LIMING EFFECT ON SOIL HEAVY METALS AVAILABILITY

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SUMMARY

The aim of this paper was to determine the influence of acid soils liming and initial soil acidity as well as organic matter content on availability of four heavy metals (Zn, Pb, Cr and Cd). Liming experiment was conducted in laboratory conditions with six soils of different acidity according to rapid incubation method which was conducted in sealed containers for three days at a constant temperature of 60°C. Liming treatments resulted in trend of heavy metals availability decrement in all soils, but intensity of decrement differed considering initial soil acidity and initial heavy metals availability. According to relative heavy metals availability decrement, liming resulted in the strongest effect in extremely acid soils with the highest initial concentrations of available Zn, Pb, Cr and Cd. On the other side, the weakest relative liming effect on heavy metals availability decrement was recorded in moderately acid soils with the lowest initial concentrations of available heavy metals. Considering impact of initial humus content in soil, higher relative liming efficiency of heavy metals availability decrement was determined in soils with higher soil organic matter content and with lower initial concentrations of available heavy metals.

Key-words: *acid soils, liming, heavy metals availability, total concentrations of heavy metals*

INTRODUCTION

Necessity of excessive soil acidity correction is related to soil reaction influence to a whole range of chemical, biological and physical soil properties. Therefore, liming represents common amelioration measure for conditioning of excessive acid soil reaction. Most heavy metals availability is impacted by soil pH. Liming is also widely recommended strategy to reduce mobility and plant availability of soil contaminants such as cadmium (Smolders et al., 1999). For elements such as Pb and Cd it has been shown that large quantities in soils and consequently in food chain produce toxic effects because they tend to accumulate in body, vital organs and tissues (Lončarić et al., 2012). Consequently, heavy metals toxicity is one of the main forms of abiotic stress that may cause harmful effects to plant and animal health (Lisjak et al., 2008). So, the aim of this paper was to determine the influence of acid soils liming and initial soil acidity as well as organic matter content on availability of four heavy metals (Zn, Pb, Cr and Cd).

MATERIAL AND METHODS

Liming experiment was conducted in laboratory conditions with six soils of different acidity in four replications. Six acid soils were characterized by different levels of acidity and different organic matter content. Soil samples were divided according to exchangeable acidity (pH (KCl)) treshold values into three categories with two soils in each category. First set of soils were extremely acid dystric luvisol and dystric gleysol in range of pH (KCl) < 4, followed by heavily acid dystric luvisol and dystric gleysol in range of pH (KCl) 4 − 5 and moderately acid dystric luvisol and stagnosol in range of pH (KCl) 5 − 6. Threshold value of soil organic matter content was 2 %, dividing acid soil samples into two main groups. Lime requirement calculations were determined according to target soil pH (KCl) 7.0 for each soil set. Liming was conducted with hydrated lime (Ca(OH)$_2$) in range of 10.94 − 12.33 t/ha (1.22-1.37 g/kg)

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for extremely acid soils, 6.79 – 10.51 t/ha (0.75-1.17 g/kg) for heavily acid soils and 6.27 – 7.59 t/ha (0.70-0.84 g/kg) for moderately acid soils. Mass of 1 kg of all experimental soils were used for liming experiment in a rapid incubation method conducted in sealed containers for three days at a constant temperature of 60 °C (Barrow and Cox, 1990). Each experimental treatment (control and liming) was applied in four replications. After three days of incubation, soils were extracted with EDTA extraction solution (mixture of 1 M $(NH_4)_2CO_3$ and 0.01 M EDTA) (Jones, 2001) and digested with aqua regia (ISO 11466) in order to determine available heavy metals concentration and total heavy metals content in soil. Essential and potentially toxic heavy metals (Zn, Cr, Pb, Cd) were determined by inductively coupled plasma optical emission spectrometer (ICP-OS). In addition, the basic chemical properties were analyzed such as soil pH (ISO 10390) and soil organic matter content by sulfochromic oxidation (ISO 14235).

RESULTS AND DISCUSSION

Liming impact on soil reaction and heavy metals status in soil

Liming treatments significantly increased soil reaction. According to all samples mean, actual acidity (pH (H_2O)) increment was 1.89 pH units and exchangeable acidity (pH (KCl)) increment was 2.35 pH units, with 4.73 cmol/kg of hydrolytic acidity neutralised by liming (Table 1). Kovačević and Rastija (2010) reported that application of 15 t/ha dolomite raised pH(KCl) value by 2.62 units. The effect of liming on soil pH increment was proportional to liming material doses, where liming rates were adequately higher as initial soil pH was lower because all soils were limed in order to reach the same target pH. In category of extremely acid soils (Table 2), the highest liming dose resulted in the highest average soil pH increment of 2.56 pH units for actual acidity and 3.01 pH units for exchangeable acidity because liming neutralised the highest value of hydrolytic acidity 5.63 cmol/kg. Increment of soil pH in category of heavily acid soils was less and amounted 1.82 pH units for actual acidity and 2.42 pH units for exchangeable acidity, where 4.50 cmol/kg of hydrolitic acidity was neutralised. The lowest soil pH change was recorded in moderately acid soils category where soil pH increment was 1.30 pH units for actual acidity and 1.62 pH units for exchangeable acidity with the lowest neutralised value of hydrolytic acidity 4.06 cmol/kg.

Table1. Mean values of soil acidity, humus content, total and plant available heavy metals concentration in analysed soils

Tablica 1. Prosječne vrijednosti kiselosti tla, sadržaja humusa, ukupnih i biljkama pristupačnih koncentracija teških metala u analiziranim tlima

Treatment *Tretman*	pH_{H2O}	pH_{KCl}	Humus %	Hy cmol/kg	Total content (mg/kg) *Ukupan sadržaj (mg/kg)*				Available concentration (mg/kg) *Pristupačna koncentracija (mg/kg)*			
					Zn	Pb	Cr	Cd	Zn	Pb	Cr	Cd
Control	5.83b	4.45b	2.02	5.10a	51.38	11.50	37.14	0.227	2.31	2.44a	0.168a	0.082
Liming	7.72a	6.80a	1.93	0.37b	50.33	11.80	37.26	0.214	1.73	1.73b	0.103b	0.070

Considering average total content of heavy metals, similar results were determined on control and liming treatments for all of investigated soils. As it was expected, liming did not have impact on statistically significant change of total heavy metals content in soil compared to control treatment because additional quantities of heavy metals were not added with liming treatments. Comparing average total content and plant available heavy metals concentrations, significantly lower concentrations were recorded for available heavy metals. Lime application significantly decreased average concentrations of available Pb and Cr in all soils comparing to control treatment. Liming also resulted in trend of availability decrement for Zn and Cd, but without statistical significance. So in this way liming resulted in decreased extractability of available fraction of examined essential and potentially toxic heavy metals Zn, Pb, Cr, Cd in analysed soils (Table 1). Kovačević et al. (2009) reported that on field liming experiment mobile Zn fraction was decreased from 1.52 mg/kg (control) to 0.64 mg/kg (90 t/ha of sugar waste liming material with 34.4 % Ca). Popović et al. (2009) found that application of 10 t/ha dolomite decreased Zn concentration for 30 % compared to the control and by increasing lime rates it was decreased additionally by 7 %.

Impact of initial soil reaction and liming on heavy metals status in soil

In the analyzed soils, considering all three categories of soil acidity, the highest total content was recorded for Zn, followed by Cr, Pb content and Cd content being the lowest one (Table 2). Order of available heavy metals concentrations was different compared to order of total heavy metals content. In all the three categories of soil acidity, the highest concentrations were determined for plant available Zn and Pb, low concentrations of available Cr and the lowest concentrations of available Cd were recorded (Table 2). The highest initial concentrations of available Zn, Pb and Cr were measured

in extremely acid soils and the lowest in moderately acid soils. The initial concentrations of available Pb were almost identical in heavily and moderately acid soils. The highest concentrations of available Cd were determined in moderately acid soils and almost identical low concentrations in heavily and extremely acid soils. Liming treatments resulted in trend of availability decrement for Zn, Pb, Cr and Cd in all soils. However, intensity of liming impact to heavy metals decrement differed considering initial soil acidity and initial heavy metals availability.

Table 2. Soil reaction, total and available heavy metals concentrations mean according to soil acidity

Tablica 2. Reakcija tla, prosječne koncentracije ukupnih i pristupačnih teških metala prema kiselosti tla

Treatment *Tretman*	pH_{H2O}	pH_{KCl}	Humus %	Hy cmol/kg	Total content (mg/kg) *Ukupan sadržaj (mg/kg)*				Available concentration (mg/kg) *Pristupačna koncentracija (mg/kg)*			
					Zn	Pb	Cr	Cd	Zn	Pb	Cr	Cd
Extremely acid soils (pH_{KCl} <4)												
Control	5.21b	3.82b	1.76	6.32a	48.04	11.55	32.89	0.223	3.09	2.58a	0.205a	0.067a
Liming	7.77a	6.83a	1.60	0.69b	47.22	12.35	32.69	0.202	2.02	1.45b	0.103b	0.054b
Heavily acid soils (pH_{KCl} 4-5)												
Control	5.80b	4.32b	1.96	4.92a	52.53	11.86	34.89	0.199	2.30	2.37a	0.167a	0.066
Liming	7.62a	6.74a	1.93	0.42b	50.39	11.76	37.03	0.187	1.70	1.70b	0.110b	0.060
Moderately acid soils (pH_{KCl} 5-6)												
Control	6.48b	5.22b	2.35	4.06a	53.58	11.09	43.63	0.260	1.55	2.38	0.133	0.111
Liming	7.78a	6.84a	2.26	0.00b	53.39	11.29	42.07	0.252	1.47	2.03	0.095	0.097

As it was expected, soil pH change by liming did not have a significant influence on total heavy metals content through soil acidity categories. Impact of soil pH change by liming was determined for plant available heavy metals extracted by EDTA considering three different categories of soil acidity. Portion of EDTA extractable heavy metals in total heavy metals content represents relative portion of available fraction in total content. According to relative portions of available heavy metals in total content, Zn availability was decreased by liming in extremely acid soils from 6.44% to 4.29%, in heavily acid soils from 4.38% to 3.38% and from 2.89% to 2.75% in moderately acid soils. Liming also decreased Pb availability in extremely acid soils from 22.34% to 11.77%, in heavily acid soils Pb availability was decreased from 19.94% to 14.45% and in moderately acid soils from 21.45% to 17.94%. Decrement of Cr availability under impact of liming was from 0.62% to 0.32%, from 0.48% to 0.30% in heavily acid soils and from 0.30% to 0.23% in moderately acid soils. In extremely acid soils liming decreased Cd availability from 30.30% to 26.77%, in heavily acid soils from 33.30% to 32.02% and in moderately acid soils from 42.88% to 38.26%. So, liming impact on heavy metals availability decrement was different among three sets of soils with different initial soil acidity. In extremely acid soils, availability was significantly decreased just for Pb from 22.34% to 11.77%. Significant decrement in heavily acid soils was recorded for Pb from 19.94% to 14.45% and for Cr from 0.48% to 0.30%. In moderately acid soils decrement of analyzed heavy metals was not statistically significant. Chaudhary et al. (2011) found out that plant available Cd concentrations, after 10 years application of phosphate fertilizers containing Cd, were significantly higher at pH 5.0 compared to pH 6.0, and this was reflected in the increased Cd uptake by wheat at the lower pH.
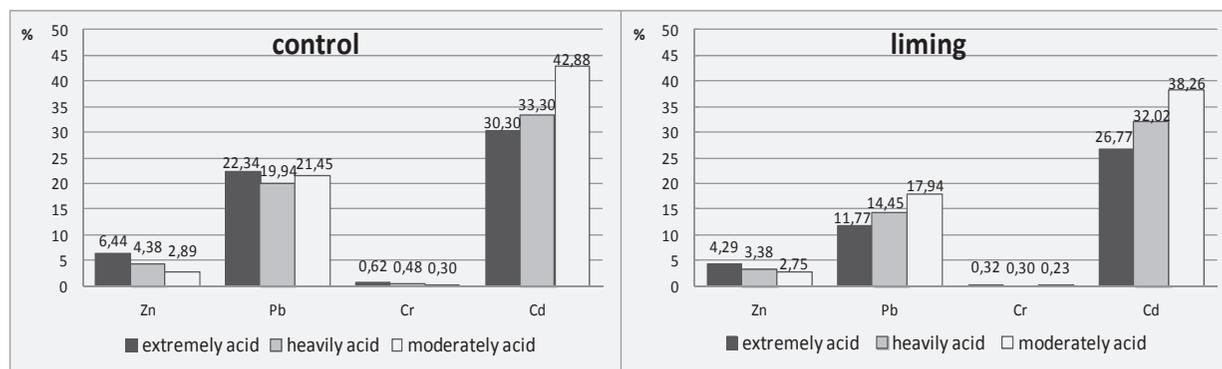
Graph 1. Relative portion of plant available heavy metals fraction (%) in total heavy metals content in soil

Grafikon 1. Relativni udjel frakcije biljkama pristupačnih teških metala (%) u ukupnome sadržaju teških metala u tlu

The lowest average portion of available heavy metals in total content was determined on the control treatment for Cr 0.50%, then for Zn 4.82%, considerably higher average portion was found for Pb 21.36%, and the highest was determined for Cd 35.70%, respectively. Average portion of available heavy metals on limed soils were significantly lower compared to control treatment, 0.30% for Cr, 3.61% for Zn and considerably higher for Pb 14.79% and for Cd 34.33%, respectively. Lehoczky et al (1998) reported that 42–44% and 64–67% of the total Zn and Cd soil content was determined in the Lakanen-Ervio extract, which is considered easy available for the plants. Consequently, the lowest total content in the soil was measured for Cd as potentially toxic heavy metal,

but Cd at the same time resulted in the highest plant availability. Also, in despite of low Pb total content in soil, lower than Zn and Cr, Pb resulted in higher plant availability compared to Zn and Cr.

Soil pH increase as a result of liming impacted with different intensity on change of available heavy metals portion in total content. Liming treatment in general resulted in lower relative portion of analyzed heavy metals available fractions in total content compared to control treatment, but with different efficiency. Pb and Cr relative portion were significantly decreased, but differences for Zn and Cd were not statistically significant.

Table 3. Absolute and relative decrement of heavy metals availability by liming considering soil acidity

Tablica 3. Apsolutno i relativno smanjenje pristupačnosti teških metala kalcizacijom, s obzirom na kiselost tla

Soil acidity *Kiselost tla*	Zn	Pb	Cr	Cd	Zn	Pb	Cr	Cd
	(mg/kg)				(%)			
Extremely acid	1.07	1.13	0.102	0.013	34.56	43.69	49.66	19.77
Heavily acid	0.60	0.67	0.056	0.006	26.01	28.20	33.78	9.67
Moderately acid	0.08	0.35	0.038	0.015	5.03	14.84	28.23	13.35

Liming impacted on heavy metals availability decrement, but decrement effect differed among analysed heavy metals considering initial soil acidity and initial EDTA concentrations. The strongest liming effect on heavy metals availability decrement was determined for Cr (49.66%), then Pb (43.69%), weaker impact was recorded for Zn (34.56%) and the weakest was for Cd (19.77%) in extremely acid soils with the highest initial concentrations of available heavy metals. In heavily acid soils with middle level of initial concentrations of available heavy metals, the same order of liming effect on availability decrement was recorded for analyzed heavy metals, where decrement for Cr was 33.78%, then for Pb 28.20%, for Zn 26.01% and for Cd 9.67%. Liming effect on availability decrement was different in moderately acid soils with the lowest initial availability of analyzed heavy metals, where the strongest effect was determined for Cr (28.23%) then Pb (14.84%), Cd (13.55%) and the weakest for Zn (5.03%) (Table 3). Smolders et al. (1999.) reported that most Cd sorption models predict a 4–6 fold decrease in metal activities in soil solution per unit pH increase. Soil pH increase by liming resulted in the strongest intensity of liming effect on heavy metals availability decrement in extremely acid soils with the highest initial concentrations of available heavy metals. In the opposite, the weakest effect of liming on heavy metals availability decrement was recorded in moderately acid soils with the lowest initial concentrations of available heavy metals.

Impact of initial humus content and liming on heavy metals status in soil

Analysed soils were also divided in two sets according to humus content threshold value of 2%. Soil acidity neutralization by liming was more intensive in soils with

higher humus content (>2%) because liming neutralised higher value of hydrolytic acidity 5.55 cmol/kg, compared to soils with lower level of humus content (<2%) where liming neutralised 4.31 cmol/kg, although lime application resulted in lower soil pH change in soils with higher soil organic matter content. Liming did not result in significant humus content decrement in both sets of soils. In soils with less than 2% of humus content, lower initial concentrations of total heavy metals and higher available Zn, Pb and Cr on control treatment were detected compared to soils with higher level of humus content. Higher relative portion of available heavy metals Zn, Pb, Cr and Cd in total content was recorded in soils with lower soil organic matter content on liming treatment in comparison with higher soil organic matter content soils. Liming decreased Zn availability in soils with lower humus content from 5.81% to 4.46% and in higher humus content soils from 2.44% to 1.84%. Liming also decreased Pb availability from 22.18 % to 15.33% in soils with lower level of humus and in soils with higher humus level from 19.27% to 13.21%. Decrement of Cr availability by liming was from 0.58% to 0.35% in soils with lower humus content and from 0.28% to 0.17% in soils of higher humus content. In soils with higher humus level liming decreased Cd availability from 34.45% to 32.99% and in soils with lower humus level from 38.40% to 32.28% (Table 4).

Table 4. Soil reaction, total and plant available heavy metals concentrations mean according to soil organic matter content

Tablica 4. Reakcija tla, prosječne koncentracije ukupnih i biljkama pristupačnih teških metala, s obzirom na sadržaj humusa u tlu

Treatment Tretman	Soil acidity Kiselost tla		Hy cmol/kg	Humus(%)	Total content (mg/kg) Ukupni sadržaj (mg/kg)				Available (EDTA) concentration (mg/kg) Pristupačna (EDTA) koncentracija (mg/kg)			
	pH$_{H2O}$	pH$_{KCl}$			Zn	Pb	Cr	Cd	Zn	Pb	Cr	Cd
	Humus < 2%											
Control	5.72b	4.32b	4.66 a	1.71	46.97	11.36	31.46	0.209	2.73	2.52a	0.183a	0.072
Liming	7.75a	6.85a	0.35 b	1.64	46.45	11.87	32.51	0.194	2.07	1.82b	0.115b	0.064
	Humus > 2%											
Control	6.03b	4.71b	5.97 a	2.64	60.21	11.78	48.49	0.263	1.47	2.27a	0.138a	0.101
Liming	7.65a	6.69a	0.42 b	2.50	58.09	11.66	46.77	0.254	1.07	1.54b	0.078b	0.082

Therefore, significant impact of liming on all analyzed heavy metals availability decrement was detected for each set of soils considering humus content. Sklodowski et al. (2006) explained that enrichment of soil with organic matter could reduce the content of bioavailable metal as a result of complexation of free ions of heavy metals. Zaniewicz-Bajkowska et al. (2007) reported that organic fertilization, especially farmyard manure and straw, significantly reduced the soil available cadmium content as compared to the untreated control in field experiment.

Order of heavy metals availability decrement was in accordance with liming efficiency with the strongest effect on Cr, then Pb, weaker impact was recorded for Zn and the weakest was for Cd in both sets of soils regardless initial concentrations of available heavy metals (Table 5).

Table 5. Absolute and relative decrement of heavy metals availability by liming considering humus content

Tablica 5. Apsolutno i relativno smanjenje pristupačnosti teških metala kalcizacijom, s obzirom na sadržaj humusa

Humus %	Zn	Pb	Cr	Cd	Zn	Pb	Cr	Cd
	(mg/kg)				(%)			
< 2	0.66	0.70	0.068	0.008	24.18	27.78	37.16	11.11
> 2	0.40	0.73	0.060	0.019	27.21	32.16	43.48	18.81

Liming resulted in higher relative decrement of analyzed heavy metals availability in soils with higher humus content (> 2 %). Therefore, higher relative liming efficiency of heavy metals immobilization was determined in soils with higher soil organic matter content for Zn, Pb, Cr and Cd (Table 5) with lower initial concentrations of available heavy metals.

Furthermore, regression calculation for prediction of heavy metals availability was conducted based on variables that included total heavy metals concentration (mg/kg), soil pH land humus content (%). Regression calculation for prediction of EDTA extractable heavy metals concentration was statistically significant for Zn (r^2=0.782, n=6) and for Cr (r^2=0.864, n=6) with the following regression equations:

EDTA-Zn (mg/kg) = 2.356 + 0.154 x total Zn conc. (mg/kg) – 0.294 x pH - 3.307 x humus (%)

EDTA-Cr (mg/kg) = 0.371 + 0.0016 x total Cr conc. (mg/kg) – 0.0304 x pH – 0.064 x humus (%)

Lončarić et al. (2008) reported that humus content and pH could be sufficient for available micronutrients prediction in acid soils using regression formulas.

CONCLUSION

Expectedly, the highest soil acidity was neutralized by liming in extremely acid soils, then in heavily acid soils and the lowest in moderately acid soils in order to reach the same target soil pH. The lowest total content in soil was measured for Cd, but Cd at the same time resulted in the highest plant availability. Despite of low Pb total content in soil, lower than total Zn and Cr, Pb resulted in higher availability compared to Zn and Cr. Liming did not result in significant impact on change of total heavy metals content in the analyzed soils. In the opposite, liming treatments resulted in trend of heavy metals availability decrement in all soils, but intensity of decrement differed considering initial soil acidity and initial heavy metals availability. Considering impact of initial soil pH, in extremely acid soils availability was significantly decreased just for Pb, in heavily acid soils significant decrement was recorded for Pb and Cr. In moderately acid soils decrement of four heavy metals was not significant. According to relative heavy metals availability decrement, liming resulted in the strongest effect in extremely acid soils with the highest initial concentrations of available heavy metals. In the opposite, the weakest effect of liming on relative heavy metals availability decrement was recorded in moderately acid soils with the lowest initial concentrations of available heavy metals. Considering impact of initial humus content in soil, liming significantly decreased availability of all analyzed heavy metals in both sets of soils with lower and higher humus content. Higher relative liming

efficiency of heavy metals availability decrement was determined in soils with higher soil organic matter content and with lower initial concentrations of available heavy metals. Regression calculation for prediction of EDTA extractable heavy metals based on total concentration of analyzed heavy metals (mg/kg), soil pH and humus content (%) were significant for Zn and Cr.

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UTJECAJ KALCIZACIJE NA PRISTUPAČNOST TEŠKIH METALA U TLU

SAŽETAK

Cilj je rada bio utvrditi učinak kalcizacije kiselih tala na promjenu pristupačnosti teških metala Zn, Cr, Pb i Cd, s obzirom na različitu kiselost tla i sadržaj organske tvari. Pokus kalcizacije proveden je na šest eksperimentalnih tala različite kiselosti u četiri ponavljanja, uz inkubaciju u zatvorenim posudama tijekom tri dana, uz konstantnu temperaturu od 60°C. Tretmani kalcizacije rezultirali su trendom smanjenja pristupačnosti teških metala u analiziranim tlima, ali se intenzitet smanjenja razlikovao ovisno o početnoj kiselosti tla te inicijalnoj pristupačnosti teških metala. Na temelju relativnoga smanjenja pristupačnosti teških metala, najveći učinak kalcizacije zabilježen je u ekstremno kiselim tlima, s najvećom inicijalnom koncentracijom pristupačnog Zn, Pb, Cr i Cd. Nasuprot tome, najslabiji relativni učinak kalcizacije utvrđen je u umjereno kiselim tlima s najnižom početnom razinom pristupačnosti teških metala. S obzirom na početni sadržaj humusa u tlu, veći učinak kalcizacije na relativno smanjenje pristupačnosti Zn, Pb, Cr i Cd, utvrđen je na tlima s višim sadržajem humusa i nižom početnom razinom pristupačnosti teških metala.

Ključne riječi: kisela tla, kalcizacija, pristupačnost teških metala, ukupne koncentracije teških metala