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EVALUATION OF THE ADDITION OF IMMOBILIZING AGENTS  
ON SELECTED PHYSICOCHEMICAL PROPERTIES OF SOIL  
CONTAMINATED WITH HEAVY METALS

*Received: 13.09.2017*

*Accepted: 06.02.2018*

*Abstract.* This study was aimed at determining effects of increasing simultaneous contamination of soil with Pb and Zn at doses of 200, 400 and 800 mg·kg<sup>-1</sup> and with Cd at doses of 5, 10 and 20 mg·kg<sup>-1</sup> soil, after the addition of immobilizing agents (zeolite, diatomite, chalcedonite, dolomite, limestone, and activated carbon), on selected properties of soil. In the soil without the immobilizing agents, the highest doses of metals caused a decrease in pH value, total sorption capacity, total exchangeable base cations and saturation of the sorption complex with base cations as well as an increase in hydrolytic acidity and electrical conductivity of the soil. Among the agents applied, dolomite and activated carbon exerted a positive effect on the analyzed properties of soil, as they contributed to an increase in pH value, total exchangeable bases, and total sorption capacity, and to a decrease in hydrolytic acidity of the soil. The addition of zeolite caused soil electrical conductivity to decrease compared to the control pot, at all levels of soil contamination with Pb, Cd, and Zn.

**Keywords:** soil contamination, immobilizing agents, soil physicochemical properties, analyses

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## INTRODUCTION

Land degradation caused by human activities has significant adverse effects on the environments especially on the soils worldwide (Mandal and Sharda 2013, Lago-Vila *et al.* 2015, Adamcová *et al.* 2016). Soil is a complex environmental medium with high heterogeneity where solid, liquid and gaseous components interact within a multitude of physical, chemical and biological interrelated processes. Soil provides ecosystem services such as food, water, timber, and fiber; regulating services that affect climate, floods, disease, waste and water quality (Zornoza *et al.* 2015). The problem of soil contamination with heavy metals is of great significance considering the possibility of its direct or indirect impact on human health (Ali *et al.* 2013, Paz-Ferreiro *et al.* 2014, Roy and McDonald 2014, Morcillo *et al.* 2016). Especially severe problem is posed by soil contamination with heavy metals as a result of their migration or accumulation in individual components of the natural environment (Ha *et al.* 2014, Yang *et al.* 2016, Yang *et al.* 2017). Among these heavy metals, cadmium (Cd), lead (Pb) and zinc (Zn) contribute the major share of soil contamination because they are non-biodegradable and highly persistent in the soil environment (Adrees *et al.* 2015, Ahmadi *et al.* 2014, Cao *et al.* 2017). The main factors that regulate the appropriate binding of heavy metals in soil include environment pH, sorption capacity, moisture content, content of organic matter with various capability of heavy metal complexation, as well as contents of iron hydroxides, manganese, aluminum, and silty fractions (Annu *et al.* 2016). Also the physicochemical properties of soil affect the uptake of elements by plants and their incorporation into the food chain (Guo *et al.* 2016, Cao *et al.* 2017). Pb content in soils is directly influenced by their granulometric and mineralogical composition as well as by the source of bedrock formation. Its presence in the topsoil results, primarily, from the effect of anthropogenic factors (Rosestolato *et al.* 2015, Cao *et al.* 2017). In most countries, Pb content in soils not exposed to the immediate effect of this metal is low. The average natural content of Pb in soils of Poland is at  $18 \text{ mg} \cdot \text{kg}^{-1}$  (Kabata-Pendias and Szteke 2012). In turn, Cd is a natural element of geological formations and its content in magmatic rocks does not exceed  $0.2 \text{ mg} \cdot \text{kg}^{-1}$ . In sedimentary rocks, especially these formed upon biological processes, Cd content may be several times higher. It refers also to organic and mineral rocks (Kabata-Pendias and Szteke 2012). The natural content of Cd in soils is determined by a few factors including: geological origin of bedrocks, soil age, and strength of weathering processes. It occurs mainly in the form of sulfides, at deposits of Zn and Cu. Cd concentrations in soils in many countries are increasing due to inadvertent additions in fertilizer, biosolids, and soil amendments, as well as additions from the atmosphere (Czarnecki and Düring 2015, Annu *et al.* 2016). The mean concentration of Cd in soils of the globe usually does not exceed  $0.5 \text{ mg} \cdot \text{kg}^{-1}$ , whereas in Polish soils it ranges from 0.20 to

0.31 mg·kg<sup>-1</sup> soil. There are, however, some cases where it ranges from 0.01 to 24.75 mg·kg<sup>-1</sup> (Terelak *et al.* 1995). Zn is an essential micronutrient involved in photosynthesis; as such, there is a growing effort to obtain understanding of the Zn biogeochemical cycle at the Earth's surface (Opfergelt *et al.* 2017). Zn is one of the most active heavy metals of soil because it is capable to form complexes with organic matter and occurs in exchangeable forms. The organic matter of soil forms strong bonds with Zn, which results in its accumulation in the top layers of mineral and organic soils (Opfergelt *et al.* 2017). Its solubility decreases along with pH increase, regardless of the form it occurs in the soil. In the case of formation of complex anions as well as organic and mineral complexes, Zn is capable of retaining high mobility in soils with alkaline pH. In Poland, the average concentration of Zn in non-contaminated soils reaches 40 mg·kg<sup>-1</sup>, but in industrial areas it may exceed even several thousand mg·kg<sup>-1</sup> (Kabata-Pendia and Szteke 2012).

The aim of this study was to determine the effect of soil contamination with Pb, Cd, and Zn as well as to evaluate the effect of zeolite, diatomite, chalcodonite, dolomite, limestone, and activated carbon on selected physicochemical properties of soil. To achieve study goal, soil samples collected after termination of the experiment were subjected to the following laboratory analyses: pH value (pH), hydrolytic acidity, total exchangeable base cations, total sorption capacity of the soil, and saturation of the sorption complex with base cations.

## MATERIALS AND METHODS

The experiment was conducted in a vegetation hall under conditions of a pot experiment in polyethylene (PE) pots with the volume of 5.0 kg. Soil used in the experiment was collected from the topsoil of arable lands and had the following properties: pH – 4.92; hydrolytic acidity (mmol·kg<sup>-1</sup>) – 31.21; total exchangeable base cations (mmol·kg<sup>-1</sup>) – 61.10; total sorption capacity (mmol·kg<sup>-1</sup>) – 94.20; saturation of the sorption complex with base cations (%) – 65.20; total nitrogen (g·kg<sup>-1</sup>) – 1.22; organic carbon (g·kg<sup>-1</sup>) – 7.42; N-NH<sub>4</sub><sup>+</sup> (mg·kg<sup>-1</sup>) – 20.32; N-NO<sub>3</sub><sup>-</sup> (mg·kg<sup>-1</sup>) – 10.01; exchangeable P (mg·kg<sup>-1</sup>) – 23.20; exchangeable K (mg·kg<sup>-1</sup>) – 8.72; exchangeable Mg (mg·kg<sup>-1</sup>) – 31.20; Pb (mg·kg<sup>-1</sup>) – 16.38; Cu (mg·kg<sup>-1</sup>) – 8.12; Zn (mg·kg<sup>-1</sup>) – 23.22; and Mn (mg·kg<sup>-1</sup>) – 208.3.

Before the experiment was established, the soil had been contaminated with increasing doses of heavy metals, acc. to the following schemes: scheme I. Pb: 200 (mg·kg<sup>-1</sup>); Cd: 5 (mg·kg<sup>-1</sup>); Zn: 200 (mg·kg<sup>-1</sup>); scheme II. Pb 400 (mg·kg<sup>-1</sup>); Cd: 10 (mg·kg<sup>-1</sup>); Zn 400 (mg·kg<sup>-1</sup>); and scheme III. Pb: 800 (mg·kg<sup>-1</sup>); Cd: 20 (mg·kg<sup>-1</sup>); Zn 800 (mg·kg<sup>-1</sup>). Heavy metals were introduced into the soil in the form of aqueous solutions of: Pb as Pb(NO<sub>3</sub>)<sub>2</sub>, Cd as 3CdSO<sub>4</sub>·8H<sub>2</sub>O, and Zn as Zn(NO<sub>3</sub>)<sub>2</sub>. Sorption materials including: zeolite, diatomite, chalcodonite, dolo-

mite, limestone, and activated carbon, were applied additionally in the amount of 3% of soil mass. Table 1 provides oxide composition and specific surface of the immobilizing agents. Also, a control series was conducted, which was free from heavy metals and immobilizing agents. Aqueous solutions of macro- and microelements were poured into each pot to ensure the appropriate growth of plants. The plant grown in the study was perennial ryegrass (*Lolium perenne* L.) of Bokser cultivar (Fig. 1).

Table 1. Characteristics of immobilizing agents used in the experiment

Type of immobilizing agents	Specific surface ( $\text{m}^2 \cdot \text{g}^{-1}$ )	Oxide composition (%)
Zeolite	34.5	$\text{SiO}_2$ – 70.10; $\text{Al}_2\text{O}_3$ – 12.15; $\text{CaO}$ – 4.96; $\text{K}_2\text{O}$ – 3.21; $\text{Fe}_2\text{O}_3$ – 1.75; $\text{MgO}$ – 1.08; $\text{Na}_2\text{O}$ – 1.22; $\text{TiO}_2$ – 0.22; SiAl – 5.31
Diatomite	29.3	$\text{SiO}_2$ – 54.72; $\text{Fe}_2\text{O}_3$ – 25.50; $\text{Al}_2\text{O}_3$ – 14.82; $\text{C}_2\text{O}$ – 4.18; $\text{MgO}$ – 0.79
Chalcedonite	7.44	$\text{SiO}_2$ – 84.77; $\text{Al}_2\text{O}_3$ – 9.33; $\text{C}_2\text{O}$ – 4.29; $\text{K}_2\text{O}$ – 1.21; $\text{MgO}$ – 0.40
Dolomite	1.22	$\text{CaO}$ – 38.12; $\text{N}_2\text{O}_2$ – 22.30; $\text{C}_2\text{O}$ – 20.18; $\text{SiO}_2$ – 6.91; $\text{Al}_2\text{O}_3$ – 4.51; $\text{Fe}_2\text{O}_3$ – 4.41; $\text{CuO}$ – 2.28; $\text{MgO}$ – 1.29
Limestone	0.91	$\text{CaO}$ – 87.47; $\text{Fe}_2\text{O}_3$ – 7.80; $\text{C}_2\text{O}$ – 1.50; $\text{SiO}_2$ – 1.34; $\text{N}_2\text{O}_5$ – 0.78; $\text{Al}_2\text{O}_3$ – 0.49; $\text{K}_2\text{O}$ – 0.48; $\text{MgO}$ – 0.05
Activated carbon	856.05	–

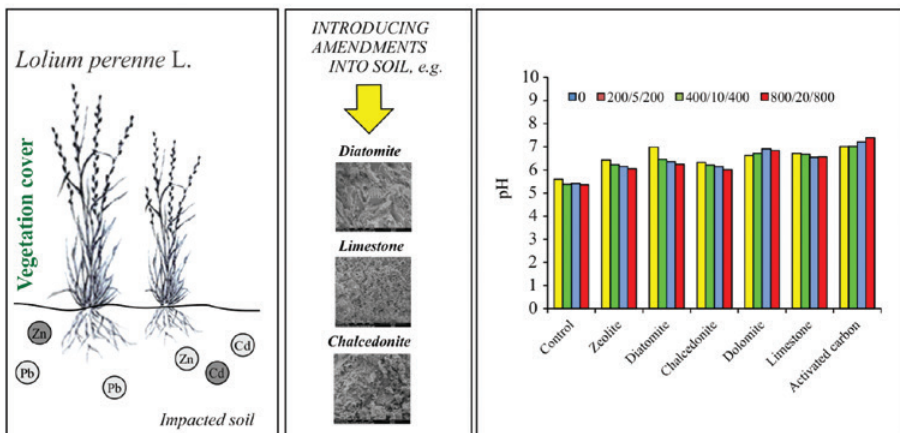


Fig. 1. Overview of the experiment activities

Before the experiment was established and after its termination, soil samples were collected from each pot, next dried at room temperature, disintegrated, and sieved through a sieve with a mesh diameter of 1 mm. Thus prepared

soil was determined for its selected properties: soil pH (pH) – potentiometrically in an aqueous solution of potassium chloride (KCl) with the concentration of  $1 \text{ mol} \cdot \text{dm}^{-3}$  (Lityński *et al.* 1976); hydrolytic acidity (Hh) – with Kappen’s method (Lityński *et al.* 1976); total exchangeable base cations (S) –with Kappen’s method (Lityński *et al.* 1976); total sorption capacity of soil (T) acc. to the following formula:  $T = \text{Hh} + \text{S}$  (Lityński *et al.* 1976); and saturation of the sorption complex with base cations (V) acc. to the following formula:  $V = \text{S}^{-1} \cdot 100$  (Lityński *et al.* 1976).

Statistical analysis of study results was conducted using Statistica 12 software package.

## RESULTS AND DISCUSSION

In spite of heavy metals’ natural occurrence in nature, these elements can act as pollutants, contaminating ecosystems, and can also exhibit toxicity towards living organisms (Hu *et al.* 2017). Many forms of heavy metals in the environment are generally stable; they do not degrade over time. So, they accumulate in the environment reaching dangerous concentrations (Vareda and Durães 2017). However, both anthropogenic and natural factors determine contents of heavy metals in soils, including contents of Cd, Zn, and Pb, as well as modify its physicochemical properties (Mazur *et al.* 2015, Adamcová *et al.* 2016, Mazur and Mazur 2016). These properties are affected by the extent of soil contamination with heavy metals and by the type of immobilizing agents added to the soil (Gul *et al.* 2015). In the presented study, the pH value, electrical conductivity, hydrolytic acidity, and sorption properties of the soil the test plant was cultivated in were influenced by both: doses of heavy metals used to contaminate the soil and immobilizing agents applied, i.e. zeolite, diatomite, chalcedonite, dolomite, limestone, and activated carbon (Table 2–3). Being one of the factors that affect the form the trace elements occur in the soil environment, the pH value determines their availability to plants (Boente *et al.* 2017). A negative correlation ( $r = -0.751$ ) was observed in our study for the soil sampled from control pots between its pH value and increasing contamination with Pb, Cd, and Zn. Activated carbon caused the highest increase in soil pH which attained values ranging from 7.01 to 7.39. A similar, though lesser, effect on contaminated soil pH increase was induced by the addition of diatomite, dolomite, and limestone.

The effect of high contents of metals: Pb, Cd, Zn and Cu in soil on soil pH decrease was demonstrated by Friedlova (2010). Soil pH decrease may affect increased uptake of heavy metals from soil by plants (Fitamo *et al.* 2011, Chibuike and Obiora 2014).

Table 2. The pH value (pH) and electrical conductivity (EC) of soil

Pb, Cd and Zn dose in mg·kg of soil	Type of immobilizing agents							
	Control	Zeolite	Diatomite	Chalcedonite	Dolomite	Limestone	Activated carbon	Average
	pH							
0	5.59	6.42	6.99	6.32	6.62	6.72	7.01	6.52
200/5/200	5.38	6.22	6.45	6.21	6.72	6.68	7.02	6.38
400/10/400	5.41	6.15	6.36	6.14	6.91	6.55	7.21	6.39
800/20/800	5.36	6.05	6.24	6.01	6.84	6.57	7.39	6.35
r	-0.751*	-0.936**	-0.848**	-0.992**	0.766**	-0.824**	0.972**	-0.373
LSD for: metal dose – 0.05**; immobilizing agents – 0.04**; interaction – 0.11**								
	Electrical conductivity (EC), $\mu\text{S}\cdot\text{cm}^{-1}$							
0	182	156	202	204	240	240	212	205
200/5/200	197	168	217	185	281	274	223	220
400/10/400	242	208	232	221	321	292	229	249
800/20/800	619	222	391	600	415	373	237	408
r	0.928**	0.945**	0.996**	0.893**	0.999**	0.992**	0.968**	0.953**
LSD for: metal dose – 16.45**; immobilizing agents – 15.43**; interaction – 29.08**								

\*\* – significant at  $p \leq 0.01$ ; \* – significant at  $p \leq 0.05$ ; r – correlation coefficient

In all experimental pots, we observed an increase in the electrical conductivity of soil along with its increasing contamination with heavy metals. Compared to the control, the changes in conductivity varied and were due to the elution of ions from the immobilizing agents and to the sorption of metal ions by these agents from the introduced contaminating solutions (Zhao *et al.* 2011, Mishra 2014).

The elution of chemical substances, like e.g. nutrients, from soil is affected and regulated by such factors as the content of base cations and sorption properties of soil (Hartmann *et al.* 1998). The value of hydrolytic acidity may be one of the indicators of soil acidification. In the presented study, it was significantly influenced by the extent of soil contamination with heavy metals (Table 3). The hydrolytic acidity of soil after the harvest of the test plant, in the series without additives, was affected by the dose of Cd, Zn, and Pb. In this series, scheme II of contamination, i.e. 400 mg Pb and  $\text{Zn}\cdot\text{kg}^{-1}$  and 10 mg  $\text{Cd}\cdot\text{kg}^{-1}$  soil, caused a 25% increase in the value of hydrolytic acidity. The introduction of immobilizing agents in the form of zeolite, diatomite, chalcedonite, dolomite, limestone, and activated carbon, also changed its value (Table 2). Activated carbon and limestone were the most effective in decreasing the hydrolytic acidity of soil compared to objects without the addition of immobilizing agents to soil.

Káтай *et al.* (2008) demonstrated that the hydrolytic acidity of soil depended on zeolite and bentonite contents in this soil. Results obtained in our study are partly in agreement with findings reported by Wyszowski and Modrzewska (2016) who showed that a high Zn concentration in soil reduced its acidity, its saturation with base cations and its total exchangeable capacity, but increased its hydrolytic acidity.

The applied doses of Pb, Cd, and Zn evoked significant changes in the content of total exchangeable base cations in the soil after test plant harvest (Table 3). In the series without immobilizing agents, trace elements introduced into the soil caused a successive decrease in the content of these cations along with their increasing doses. In the series with the addition of immobilizing agents, contents of the total exchangeable base cations in the soil after test plant harvest were diversified (Table 3). Zeolite, diatomite, chalcedonite, and limestone, had a small but negative effect on their value, with especially negative effect observed upon zeolite addition. Compared to the control series (without additives), this soil property was positively affected by the addition of dolomite and activated carbon

The total exchangeable capacity of the soil the test plant was grown in was significantly influenced by the dose of heavy metals used to contaminate it (Table 3). In the series without immobilizing agents, a negative correlation was demonstrated between the increasing contamination of soil with trivalent chromium ( $r=-0.931$ ) and the total exchangeable capacity of soil after the harvest. The application of immobilizing agents in the cultivation of the test plant caused changes in the total exchangeable capacity of soil, however, its value was positively affected by the addition of activated carbon and dolomite. The other agents applied into soil contaminated with heavy metals had negative effects on the mean value of soil total exchangeable capacity. In an experiment conducted by Wyszowski and Radziemska (2009), additives including compost, zeolite and CaO introduced into soil contaminated with Cr compounds evoked a positive effect by increasing soil pH, soil saturation with base cation, mean value of soil exchangeable capacity, and by decreasing its hydrolytic acidity. In turn, Wyszowski and Modrzewska (2016) demonstrated that the total exchangeable base cations, total exchangeable capacity, and saturation of the soil with base cations were the most beneficially affected by the addition of bentonite. Successively, the application of zeolite and compost had a little effect on hydrolytic acidity and sorption properties of soil contaminated with Zn.

In the presented experiment, soil saturation with base cations was significantly influenced by the dose of Pb, Cd, and Zn (Table 3). In the series without immobilizing agents, after the harvest, increasing doses of contaminating metals decreased soil saturation with base cations by 6% compared to the control pot. All neutralizing additives, except for dolomite and activated carbon, diminished soil saturation with base cations.



Table 3. Hydrolytic acidity, total exchangeable base cations, total exchangeable capacity, and soil saturation with base cations

Pb, Cd and Zn dose in mg·kg of soil	Type of immobilizing agents							
	Control	Zeolite	Diatomite	Chalcedonite	Dolomite	Limestone	Activated carbon	Average
Hydrolytic acidity, mmol(H <sup>+</sup> )/kg								
0	11.40	8.18	15.68	12.12	7.58	6.83	3.75	9.35
200/5/200	12.80	9.53	15.91	12.87	8.18	6.75	4.35	9.91
400/10/400	14.25	10.73	17.42	14.12	7.88	7.88	3.61	10.83
800/20/800	13.43	13.05	18.91	17.03	7.05	8.85	3.08	11.63
r	0.696*	0.999**	0.987**	0.971**	-0.633**	0.958**	-0.747*	0.461
LSD for: metal dose – 0.35**; immobilizing agents – 0.33**; interaction – 0.76**								
Total exchangeable base cations, mmol(H <sup>+</sup> )/kg								
0	126.2	142.3	153.3	93.8	121.0	162.7	118.7	131.1
200/5/200	124.1	108.4	109.4	85.7	122.9	157.7	138.9	121.0
400/10/400	94.11	100.5	89.4	84.7	136.9	130.6	169.4	115.0
800/20/800	84.81	80.21	84.4	68.5	144.2	123.5	197.6	111.8
r	-0.927**	-0.942**	-0.859**	-0.978**	0.957**	-0.927**	0.987**	-0.380
LSD for: metals dose – 2.14**; immobilizing agents – 2.09**; interaction – 5.03**								
Cation exchange capacity, mmol(+)/kg								
0	137.6	150.5	169.1	105.8	128.6	169.5	122.5	140.5
200/5/200	136.8	117.9	125.3	97.71	131.1	164.5	143.3	130.9
400/10/400	108.4	111.2	106.8	98.82	144.8	138.5	173.1	125.9
800/20/800	98.2	93.3	103.3	85.51	151.3	132.4	200.7	123.5
r	-0.931**	-0.932**	-0.845**	-0.957**	0.955**	-0.923**	0.987**	-0.401
LSD for: metal dose – 2.62**; immobilizing agents – 2.59**; interaction – 6.02**								
Saturation of the sorption complex with base cations, %								
0	91.52	94.57	90.68	88.64	94.11	95.97	96.94	93.20
200/5/200	90.72	91.91	8.22	87.73	93.76	95.91	96.96	92.03
400/10/400	86.84	90.35	83.71	85.73	94.56	94.28	97.92	90.48
800/20/800	86.36	85.95	81.71	80.09	95.31	93.31	98.47	88.74
r	-0.901**	-0.997**	-0.954**	-0.981**	0.898**	-0.956**	0.952**	-0.421
LSD for: metal dose – 1.32**; immobilizing agents – 1.27**; interaction – 3.21**								

\*\* – significant at  $p \leq 0.01$ ; \* – significant at  $p \leq 0.05$ ; r – correlation coefficient

## CONCLUSIONS

1. In the soil without the immobilizing agents, the highest doses of metals caused a decrease in pH value, total sorption capacity, total exchangeable base cations and saturation of the sorption complex with



- base cations as well as an increase in hydrolytic acidity and electrical conductivity of the soil.
2. Among the agents applied, dolomite and activated carbon exerted a positive effect on the analyzed properties of soil, as they contributed to an increase in pH value, total exchangeable bases, and total sorption capacity, and to a decrease in hydrolytic acidity of the soil.
  3. The addition of zeolite caused soil electrical conductivity to decrease compared to the control pot, at all levels of soil contamination with Pb, Cd, and Zn.

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