PROPERTIES OF LEACHED FOREST-MEADOW CHERNOZEM POLLUTED WITH LEAD AND CADMIUM^{*}

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Abstract. The object of the study was leached forest-meadow chernozem, polluted by lead and cadmium. The samples were taken from the research field of Lviv State Agrarian University, from the layer of 0-60 cm deep. Heavy metals were applied to top layer of soil as water salt solutions with different doses, namely 0, 32, 160, 320 mg kg⁻¹ for Pb (Pb(CH₃COO)₂) and 0, 3, 15, 30 mg kg⁻¹ for Cd (CdCl₂·2.5 H₂O). Basic chemical and physical properties, such as soil reaction, organic carbon content, grain size composition, total surface area, porosity, hydrolytic acidity of the samples were analysed using routine laboratory procedures. The soluble forms of Pb and Cd were also investigated. The soil reaction ranged between 6.8 and 7.5 in 1M KCl and 7.4-8.1 in H₂O, whereas the content of organic C ranged between 0.62 and 1.59%. No significant relationship was detected between cadmium and lead amounts and the grain size composition of investigated samples. The pore volume of polluted soils ranged from 370 mm³ g⁻¹ to 515 mm³ g⁻¹ and was higher for Cd- polluted than for Pb-polluted soils. The total surface area of the soils varied from 26.83 m²g ¹ to 40.02 m^2g^{-1} . Content of available forms of cadmium and lead was the highest in the top layers of soils. Presence of those elements was similar to that in the control sample at the depth of 30 cm only for small initial doses of Cd and Pb. Content of Cd and Pb for the highest doses and at the deepest soil level was higher than for the control sample.

Keywords: soil pollution, heavy metals, lead, cadmium, forest-meadow chernozem, physicochemical properties, soluble forms of heavy metals

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INTRODUCTION

One of the elements of chemical degradation of soils is related with heavy metals content and they rank among the main pollutants in the environment. More than 85% of the total heavy metal content in soils origins from anthropogenic pollution. Contamination with heavy metals is a major concern because of their toxicity and threat to human life and environment. Soils are receptors of large quantities of heavy metals. Their accumulation and mobility in soils is determined largely by the extent of their adsorption by soil particles. In particular, soil organic and mineral particles can bind toxic elements by formation of complexes. Three principal stages of technogenic heavy metals transformation in soil are: 1) the formation of complexes; 2) heavy metals highly intensive migration; 3) high humus horizon intrusion (de Haan and van Riemsdijk 1986, McLean and Bledsoe 1992, Appel and Ma 2002). Some of agricultural soils are contaminated with lead (Pb) and cadmium (Cd), both of them belonging to 10 chemical elements that are the basic contaminants of the environment and they can be found in solid as well as in liquid phase. Parent rock of which the soils are developed is the natural source of heavy metals in those soils.

Soils exhibit high absorbing capacity to lead and cadmium. Several soil properties, such as the pH value, organic carbon content, grain size composition, specific surface area, porosity, hydrolytic acidity, can influence the availability of exchangeable and water soluble Cd and Pb determined as available for plants (McLean and Bledsoe, 1992). On the other hand, several physicochemical processes occurring in soils, such as water and ions adsorption, acid-base equilibrium and transport phenomena, are related to or governed by the surface properties of soil solid phase.

The main factor influencing the mobility of heavy metals in the soil environment is its reaction (pH) which affects the stability of humus complexes with metals. In soil with pH considerably above 7 (alkaline and carbonate soils of high sorption capacity), the transfer of heavy metals into non-soluble forms takes place without any additional agrotechnical treatments (Davies and Ballingen, 1990). The relationship between the amount of metal adsorbed and composition of the soil is quite complex, due to solid phase heterogeneity and influence of soil solution chemistry (Zhou De-Zhi et al. 1991, Appel and Ma 2002, Diatta et al. 2003). On the basis of stability diagrams, Santillan-Medrano and Jurinak (1975) pointed out that the solution activity of Cd is consistently higher than that of Pb, indicating that Cd is more mobile in the environment. In soils dominated by permanent charge surface, heavy metals are not mobile, but in variable charge soils, the low surface charge density creates conditions conductive to increased mobility. Consequently, the adsorption of heavy metals, in particular cadmium, by strongly weathered soils in relation to the effects of inorganic and organic ligands and the implications for metal transport were reviewed by Naidu et al. (1997).

The aim of this study was to investigate the basic properties of leached forestmeadow chernozem polluted with lead and cadmium. Basic chemical and physical properties, such as soil reaction, hydrolytic acidity, organic carbon, grain size composition, total surface area, soil porosity and content of water-soluble forms of Pb and Cd were measured.

MATERIALS AND METHODS

The soil samples were taken from the research field of the Lviv State Agrarian University. The experimental area of 300 m² was divided into 35 plots of 2 m² each. The phytotoxic effect of lead and cadmium on growth of spring barley was investigated at the contamination levels of 1.5 and 10 of the Threshold Limit Value. Heavy metals were applied onto top layer of soil as water salt solutions of Pb(CH₃COO)₂ in the case of lead and CdCl₂ in the case of cadmium, at different doses, namely 0, 32, 160, 320 mg kg⁻¹ of soil in the case of Pb, and 0, 3, 15 and 30 mg kg⁻¹ for Cd. The soil samples were taken from every successive 10 cm layer of the soil profile (of 0-60 cm depth).

Soil reaction, organic carbon, grain size composition, specific surface area, porosity, hydrolytic acidity of the studied soil were measured using routine laboratory analyses. Soil reaction was measured by a potentiometer with a combined glass/calomel electrode in 1M KCl and in H₂O at 1:2.5 soil to solution ratio. Organic carbon was determined oxidometrically with potassium dichromate in hot sulphuric acid, according to the modified Tiurin method. The grain size composition was evaluated by the areometric Cassagrande method modified by Prószyński. The specific surface area of investigated soil samples was obtained according to the Polish Standard PN-Z-19010-1. Before adsorption measurements the soil samples were dried at 105°C in a vacuum chamber with concentrated sulphuric acid until the weight of samples reached constant values. The amount of adsorbed water vapour at $T = 20^{\circ}C$ was computed as the difference between the weight of the sample with water and the dry sample. The relative water pressures were obtained from the density of sulphuric acid solutions. The adsorption measurements were replicated three times. Soil porosity was measured using a Micrometrics Mercury Porosimeter Autopore IV 9510 Model and applying the pressures from the range from 0.0036 to 413 MPa. This range allowed for determination of pores with equivalent radii ranging from 0.003 μ m to 360 μ m. Before porosity measurements the samples were oven-dried at 105°C and then degassed up 10⁻³ Pa to remove physically adsorbed water from their surface. The pore radii were calculated from the Washburn equation (Hajnos 1998). The surface tension and the contact angle of mercury were assumed to be 480 dynes

cm⁻² and 141.3°C, respectively. All calculations from obtained porosimetric data, i.e. the bulk density, pore surface area, average pore radius and total porosity, were carried out using cylindrical pore model by computer program Autopore IV Version 1.06.

Content of available forms of cadmium and lead in soils was investigated by atomic absorption spectrometry (AAS) using a Perkin Elmer 3300 apparatus. Soil was stressed with lead (CH₃(COO)₂Pb) with concentration of 0, 32, 160 and 320 mg Pb per kg of soil, and in cadmium (CdCl₂) with concentration of 0, 3, 15 and 30 mg Cd per kg of soil. Soil samples for those studies were collected in the second year of the experiment from three depths - 10, 20 and 30 cm. Afterwards, equal weight amounts of dried soils were extracted for 24 hours by mixture of 1 M hydrochloric (HCl) and 1 M nitric acid (HNO₃) at the ratio of 1:1. Received extracts of available Cd and Pb forms were filtrated. Content of lead and cadmium was analysed in clear solutions.

Received data were statistically calculated using t-student test for equal expectation value.

RESULT AND DISSCUSION

From the results obtained in this study it follows that the reaction of polluted soil samples was 6.8-7.5 in 1M KCl and 7.4-8.1 in H₂O, while the pH of control soil sample was 6.9-7.4 in 1M KCl and 7.5-7.9 in H₂O (see Table 1). The reaction of the soil was neutral. Significant differences (P > 0.05) between average values of pH in H₂O evaluated for the control plot and the plots contaminated with Pb were detected for the samples taken from 0-20 cm layers and contaminated at the doses of 160 and 320 mg Pb kg⁻¹ of soil. In contrast, such differences were not observed for any other samples polluted with Pb nor with Cd.

The fraction of organic carbon in all investigated samples was rather low. Indeed, the organic carbon content in the samples polluted with Pb and Cd ranged from 0.62% to 1.59%, whereas in the control soil it changed from 0.71% to 1.59%. Statistically significant differences (P > 0.05) between average carbon content values in the whole soil profile (0-60 cm) of contaminated samples and the control soil occurred only in the case of soils contaminated with Pb with the doses of 160 and 320 mg Pb kg⁻¹ of soil. However, within the layers of 0-20 cm (they exhibited the highest organic carbon content), significant differences in organic carbon content between contaminated and control samples were observed for all soils treated with Pb, as well as with Cd.

The transport and deposition of pollutants in soil profiles strongly depend on pH and the amount of organic matter. Several researchers have shown that heavy metals get bound to dissolved humic substances and this phenomenon significantly decreases

N	Depth	Grain size composition (%)			pН		TSA	
NO	(cm)	Sand	Silt	Clay	C_{org} , (%)	H_2O	1M KCl	(m^2g^{-1})
	Soil + Ph 32 (mg kg ⁻¹)							
1	0-10	22	58	20	1.56	8.0	7.5	29.9
2	10-20	23	56	21	1.47	8.0	7.5	30.1
3	20-30	11	54	35	1.45	8.1	7.5	27.7
4	30-40	19	55	26	1.20	7.9	7.2	27.9
5	40-50	24	45	31	0.88	7.6	7.1	37.3
6	50-60	25	51	24	0.66	7.4	6.8	37.7
Soil + Pb 160 (mg kg ⁻¹)								
1	0-10	19	60	21	1.53	7.9	7.4	29.1
2	10-20	21	56	23	1.46	8.0	7.4	29.0
3	20-30	22	50	28	1.41	8.1	7.6	28.5
4	30-40	8	54	38	1.19	7.7	7.2	29.4
5	40-50	8	57	35	0.95	7.5	6.9	35.6
6	50-60	12	55	33	0.70	7.5	7.0	37.4
				Soil + Pb 32	$0 ({\rm mg kg^{-1}})$			
1	0-10	13	65	22	1.55	7.9	7.4	28.9
2	10-20	17	59	24	1.48	8.0	7.5	29.8
3	20-30	17	61	22	1.44	8.0	7.4	29.8
4	30-40	17	54	29	1.16	7.6	7.1	31.2
5	40-50	17	48	35	0.91	7.3	6.9	33.0
6	50-60	9	58	33	0.67	7.5	7.0	37.2
				Soil + Cd 3	5 (mg kg ⁻¹)			
1	0-10	20	62	18	1.58	7.8	7.3	36.3
2	10-20	19	57	24	1.52	7.9	7.4	29.8
3	20-30	15	55	30	1.44	8.0	7.5	28.8
4	30-40	19	51	30	1.28	7.7	7.1	35.5
5	40-50	21	47	32	0.92	7.8	7.3	33.9
6	50-60	11	50	39	0.62	7.4	7.0	40.0
				Soil + Cd 1	$5 (\text{mg kg}^{-1})$			
1	0-10	25	50	25	1.55	7.9	7.4	30.2
2	10-20	19	58	23	1.49	8.1	7.6	30.8
3	20-30	13	60	27	1.39	7.8	7.4	29.3
4	30-40	14	53	33	1.30	7.9	7.3	33.9
2	40-50	/	50	43	0.85	1.1	1.2	39.2
0	50-60	9	03	28 5-11 + C1 20	0.72	1.5	0.9	30.7
- 1	0.10	20	<i>c</i> 0	Soll + Cd St	0 (mg kg)	7.0	7.4	20.5
1	0-10	20	60 47	20	1.57	/.9 0 1	1.4	30.5
2	10-20	20	4/	33	1.47	8.1	7.5	30.7
3	20-30	12	58 60	30	1.42	7.8 7.7	7.5	30.2
4	30-40 40 50	10	55	30	1.24	1.1 7 5	7.1	37.0 27.9
5 6	40-30	13	33	32	0.89	1.3 7 4	7.0	37.8 26.5
0	50-60	11	49	40	0.00 trol	/.0	7.0	30.3
1	0-10	20	54	26	1 50	77	7 2	30.1
2	10.20	20 19	50	20	1.59	7.7	7.4	30.1
3	20-30	13	57	30	1.50	79	7.4	26.8
3 4	30-40	0	56	35	1 23	7.5	7.5	30.4
5	40-50	15	55	30	0.94	7.0 7 7	7.2	33.9
6	50-60	13	54	33	0.74	75	69	38.7

Table 1. Basic properties of samples contaminated with lead and cadmium

Abbreviation: sand fraction: 1-0.1 mm, silt: 0.1-0.02 mm, clay: < 0.02 mm, C_{org.} – Organic carbon, TSA – Total surface area.

the negative impact of heavy metals on plants and soil organisms (Davies and Ballingen 1990, Wójcikowska-Kapusta *et al.* 2002). For all contaminated soil samples their grain size composition was determined. For Pb-treated samples the content of sand fraction changed from 8 to 25%; silt fraction - from 48 to 65% and colloidal clay fraction – from 20 to 35%. For Cd polluted soils the content of sand fraction was from 7 to 25 %, silt fraction – from 47 to 63% and clay – from 18 to 43%. The grain size composition of the control soil was as follows: sand – 9-20%, silt - 54-59%, and clay – 23-35%. Obviously, the grain size composition depends on the soil horizon (Table 1). However, one can state that the grain size composition of the control soil samples were not significantly different from the composition of the control soil.

For all studied samples the TSA evaluated from the adsorption isotherms ranged from 28.8 to 40.0 m²g⁻¹ for Cd-polluted soils and from 27.7 to 37.7 m²g⁻¹ for soils polluted by Pb. The BET specific surface area for control soil ranged from 26.8 to 38.7 m²g⁻¹ (Table 1) and there were no essential differences between the polluted soil samples and the control soil. The average values of specific surface area were 31.6 m²g⁻¹, 33.8 m²g⁻¹ and 31.7 m²g⁻¹ for soil-Pb, soil-Cd and control soil, respectively. The highest values of specific surface area were observed for samples taken from the layers 40-60 deep and contaminated with lead and cadmium. The differences between surface area of the control sample and soil polluted by heavy metals were rather low. That may be connected with a different factor, such as low concentration of metals in soil and heterogeneity of samples which were taken from the experimental plots.

The results of the mercury porosimetry measurements are given in Table 2. The samples were taken from the layers of 0-10 cm and 10-20 cm deep. The total intrusion volume (TV) was directly measured in quantitative way, whereas the total pore area (TPA), average pore diameter ($R_{aver.}$) and bulk density were calculated using the cylindrical pore model.

The pore volume (TV) of investigated polluted soils ranged from 370 mm³g⁻¹ to 515 mm³g⁻¹ and was higher for cadmium-polluted samples than for the samples of soil polluted with lead. Both polluted soils exhibited also different values of PV for the upper and lower horizons. While TV decreases with depth for the Pb-polluted soil, it increased for the soil containing Cd (Table 2). The porosity of samples containing Cd was higher than of samples with Pb. The changes in TP (total porosity) with depth were similar to those already observed for TV. In general, the values of TV, TP and average pore radii were found to be lower for Pb-soils than for Cd-soils. The above values point to a somewhat more developed porous structure of Cd-soil compared with that for Pb-soil.

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Ν	Depth	TV	TPA	PD_{av}	D	TP	
0	(cm)	$(mL g^{-1})$	$(m^2 g^{-1})$	(nm)	(g mL ⁻)	(%)	
	Soil + Pb 320 (mg kg ⁻¹)						
1	0-10	0.404	3.60	448.3	1.259	50.83	
2	10-20	0.370	3.72	398.2	1.300	48.13	
			Soil + Cd 30 (mg kg	g ⁻¹)			
3	0-10	0.426	3.44	495.3	1.211	51.63	
4	10-20	0.515	3.73	552.2	1.100	56.61	
Control							
5	0-10	0.420	3.50	481.1	1.223	51.42	
6	10-20	0.411	3.74	438.8	1.244	51.09	

Table 2. Parameters of pore structure of leached chernozem degraded by lead and cadmium

Abbreviations: TV- total intrusion volume, TPA – total pore area, PD_{av} – average pore diameter, D – bulk density, TP – total porosity.

Generally, the samples of Cd-soil were characterised by higher values of all investigated parameters, i.e. total volume of intruded mercury, pore volume, total porosity, average pore radius and the amounts of storage and residual pores. The above differences can be connected with the behaviour of Cd in the environment. Different influence of lead and cadmium on porosity of leached chernozem can be also related to different chemical nature of these heavy metals and to the properties of the soil.

The results of mercury porosimetry of samples from the experimental plot were compared with those for samples prepared in the laboratory. In Figure 1 A and B the cumulative curves for laboratory and plot samples are shown.



Fig. 1. Cumulative curves for chernozem polluted by lead (320 mg kg⁻¹) and cadmium (30 mg kg⁻¹). Abbreviations: r - pore radius, PV - pore volume, L - samples prepared in the laboratory, P - samples taken from the experimental plot, 0-10 cm and 10-20 cm - depth

The difference between PV of polluted soil samples prepared in the laboratory and those from the experimental plot are rather connected with heterogeneity of the samples.

The results of studies of cadmium and lead content in selected levels of investigated soil are presented in Table 3.

Table 3. Cadmium and lead content at selected depths of soil and for different initial doses of metals

Depth		Metal content (1	mg dm ⁻³)			
(cm)	Control soil	Soil+dose of Cd (mg kg ⁻¹)				
	Control son	3	15	30		
10	0.101	0.182	0.224	0.535		
20	0.077	0.122	0.195	0.166		
30	0.099	0.114	0.112	0.209		
	Control soil	Soil+dose od Pb (mg kg ⁻¹)				
	Control son	32	160	320		
10	0.61	2.93	5.17	9.81		
20	0.51	1.01	3.95	7.46		
30	0.56	0.74	3.52	6.07		

The results show that after two years of the experiment, the highest concentration of cadmium and lead remained constantly in the shallowest soil layer, irrespective of the initial dose of cation (Figure 2 and 3). In the case of cadmium, the highest dose (30 mg Cd per kg of soil) was illuviated faster than smaller doses of cadmium (3 and 15 mg Cd per kg of soil). The control sample contained also a small amount of cadmium, but it was definitely smaller in all layer levels than for the soils with metal. After two years of the experiment, the content of Cd at the depth of 30 cm and for initial doses of 3 and 15 mg Cd per kg of soil was still similar to the control sample. Only soil samples with the highest concentration of Cd showed a higher content of Cd at the depth of 30 cm than the control sample at the same level. Soil stressed with lead showed a similar relationship. The content of Pb was similar to the content of Pb in the control sample.

Decrease of heavy metal content with depth is connected with the process of gradual migration and illuviation to the soil profile. Similar contents of cadmium and lead as for the control samples can be evidence of weak rate of migration of small amounts of Cd (3 and 15 mg kg⁻¹) and lead (32 mg kg⁻¹). The highest doses of heavy metals migrate to the soil faster, and after the second year a slightly higher

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content of lead and cadmium can be observed at the depth of 30 cm. That fact can be connected with the possibility of creation of less mobile and more sparingly soluble organomineral forms of lead and cadmium in shallower layers of soil. In that way, small doses of Cd and Pb can remain longer in the layer of 0-20 cm. Application of big doses of metals causes that a part of them can be chemically bound and the rest will be illuviate to the soil. Therefore, higher presence of Cd and Pb can be observed at the depth of 30 cm.



Fig. 2. Concentration of Pb at selected depths of soil for different initial doses of Pb



Fig. 3. Concentration of Cd at selected depths of soil for different initial doses of Cd

CONCLUSIONS

The behaviour of lead and cadmium in soil is different, and different influence of lead and cadmium on basic characteristics of leached chernozem can be connected with the chemical properties of both heavy metals and the properties of the soil. Pore volume of investigated polluted soils ranged from 370 mm³ g⁻¹ to 515 mm³ g⁻¹. The reaction of the soil samples was neutral. Within 0-20 cm layers significant differences between average values of pH (in H₂O) for polluted and control soils were detected only in the case of samples contaminated with 160 mg and 320 mg doses Pb (per 1 kg of soil). The soil samples were characterised by a low content of organic carbon fraction. Statistically significant differences (P > 0.05) between the average fraction of organic carbon in the whole soil profile (0-60 cm) occurred only for samples contaminated with 160 and 320 mg of Pb per 1 kg of soil. In the case of results obtained from mercury intrusion experiments, the Cd-polluted samples possessed higher values of all investigated parameters, i.e. the total intrusion volume of mercury, pore volume, total porosity, average pore radii and amounts of storage and residual pores. For both polluted solids the above-listed parameters varied with the soil horizon, but they did not essentially change with the dose of the applied pollutant. The highest content of cadmium and lead was observed at the depth of 0-20 cm for all doses of Cd and Pb. Presence of those elements at the depth of 30 cm was significantly smaller for the smallest doses of lead and cadmium because of the possibility of creation of less mobile and more sparingly soluble organomineral forms of lead and cadmium in shallower layers of soil. Rate of migration of cadmium and lead forms in soils can be evidence and index of soil self-purification.

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WŁAŚCIWOŚCI WYŁUGOWANEGO CZARNOZIEMU LEŚNO-STEPOWEGO, ZANIECZYSZCZONEGO OŁOWIEM I KADMEM

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Streszczenie. Przedmiotem badań była gleba wyługowanego czarnoziemu leśno-stepowego, zanieczyszczonego ołowiem i kadmem. Gleby pochodziły z poletek doświadczalnych stacji badawczej Uniwersytetu Rolniczego we Lwowie. Obszar 300 m² podzielono na poletka doświadczalne wielkości 2 m². Do gleby dodano kadm w postaci CdCl₂ o stężeniach 0, 3, 15, 30 mg Cd na kg gleby i ołów w postaci Pb(CH₃COO)₂ o stężeniach 0, 32, 160, 320 mg Pb na kg gleby. Na poletkach wysiano jęczmień jary odmiany Pejas. Po dwóch latach uprawy pobrane zostały próbki gleby z głębokości 0-60 cm. Podstawowe charakterystyki gleby takie jak: odczyn gleby, zawartość węgla organicznego, skład granulometryczny, powierzchnia właściwa, porowatość gleby, kwasowość wymienna badanych gleb oznaczono wykorzystując standardowe metody laboratoryjne. Odczyn badanych gleb zawierał się w przedziale 6,8-7,5 (w KCl) i 7,4-8,1 (w H2O). Zawartość węgla organicznego oscylowała w granicach 0,62-1,59%. Badana gleba charakteryzowała się niską zawartością węgla organicznego. Nie odnotowano istotnego wpływu jonów kadmu i ołowiu na skład granulometryczny gleb. Objętość porów w badanych glebach zmieniała się od 370 mm3·g-1 do 515 mm³·g⁻¹ i była wyższa dla gleb zanieczyszczonych kadmem niż dla gleby zanieczyszczonej ołowiem. Powierzchnia właściwa badanych gleb zawierała się w przedziale 26,83 m²·g⁻¹ – 40,02 m²·g⁻¹. Pomiędzy glebami zanieczyszczonymi ołowiem (0, 32, 160, 320 mg Pb na kg gleby) i kadmem (0, 3, 15, 30 mg Cd na kg gleby) nie odnotowano istotnej różnicy badanych parametrów.

Słowa kluczowe: metale ciężkie, ołów, kadm, porowatość, pH, węgiel organiczny, skład granulometryczny, powierzchnia właściwa