Biochar application to metal-contaminated soil: Evaluating of Cd, Cu, Pb and Zn sorption behavior using single- and multi-element sorption experiment

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ABSTRACT

The aim of this study was to evaluate metals (Cd, Cu, Pb and Zn) sorption behavior after biochar application into a metal-contaminated soil. Additionally, two different types of biochar originated from the same organic material (contaminated and uncontaminated) at different application rates (1% and 2% w/w) were evaluated as a novelty of the experiment. Batch sorption/desorption experiments were established to compare the sorption behavior of metals originating from single- and multi-element solutions. Zinc as one of the main contaminants in the studied soil was easily desorbed in the presence of Cu, Pb and to a lesser extent by Cd. This desorption was reduced after biochar application. The obtained results proved the different sorption behavior of metals in the single-metal solution compared to the multi-metal ones due to competition effect. Moreover, during multi-element sorption, Zn was significantly desorbed. The applied biochar enhanced Cu and Pb sorption and no changes were observed when contaminated and uncontaminated biochar was used. Furthermore, the application rate (1% and 2% w/w) had no effect as well. In summary, it is needed to point out that the applied rates of biochars were insufficient for metal immobilization in such contaminated soils.

Keywords: biochar; metal retention; isotherms; contamination; soil

Mobile (bio)available metal concentration in contaminated soils can be minimized through biological immobilization and stabilization methods using a range of organic compounds, such as biochar, which is a form of environmental black carbon produced using the pyrolysis of C-based biomass (Verheijen et al. 2010). Incorporation of biochar can influence soil structure, texture, porosity, particle size distribution and density, thereby potentially altering air oxygen content, water storage capacity and microbial and nutritional status of the soil within the plant rooting zone (Amonette and Joseph 2009). Biochar soil additions also influence chemical properties of the soil such as changes in pH, electrical conductivity (EC), cation exchange capacity (CEC), nutrient levels (Gundale and DeLuca 2007, Warnock et al. 2007, Amonette and Joseph 2009) and consequently metal sorption efficiency (Beesley et al. 2010, Uchimiya et al. 2010).

Sorption efficiency as a major process responsible for the fate of metals in soils is usually based on laboratory batch experiments aimed to quantify the distribution coefficient, an operational parameter that relates the amount of metal sorbed in the soil solid phase to the metal concentration in the soil solution at equilibrium (Vidal et al. 2009). In order to describe the sorption behavior of metals onto the soil, several models (empirical/mechanistic), which describe the distribution of the metal between the solid and the liquid phases, were proposed (Bradl 2004). The most commonly used are the Langmuir and the Freundlich isotherms (Hinz 2001, Limousin et al. 2007).

According to Uchimiya et al. (2010) we try to evaluate metal sorption behavior after biochar application to a metal-contaminated soil (Gleyic Fluvisol from the alluvium of the Litavka river in the Příbram mining/smelting district, Czech

Republic) (Vaněk et al. 2008). This site belongs to the most polluted ones in Central Europe due to atmospheric deposition of potentially toxic metals from a Pb smelter that has been operational for over 50 years (Šichorová et al. 2004). Nevertheless, there is still limited information about metal sorption/retention in biochar-amended soil.

The main aim of this study was to evaluate two different types of biochar originated from the same organic material (willows) at different application rates (1% and 2% w/w; according to Novak et al. 2009, Ding et al. 2010), which were grown at two different experimental sites (contaminated vs. uncontaminated) and to evaluate the sorption parameters of such treated soils (involving single- and multi-metal experimental solutions).

MATERIAL AND METHODS

Biochar and soil samples preparation. Stems of willow, which were growing on metal-contaminated and uncontaminated soil, were collected, chipped, air-dried and finally pyrolyzed, using autothermic regulated pyrolyser with oxygen-limited conditions (ÚVP Prague, Czech Republic). Temperature of pyrolysis process was continuously increased in steps 10°C per min to the maximum of 400°C. The resulting biochars were allowed to cool to room temperature overnight.

The experimental soil was collected in the smelting area of Příbram, Czech Republic from the arable layer (0–25 cm). Soil samples were air-dried, sieved through a 2 mm sieve and homogenized.

Biochar and soil characterization. Basic biochar characteristics (Table 1) were established according to (i) standard ČSN 44 1377/1980 (for water content); and (ii) standard CEN/TS 14775 (for ash content); apparent density was established using free filling into 100 mL calibrated divider. Elemental composition (CHNS-O) was determined in two-step elemental analyses using CHNS-O Analyzer Flash 1112. Additionally, elemental composition of the ash was determined using sequential wave dispersed radiographic spectrometer.

Basic soil characteristics (Table 1) such as pH, cation exchange capacity (CEC), available forms of nutrients, content of mineral N, dissolved organic carbon (DOC) and total organic carbon (TOC) were performed according to Trakal et al. (2011 submitted). Analyses of the Cd, Cu, Pb, and Zn total concentration in the soil were determined according to Száková et al. (2009). The certified reference material (RM) 7003 Silty Clay Loam was used for quality assurance of the results. Metals in the digests were analyzed using inductively coupled plasma optical emission spectroscopy ICP-OES (Varian VistaPro, Mulgrave, Australia).

Batch sorption/desorption experiment. According to Komárek et al. (2009) and Trakal et al. (2011 submitted) a desorption experiment was set up to determine concentrations of desorbed metals from the soils. Consequently, 0.01 mol/L KNO_3 was chosen for the sorption experiment. For the equilibrium batch sorption experiment a volume of 20 mL of the solutions was added to 1 g of soil. The experimental single- and multi-element solutions were prepared from metal nitrate salts (Lach-Ner, Czech Republic) at six different concentrations: Cu, Pb and Zn (0.1, 0.5, 1, 2, 4, and 8 mmol) and Cd (0.05, 0.25, 0.5, 1, 2, and 4 mmol), respectively. Each multi-elemental solution was prepared from the same concentrations of the four metals (mixture of Cd 0.05 mmol and Cu, Pb, Zn 0.1 mmol, etc.). According to Boudesocque et al. (2007) suspensions of the solution with the soil

Table 1. Physico-chemical characteristics and total initial metal concentration in the studied soil and biochars

	pH (–)	CEC (mmol/kg)	TOC (%)	DOC (mg/kg)	Available form of nutrients* (mg/kg)				N_{min} (mg/kg)	Total metal content (mmol/kg)			
					Ca	K	Mg	P		Cd	Cu	Pb	Zn
Soil	5.7	134 ± 3	3.72	146	995 ± 68	155 ± 4	125 ± 1	58.8 ± 1	80.5	0.41 ± 0.03	1.22 ± 0.05	12.1 ± 0.5	65.6 ± 1.0

	θ (% w/w)	ρ (g/cm³)	Volatile comp.** (%)	Basic elemental composition (%)					Ash (% w/w)	Elemental composition of the ash (%)									
				C	N	S	O	H		Ca	Cl	K	Mg	Mn	Na	P	S	Si	others***
B	0.036	0.173	3.8	69.2	1.53	0.04	11.8	3.6	7.89	60.3	0.09	24	5.11	0.15	0.47	6.15	2.06	0.55	1.43
B_C	0.029	0.223	4.1	67.5	1.04	0.01	11.7	3.58	8.79	53.6	0.08	17.1	5.28	0.37	0.61	5.42	2.4	6.67	8.88

*mobile portions of nutrients determined in extracts obtained by Mehlich III extraction procedure (Zbíral 2000); **volatile components are to 100°C; ***other elements means (Al, Ba, Cd, Cu, Fe, Pb, Rb, Se, Sr, Ti and Zr)

sample were shaken for 48 h in order to reach equilibrium, centrifuged and obtained supernatants were analyzed using ICP-OES. The sorbed concentrations of metals were calculated according to equation (1):

$$c_{sorb} = c_0 - (c_{desorb} + c_{eq}) \qquad (1)$$

where: c_{sorb} is the sorbed concentration, c_0 is the initial concentration and c_{desorb} is the desorbed concentration and c_{eq} is concentration in the solution. The obtained values of c_0 and c_{sorb} were then used for the construction of Freundlich and Langmuir isotherm, described by equations (2) and (3), respectively:

$$c_{sorb} = K_F c_{eq}{}^n \qquad (2)$$

$$c_{sorb} = \frac{K_F c_{sorbmax} c_{eq}}{1 + K_L c_{eq}} \qquad (3)$$

where: c_{sorb} is sorbed concentration, $c_{sorbmax}$ is the maximum sorption concentration, K_F and K_L are the Freundlich and Langmuir sorption coefficient, is concentration in the solution and n is an empirical coefficient. The model of (Bolster and Hornberger 2007, Bolster 2008) was used to evaluate non-linear isotherm parameters and fitting models efficiencies (E).

RESULTS AND DISCUSSION

Desorption. The results obtained from the experiment showed a low desorption of Cd (3.29 ± 0.35 nmol/g) and Cu (4.48 ± 0.15 nmol/g), a relatively low desorption of Pb (48.0 ± 2.4 nmol/g) and a high desorption of Zn (667 ± 25 nmol/g) in the presence of 0.01 mol/L KNO_3. This finding was due to the initial low Cd and Cu total content and extremely high Zn and Pb total content in the studied soil. Moreover, Pb compared to Zn was reduced after desorption by 0.01 mol/L KNO_3 due to their stronger fixation in the soil (Lair et al. 2007). No significant effect on desorption was observed when biochar was present in the soil, probably due to the rather high initial pH value of the soil (Table 1). This finding was in agreement with negligible pH effect of biochars amended variants (6.95) in comparison with the control (6.88). Additionally, comparison of the contaminated vs. uncontaminated biochar and their application rates showed no significant concentration changes in the desorbed final solution.

The desorption of metals during single-metal sorption experiments was also monitored. Cadmium and Zn were desorbed during Cu and Pb sorption. Copper and Pb desorption was negligible due to their strong fixation onto soil organic matter and their affinity for Mn and Fe (hydr)oxides (Bradl 2004). On the other hand, Cd and Zn desorption was high (mainly in the case of Zn with a 10-times increased concentration in the solution; (Figure 1). In general, Cd and Zn are easily desorbed in the presence of other competitive metals such as Cu and Pb due to their weaker binding in the soil (Markiewicz-Patkowska et al. 2005, Trakal et al. 2011 submitted).

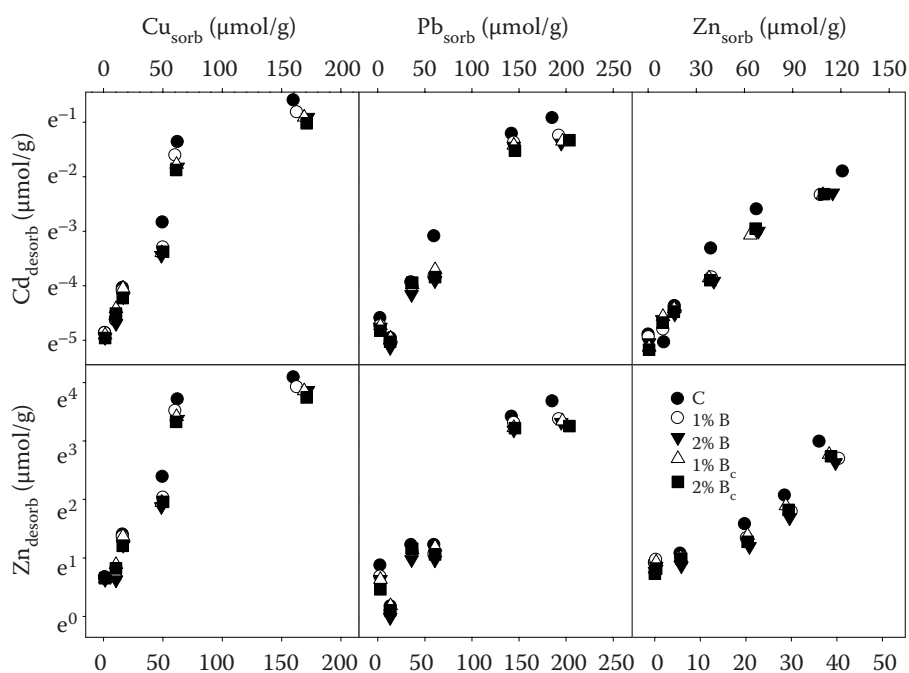Additionally, biochar application resulted in a significant reduction of desorbed Cd and Zn.

Figure 1. Relationship between metal sorption and desorption of Cd and Zn

Table 2. Freundlich and Langmuir parameters and model efficiencies (*E*) obtained for the single- and multi-metal experiments

		E	Freundlich parameters		E	Langmuir parameters	
			K_F	n		K_L	S_{max}
Cd$_{single}$	C	0.94	3.40	0.32	> 0.99	0.007	37.7
	1% B	0.96	3.52	0.33	0.99	0.007	42.2
	2% B	0.95	4.01	0.31	0.99	0.010	39.9
	1% B$_c$	0.95	3.71	0.31	0.99	0.009	38.8
	2% B$_c$	0.95	3.95	0.31	0.99	0.009	39.5
Cu$_{single}$	C	0.93	9.40	0.36	0.87	0.003	178
	1% B	0.95	10.6	0.33	0.86	0.002	196
	2% B	0.96	10.1	0.37	0.89	0.002	204
	1% B$_c$	0.95	10.4	0.36	0.88	0.002	197
	2% B$_c$	0.95	10.7	0.36	0.88	0.003	197
Pb$_{single}$	C	0.80	26.9	0.27	0.89	0.030	190
	1% B	0.81	27.3	0.29	0.90	0.034	200
	2% B	0.80	28.6	0.28	0.89	0.036	202
	1% B$_c$	0.81	28.7	0.29	0.91	0.038	202
	2% B$_c$	0.84	28.3	0.33	0.92	0.037	213
Zn$_{single}$	C	0.98	0.71	0.63	n.a.	n.a.	n.a.
	1% B	0.97	1.19	0.55	n.a.	n.a.	n.a.
	2% B	0.97	1.14	0.56	n.a.	n.a.	n.a.
	1% B$_c$	0.98	0.98	0.57	n.a.	n.a.	n.a.
	2% B$_c$	0.98	0.99	0.57	n.a.	n.a.	n.a.
Cd$_{multi}$	C	0.45	2.05	0.11	0.86	0.15	4.4
	1% B	0.42	2.17	0.10	0.83	0.16	4.5
	2% B	0.50	2.17	0.11	0.89	0.15	4.8
	1% B$_c$	0.46	2.13	0.11	0.87	0.16	4.5
	2% B$_c$	0.38	2.18	0.10	0.82	0.2	4.3
Cu$_{multi}$	C	0.77	10.2	0.18	0.97	0.06	39.6
	1% B	0.84	9.53	0.19	0.98	0.05	41.5
	2% B	0.85	9.79	0.19	0.94	0.07	41.9
	1% B$_c$	0.83	10.5	0.18	0.97	0.08	41.9
	2% B$_c$	0.85	9.91	0.18	0.98	0.06	39.9
Pb$_{multi}$	C	0.90	6.45	0.28	0.9	0.03	50.1
	1% B	0.89	6.23	0.29	0.85	0.03	52.0
	2% B	0.91	7.04	0.28	0.88	0.03	53.7
	1% B$_c$	0.90	6.86	0.28	0.88	0.03	52.1
	2% B$_c$	0.88	6.94	0.27	0.86	0.04	49.6
Zn$_{multi}$	C	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	1% B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	2% B	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	1% B$_c$	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	2% B$_c$	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.

n.a. – results not available

This finding is in agreement with Beesley and Marmiroli (2011), who declared that biochar was an effective amendment for reducing high concentrations of soluble Cd and Zn in contaminated soils. Furthermore, contaminated biochar and a lower rate of applied biochar showed no significant increase of desorbed Cd and Zn concentrations.

Sorption. The sorption of metals in soils was evaluated from single- and multi-element metal solutions as a function of their initial concentrations (Vidal et al. 2009). For all experiments, Freundlich and Langmuir isotherms were used and compared. Table 2 shows that for described single-element sorption fitting, Langmuir isotherms were more suitable. However, the mathematical model was not able to determine isotherms parameters for Zn.

A significant Zn desorption occurred during the multi-element experiment; hence no evaluation of sorption efficiency was suitable. Additionally, the calculation of saturation indices using PHREEQC-2 software showed that $Cu(OH)_2$ and $Pb(OH)_2$ were close to equilibrium and some precipitation may have occurred. However, the final pH of the experiment solution (Figure 4) after the contact with the soil most probably maintained the metals solubilized.

Evaluation of pH during the sorption experiment. Soil pH decreases at increased metal concentrations during single- and multi-element sorption can be attributed to the hydrolysis effect and release of H^+ from soil sorption sites (Ponizovsky et al. 2007, Mouta et al. 2008). As adverted in Figure 4,
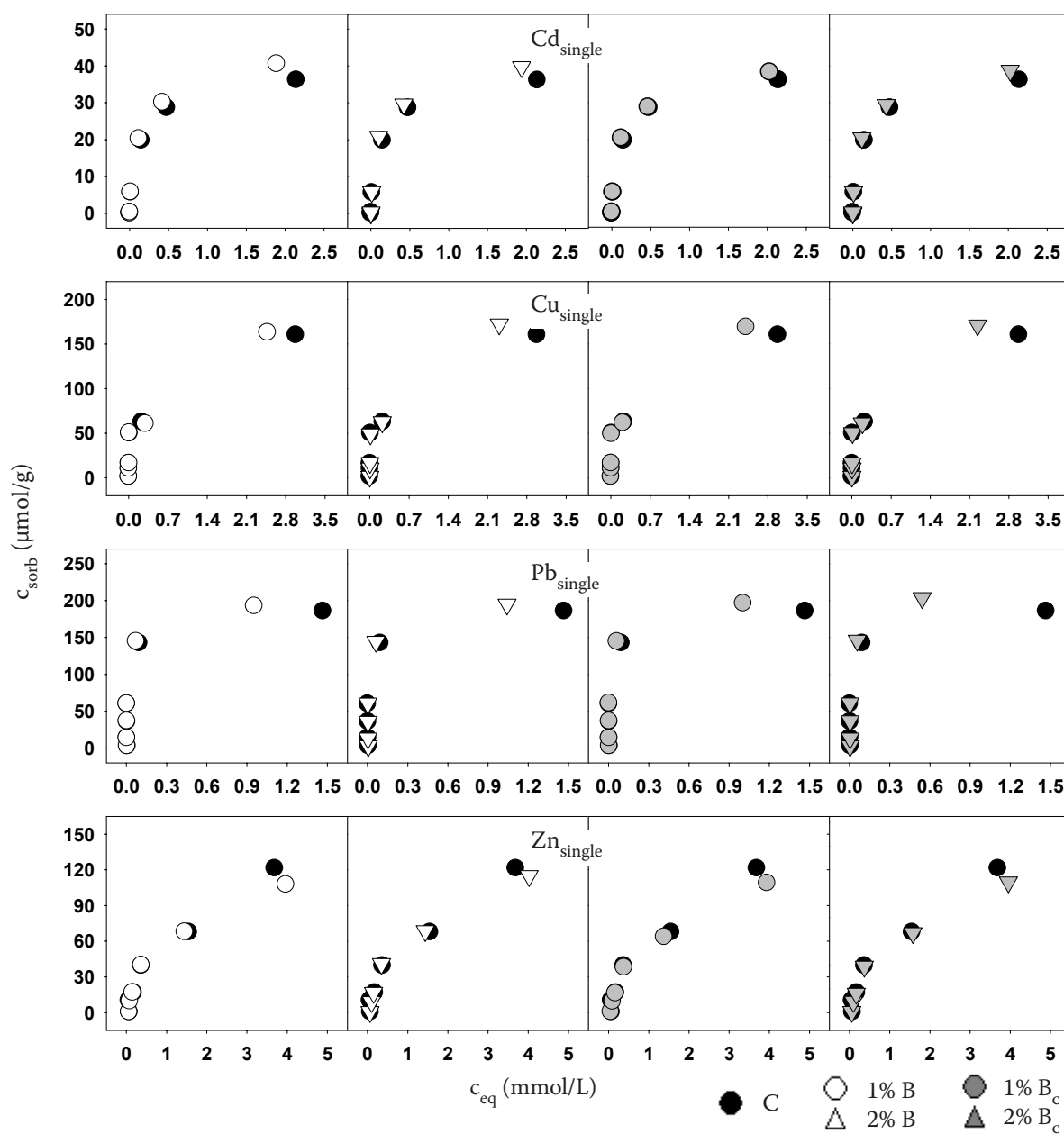

Figure 2. Sorption isotherms of the studied metals (single-metal sorption); c_{sorb} – sorbed metal at equilibrium; c_{eq} – final solute metal concentration at equilibrium

pH decrease during single-element sorption at equilibrium was the most pronounced for Cu and followed the order Pb > Zn > Cd. Additionally, the final pH of multi-metal solutions decreased from 6.8 (lowest metals concentration) to 4.7 (highest metals concentration) with the major influence of Cu and Pb.

Single- and multi-metal sorption. The results from the single-element sorption confirmed the well-known fact that soil sorption decreased in the order Pb > Cu > Zn > Cd, which was previously reported by many authors (Sauvé et al. 2000, Sastre et al. 2007, Vidal et al. 2009). We also confirmed that sorption efficiency of studied metals was reduced under competitive conditions represented

in multi-metal experiments. This finding was also presented before by (Markiewicz-Patkowska et al. 2005, Vidal et al. 2009, Trakal et al. 2011 submitted). Sorption efficiency of Cd and Zn as the target metals was mostly affected by the presence of Cu and Pb as competitive metals (Vidal et al. 2009). The maximum sorption concentration of Cd was 5-times lower in the presence of other competitive metals, whereas Zn was desorbed during multi-metal sorption probably due to (i) extremely high initial Zn content in the soil and (ii) its high desorption efficiency caused by other competitive metals (Cu and Pb; Figure 2, 3). Moreover, multi-metal sorption efficiency was affected also in the case of Cu and Pb despite their generally
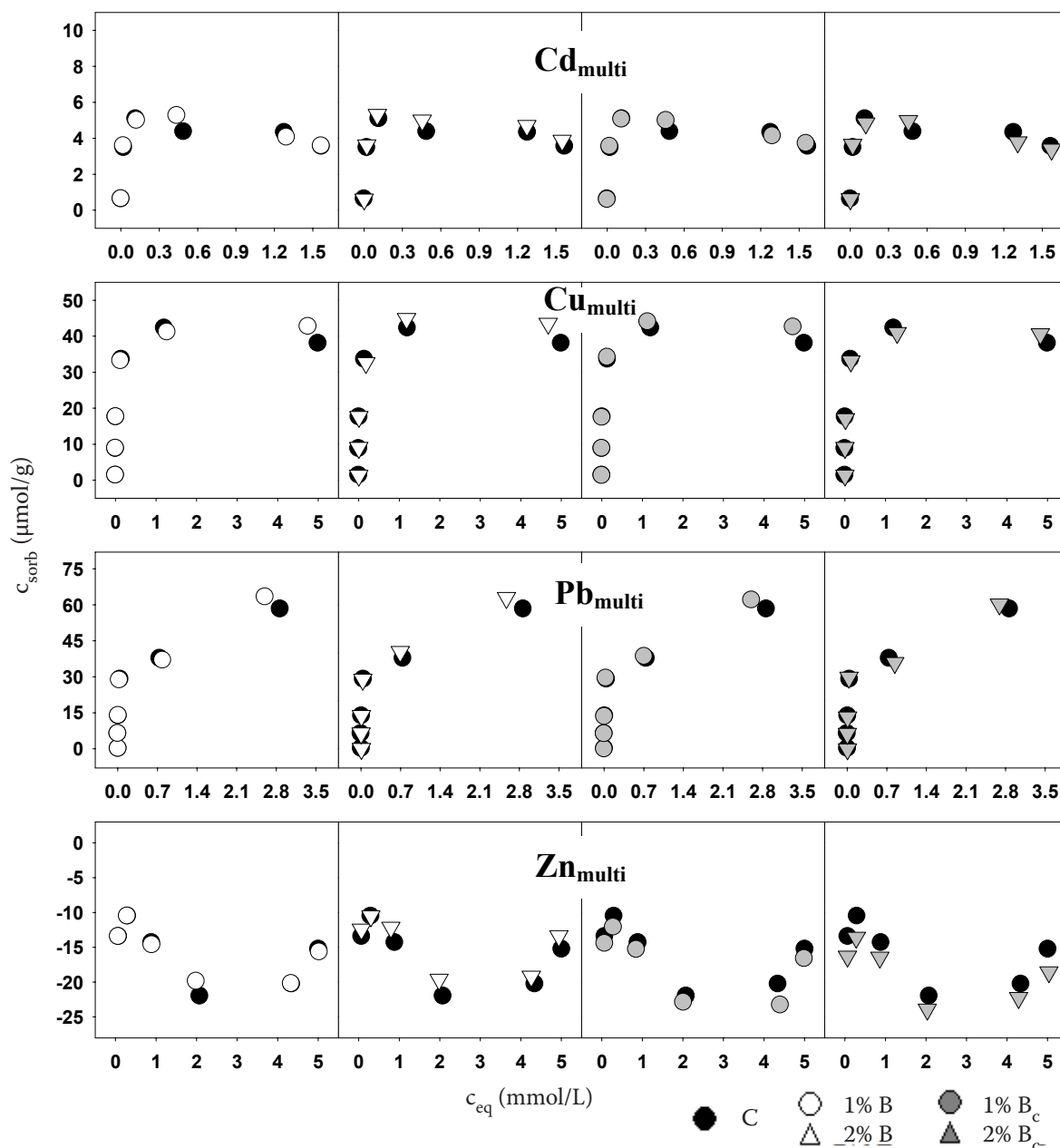

Figure 3. Sorption isotherms of the studied metals (multi-metal sorption); c_{sorb} – sorbed metal at equilibrium; c_{eq} – final solute metal concentration at equilibrium

high ability to sorb in the soil (Bradl 2004). This finding was probably caused by the low sorption capacity of the studied soil (low CEC; Table 1), high metal contents in the soil and due to their mutual competition during multi-element sorption (Vidal et al. 2009).

Single- and multi-metal sorption in biochar amended soil. The addition of biochar (both contaminated and uncontaminated to the soil) increased the sorption efficiency of Cu and Pb in single- and multi-metal experiments (Figures 2 and 3), due to addition of the organic matter (in biochar form) into the soil. Results of Cu sorption are in agreement with (Uchimiya et al. 2011a,b). On the other hand, biochar amendment showed

no enhanced sorption of Cd and Zn as opposed to (Beesley and Marmiroli 2011), most probably due to (i) their lower sorption compared to Cu and Pb; (ii) Cd, Zn predominant fixation onto the Mn, Fe hydr(oxides) and clay minerals; and additionally due to (iii) high Zn content in the soil.

A novel approach was to compare contaminated and uncontaminated biochar. Contaminated biochar had no additional negative effect on metal sorption most probably due to the strong fixation of metals on the biochar surface (Beesley et al. 2010, Uchimiya et al. 2010c). Additionally, a comparison of two different rates of applied biochars also showed no significant differences during single- and multi-metal sorption. It is also
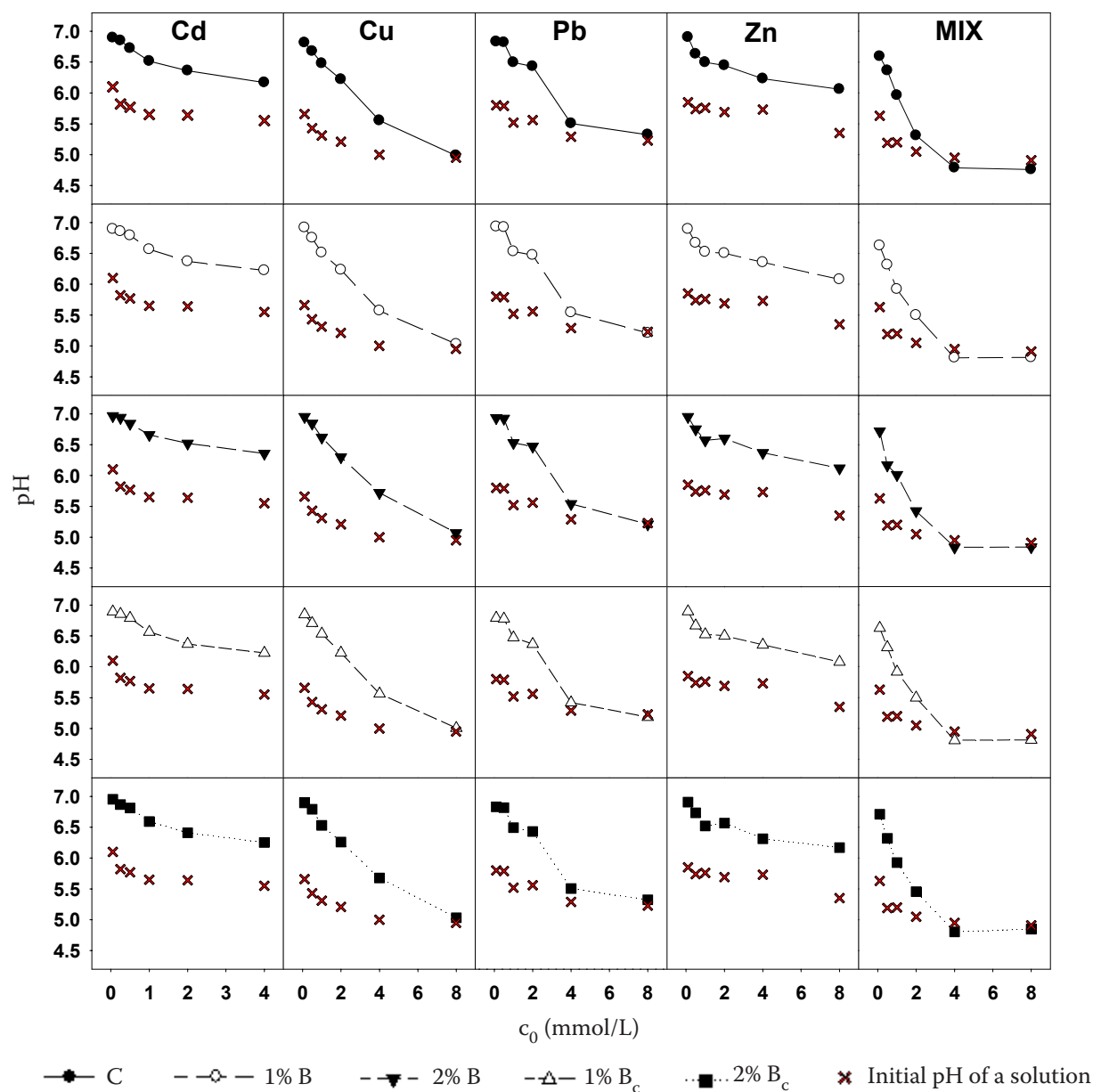

Figure 4. Observed pH changes of the experimental solution during the sorption experiment, compared with the initial pH of the solutions (without soil); c_0 – initial solute metal concentration; pH – final pH of the sorption solutions at equilibrium

necessary to mention that the applied rates (1% and 2% w/w) were insufficient in several cases for such extremely contaminated soil, where intensive competition during sorption of studied metals was recorded. Other recent works conducted on contaminated soils and where biochar was applied at rates of (5%, 10%, and 20% w/w; Beesley et al. 2010, Uchimiya et al. 2011a,b) showed the most promising results.

In summary, metal sorption efficiency in metal-contaminated soil was studied with and without biochar application. Additionally, two different types (contaminated and uncontaminated) and rates (1% and 2% w/w) were compared. In general, biochar application enhanced Cu and Pb sorption in all cases, whereas Cd and Zn sorption efficiency showed no significant changes. Results indicate low desorption of Cu a rather low Cd and Pb desorption and a high desorption of Zn. Zinc was predominately desorbed during Cu and Pb single-metal sorption experiment. Biochar treatment reduced Zn and Cd desorption during the single-metal sorption experiment of other competitive metals. Comparison of biochars originated from contaminated and uncontaminated willows showed no significant difference on metal sorption. Furthermore, two different applied rates (1% and 2% w/w) had a negligible effect on metal sorption. In summary, it is needed to point out that the applied rates of biochars were insufficient to significantly enhanced metal sorption efficiency.

Acknowledgements

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