flame atomization (F-AAS). The fast sequential atomic absorption spectrometer SpectrAA 280 FS manufactured by Varian (Australia) was used in the determinations. The

TABLE 1. CONDITIONS AND PARAMETERS OF THE ANALYTICAL TECHNIQUE (F-AAS) USED FOR DETERMINATIONS OF Cd, Cr, Cu, Pb, Zn AND Ni.

Parameter		Cd	Cr	Cu	Pb	Zn	Ni
Wavelength	[nm]	228.8	357.9	213.9	217.0	213.9	232.0
Slit width	[nm]	0.5	0.2	1.0	1.0	1.0	0.2
Lamp current	[mA]	4.0	7.0	4.0	10.0	5.0	4.0
Oxid flow	[L min ⁻¹]	12.7	11.5	12.7	12.7	12.7	12.7
Fuel flow	[L min ⁻¹]	2.5	3.1	2.5	2.5	2.5	2.5
Sample flow rate	[mL min ⁻¹]	5.0	5.0	5.0	5.0	5.0	5.0
Flame type	Air/ Acetylene						
Detection limit	[3 σ ; mg kg ⁻¹]	0.01	0.02	0.01	0.03	0.02	0.03
Determination limit	[6 σ ; mg kg ⁻¹]	0.03	0.06	0.03	0.09	0.06	0.09

samples were batched using the autosampler SPS3 manufactured by Varian (Australia). The instrumental parameters and the parameters of analytical method have been presented in Table 1.

The reagents used in determinations were analytically pure and water was deionised to a resistivity of $18.2 \text{ M}\Omega \cdot \text{cm}$ in a Direct-Q[®] 3 Ultrapure Water System apparatus (Millipore, France). Standard solutions were made using Merck commercial standards for AAS (Merck, Darmstadt, Germany).

The accuracy of measurements was determined based on the coefficient of variation (CV), with the assumed acceptance criterion of 15%. Its value was the lowest for 0.1M HCl extraction

(from 3 to 12%). Higher values of CV were obtained for the extraction with complexing agents (from 2 to 17%).

Results and discussion

Total concentration of heavy metals in fluvial sediment samples

The research on the total content of heavy metals aimed at defining the level of pollution of fluvial sediment samples with heavy metals and evaluating the degree of metal extraction using specific extractants. The concentrations of

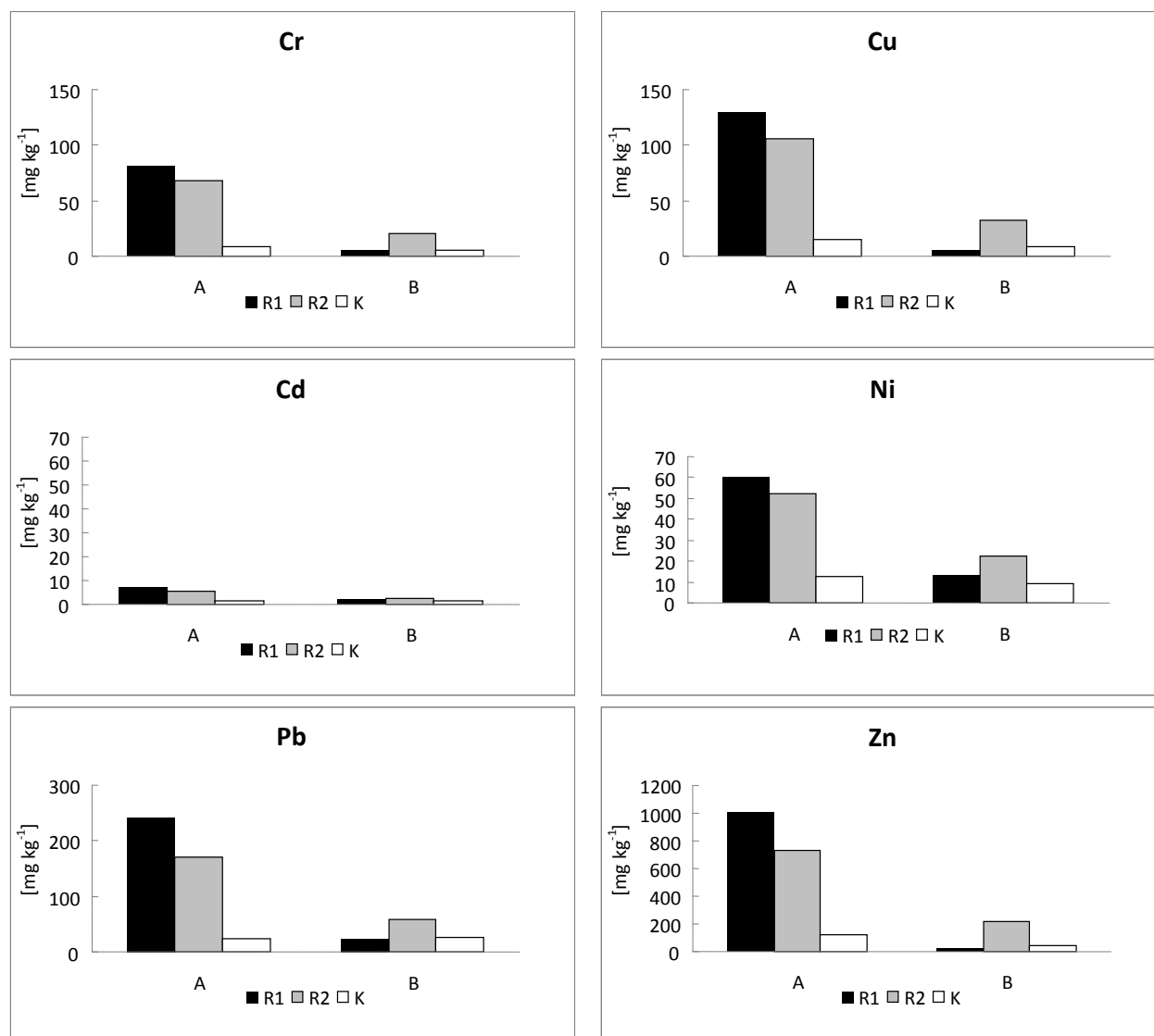


Fig. 2. Total concentrations of heavy metals in fluvial sediment samples.

A – presently flooded area, B – formerly flooded area; Cross-sections: R1 – Rybocice 1, R2 – Rybocice2, K – Kunice

the determined metals amounted (mg kg^{-1}): 1.4 – 7 cadmium, 9.2 – 60 nickel; 5.4 – 81 chromium; 5.7 – 129 copper; 24.5 – 242 lead; 23.6 – 1006 zinc (Fig. 2). With the exception of zinc, these values exceeded the concentrations of metals determined in the previous years (Bojakowska & Sokołowska, 1998), which amounted to (mg kg^{-1}): <0.5 – 6.6 cadmium, 3 – 42 nickel, 5 – 276 copper, 3 – 681 lead, and 39 – 1663 zinc.

Alarmingly high concentrations of cadmium were determined in the investigated area, with the obtained values comparable to the level of pollution observed in the fluvial sediment samples in the River Kilmastulla (Ireland), within the area of impact of former coal mines (Aslibekian & Moles, 2003), as well as in the valleys of the rivers Scheldt and Leie (Vandecasteele *et al.*, 2004). The observed concentrations of lead in the investigated samples are lower than the concentrations of lead in the sediments samples collected in the valleys of the rivers Scheldt and Leie in Belgium (Vandecasteele *et al.*, 2004). Much lower concentrations of zinc and copper occur in the agricultural and fertilized soils of the lower Vistula Valley (Dąbkowska-Naskręt *et al.*, 2000), while similar values of concentrations of these metals were determined in the sediments samples collected in the valleys of rivers Scheldt and Leie (Vandecasteele *et al.*, 2004).

The issue of polluting the alluvium of the River Oder with heavy metals has been addressed by many researchers (Adamiec & Helios-Rybicka, 2002; Boszke *et al.*, 2004; Helios-Rybicka *et al.*, 2005; Głosińska *et al.*, 2005). However, the direct comparison of the studies is difficult as they discussed the concentrations in fractions of either different particle size distribution or obtained by means of different extraction methods.

The presented results of determination of total heavy metals concentrations were compared with the allowable concentrations defined in the Minister of the Environment Directive (2002) in mg kg^{-1} : 1 – cadmium, 35 – nickel, 30 – copper, 50 – lead, 100 – zinc and 50 – chromium. The comparison revealed that the total concentrations exceed the values of allowable concentrations for zinc, lead, copper, chromium, nickel and cadmium. It was also observed that the total concentrations of determined metals in the present flood area were

higher than the concentrations in the former flood area (Fig. 2).

Available forms of heavy metals in fluvial sediment samples

Based on the comparison of the obtained results, it may be stated that the concentrations of extracted metals differ from one another depending on the extractant used, location of the sampling site at the flood bank, as well as on the location of the sampling site along the course of the Oder River.

The concentrations of the investigated metals extracted using 0.01M CaCl_2 amounted to (mg kg^{-1}): 0.53 – 5.61 zinc; 0.39 – 0.91 lead; 0.22 – 0.57 copper; <0.02 – 0.23 chromium; <0.03 – 0.13 nickel; 0.04 – 0.13 cadmium (Fig. 3). The obtained concentrations were compared with the total content and the percentage content of the extracted metals was calculated. The values obtained for 0.01M CaCl_2 were as follows: 0.2 – 2.3% zinc, 0.2 – 3.5% lead, 0.4 – 7.4% copper, BLD – 1.1% chromium, BLD – 0.9% nickel; 1.7 – 8.6% cadmium (Table 2). The extractant proved least effective in the case of nickel and most effective in the case of cadmium.

As a result of 0.1M HCl extraction, higher concentrations of the investigated metals were determined in comparison with 0.01M CaCl_2 extraction. The concentrations were as follows (mg kg^{-1}): 3.30 – 529 zinc; 3.29 – 33.9 lead; 1.21 – 65.6 copper; 0.24 – 4.92 chromium; 1.97 – 16.7 nickel and 0.33 – 4.98 cadmium (Fig. 4). The use of hydrochloric acid enabled to extract: 1.5 – 72% zinc, 8.0 – 33.6% lead, 3.6 – 61.9% copper, 1.7 – 11% chromium, 11.7 – 33.6% nickel; 18.7 – 88.6% of cadmium (Table 2). The extractant proved least effective in the case of chromium and most effective in the case of cadmium.

The concentrations of the investigated heavy metals determined after 0.005M DTPA extractions were as follows (mg kg^{-1}): 0.54 – 227 zinc; 0.63 – 18.9 lead; 0.40 – 53.7 copper; 0.23 – 4.93 nickel and 0.08 – 2.24 cadmium. In the case of 0.005M DTPA extraction, chromium was not determined (the concentration below the limit of analytical technique determination) (Fig. 5), whereas the concentrations of the remaining metals were lower

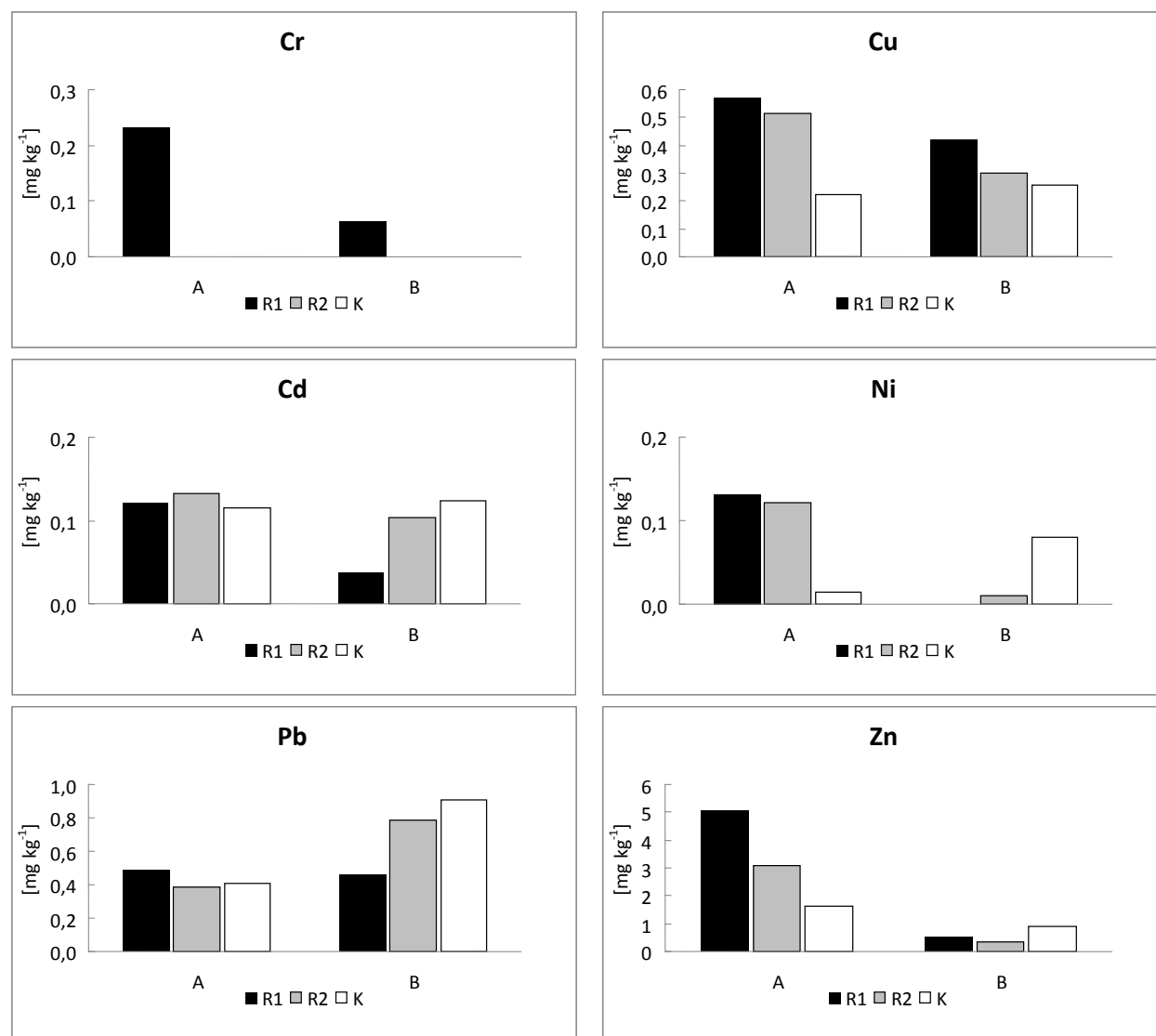


Fig. 3. Concentrations of CaCl₂ - extractable metals.

A - presently flooded area, B - formerly flooded area; Cross-sections: R1 - Rybocice 1, R2 - Rybocice2, K - Kunice

in comparison with 0.1M HCl extraction. The concentrations of the investigated heavy metals determined after 0.02M EDTA extractions were as follows (mg kg⁻¹): 1.43 - 378 zinc; 3.39 - 92.0 lead; 1.29 - 81.6 copper; 1.36 - 16.2 nickel and 0.30 - 3.66 cadmium. The use of two complexing extractants enabled to draw the conclusion that the concentrations determined as a result of 0.02M EDTA extraction (Fig. 6) were at a similar level as or higher than those obtained in 0.005M DTPA extraction (Table 2).

The percentage amount of the released metal in comparison with the total value indicated that 0.02M EDTA released larger quantities of metals compared to 0.005M DTPA. Complexing solutions

proved least effective in the case of chromium and most effective in the case of copper (0.005M DTPA - Fig. 5) and cadmium (0.02M EDTA - Fig. 6). The effectiveness of extraction of cadmium from the samples of soils limited by the flood bank in the Rybocice 2 cross-section (66%) using 0.02M EDTA solution was comparable with the results of a similar test for the soils of the polluted valley of the River Kilmastulla (Ireland); (Aslibekian & Moles, 2003). In the samples of sediments collected in the present flood area (R1A, R2A - Fig. 1), according to the soil classification based on EDTA extraction results (Aslibekian & Moles, 2003), the threshold values for some metals (0.8 mg kg⁻¹ cadmium, 25 mg kg⁻¹ lead, 20 mg kg⁻¹ zinc) were exceeded: cad-

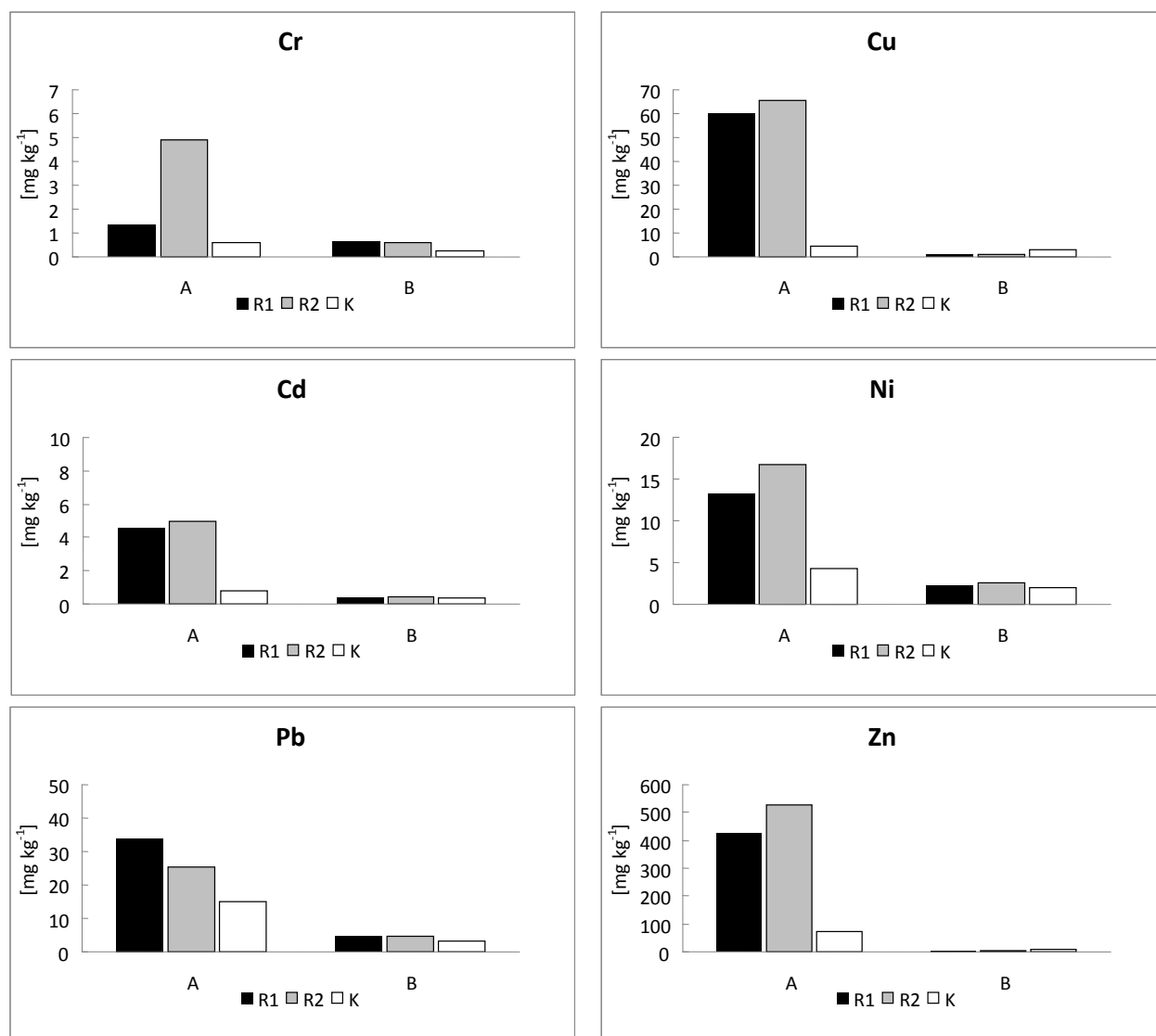


Fig. 4. Concentrations of HCl - extractable metals.

A - presently flooded area, B - formerly flooded area; Cross-sections: R1 - Rybocice 1, R2 - Rybocice2, K - Kunice

mium over four times, lead four times, zinc sixteen times.

Irrespective of the extractant used, chromium was extracted in minimal quantities, which is related to very low availability of this metal, in spite of its quite high total content. The low level of chromium extraction is also confirmed by the studies into the sediments of flood plains of the rivers Rhein and Meza in Holland (Schroeder *et al.*, 2005).

As a result of 0.005M DTPA, 0.02M EDTA and 0.1M HCl extractions, significant differences in concentrations of zinc, lead and copper obtained in the presently and formerly flooded areas were observed. Higher concentrations in the present flood area, in comparison with the concentra-

tions in the former flood area (extraction DTPA, EDTA and HCl), may be linked to the chemical form of the metal. Similar differences in pollution by metals in the areas more and less frequently flooded were also observed in the mid-Vistula Valley (Gašior & Paško, 2007) and in the valleys of the following rivers: Amer, Rhein, Dommel (Bleeker & Van Gestel, 2007; Schipper *et al.*, 2008; Van Gestel, 2008).

The forms of metals released during 0.01M CaCl₂ extraction correspond with slight changes in environmental conditions, and the capacity for 0.01M CaCl₂ extraction is the measure of present availability of metals. The forms which are stronger bound, potentially available, activated in the

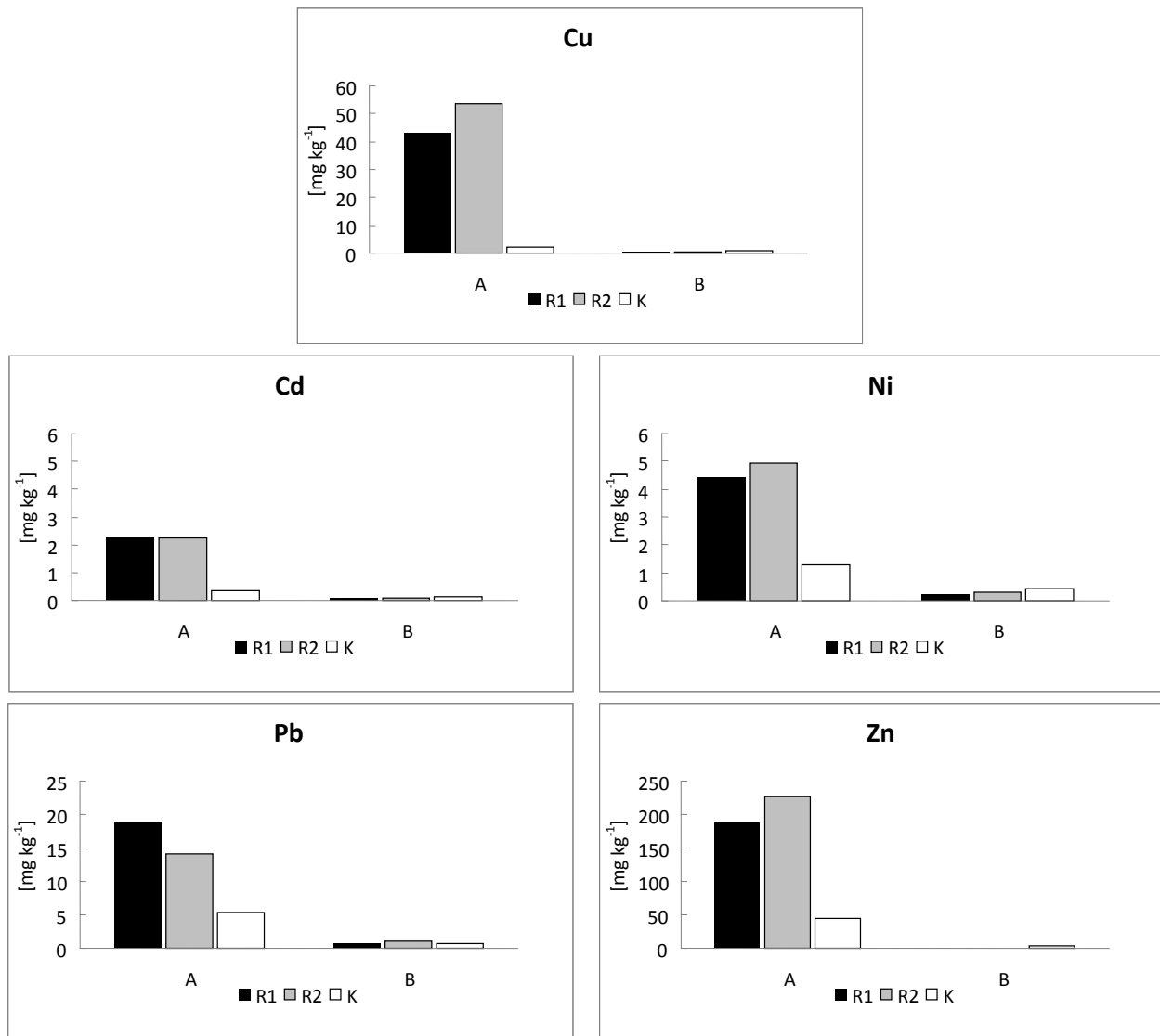


Fig. 5. Concentrations of DTPA - extractable metals.

A - presently flooded area, B - formerly flooded area; Cross-sections: R1 - Rybocice 1, R2 - Rybocice 2, K - Kunice

case of visible changes of environmental parameters, such as pH reaction or redox potential, correspond with the concentrations of metals obtained in the extractions with 0.1M HCl and complexing agents. The alarming fact is that, from among the investigated metals, the most available is cadmium (extracted with 0.01M CaCl₂), its extraction with reference to the percentage content is from 1.7 to 8.6 %. High availability of cadmium is also confirmed in other studies (Bleeker & Van Gestel, 2007).

Despite the introductory nature of the conducted research and small amount of the analysed samples, the influence of the width of the valley on the concentration values of metals deposited

in the sediments (higher concentrations of the determined metals recorded at sites R1A and R2A) may be observed for the results of available metal forms and for total concentrations. The comparison of the total content in samples collected in the present and former flood area reveals the role of the river as the agent carrying significant amounts of heavy metals.

Conclusions

- Introductory research on pollution of sediments of the mid-Oder flood plains with heavy metals, aiming at the determination of available

TABLE 2. THE VALUES (%) OF EXTRACTABLE METALS IN FLUVIAL SEDIMENT SAMPLES

Metals	Samples	Extracting solution			
		0.01M CaCl ₂	0.1M HCl	0.005M DTPA	0.02M EDTA
		[%]			
Zn	R1A	0.5	42.2	18.7	29.8
	R1B	2.2	16.0	2.9	6.8
	R2A	0.4	72.0	30.9	51.4
	R2B	2.6	1.5	0.3	0.7
	KA	1.4	59.9	37.0	49.5
	KB	2.3	24.5	7.3	16.1
Pb	R1A	0.2	14.0	7.8	38.0
	R1B	1.9	18.6	2.5	13.9
	R2A	0.2	15.0	8.3	49.6
	R2B	1.3	8.0	1.8	6.8
	KA	0.9	33.6	12.1	42.0
	KB	3.5	12.7	3.0	13.5
Cu	R1A	0.4	46.4	33.6	55.4
	R1B	7.4	21.2	7.0	22.6
	R2A	0.5	61.9	50.7	77.0
	R2B	0.9	3.5	2.0	4.5
	KA	1.5	28.9	15.2	37.6
	KB	2.9	35.0	12.2	32.4
Cr	R1A	0.3	1.7	BLD	1.0
	R1B	1.1	11.0	BLD	7.9
	R2A	BLD	7.2	BLD	1.3
	R2B	BLD	3.0	BLD	3.2
	KA	BLD	6.8	BLD	3.3
	KB	BLD	4.5	BLD	1.9
Ni	R1A	0.2	22.2	7.4	21.0
	R1B	BLD	18.2	1.8	10.6
	R2A	0.2	32.0	9.5	31.0
	R2B	0.1	11.7	1.4	7.4
	KA	0.1	33.6	10.1	28.1
	KB	0.9	21.5	4.8	16.8
Cd	R1A	1.7	64.7	31.7	52.1
	R1B	2.0	18.7	4.4	16.7
	R2A	2.4	88.6	39.9	65.1
	R2B	4.4	19.3	4.2	14.4
	KA	6.8	47.0	20.0	38.6
	KB	8.6	22.8	7.6	20.7

BLD; below limit of detection

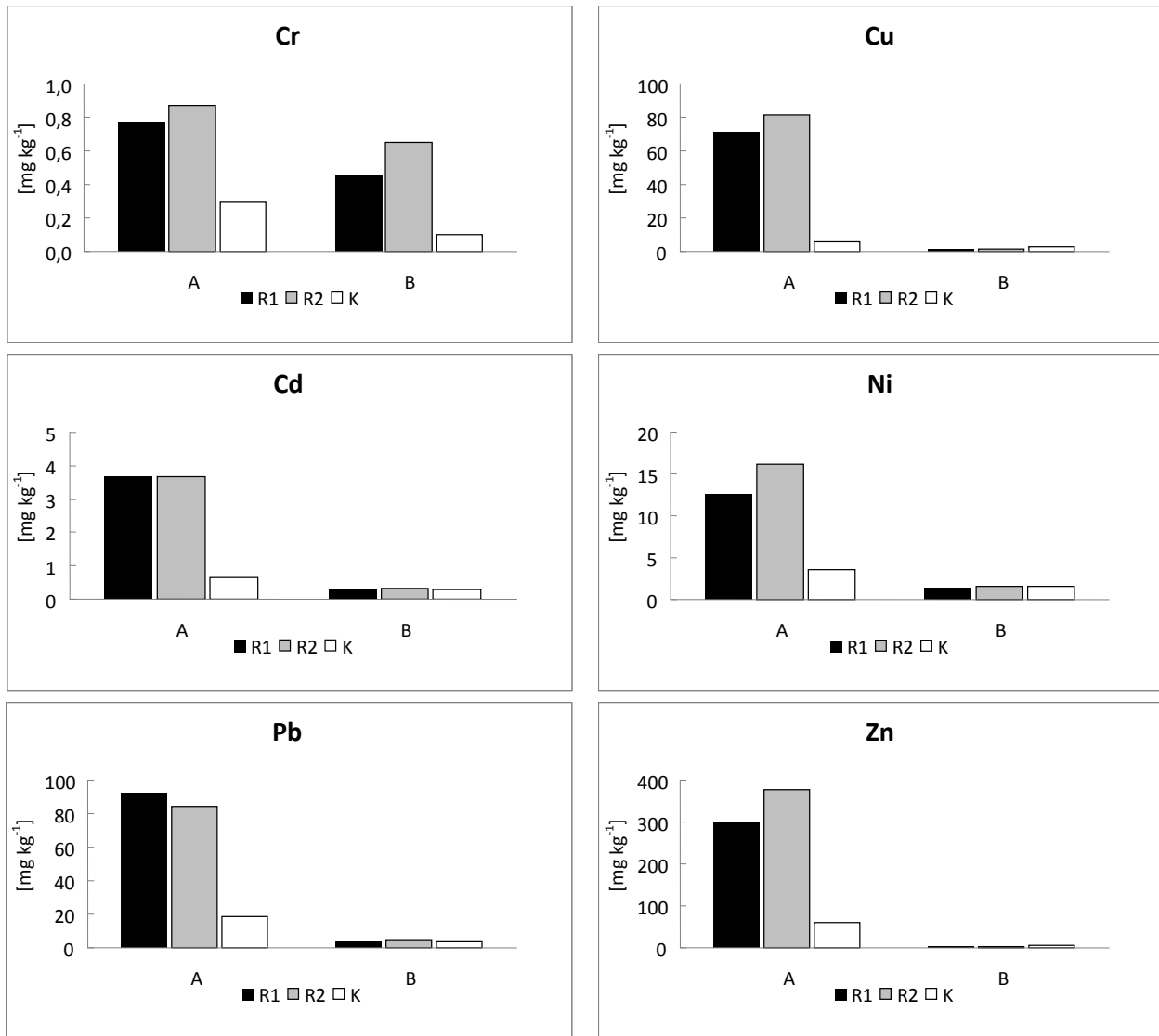


Fig. 6. Concentrations of EDTA extractable metals.

A – presently flooded area, B – formerly flooded area; Cross-sections: R1 – Rybocice 1, R2 – Rybocice 2, K – Kunice

forms concentrations, revealed high concentrations of cadmium, copper, lead and zinc.

- In the case of Cu and Pb extraction, the sequence of the most effective extractant was 0.02M EDTA > 0.1M HCl > 0.005M DTPA > 0.01M CaCl₂, while in the case of Cd, Zn, Ni and Cr, it was 0.1M HCl > 0.02M EDTA > 0.005M DTPA > 0.1M CaCl₂.

- The comparison of the total content in samples collected in the present and former flood areas reveals the role of the river as the agent carrying significant amounts of heavy metals, as well as the influence of retention time of sediments, which affects the processes shaping the form and availability of heavy metals.

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