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The Heavy Metal Status of Some Agricultural Soils

ALEXANDRINA MANEA[1], NICOLETA VRINCEANU[1], CARMEN-ALINA EFTENE[1*], DANIELA RADUCU[1]
National Research and Development Institute for Soil Science, Agro-Chemistry and Environment – ICPA Bucharest, 61 Marasti Blvd., 73 331, Bucharest, Romania

Abstract. *In order to evaluate the loadings of some agricultural soils with heavy metals, field studies were made across the Iasi County. The heavy metals concentration range were as follows: 15.4 - 36 mg/kg (Cu), 49 - 115 mg/kg (Zn), 7.4 - 20.4 mg/kg (Pb), 0.13 - 0.43 mg/kg (Cd), 6.9 - 13.15 mg/kg (Co), 25 - 100 mg/kg (Ni) and 463 - 798 mg/kg (Mn), respectively. The median of the heavy metals decreased in the next order: Mn (589) > Zn (67) > Ni (46) >Cu (22,1)>Pb (12.1)>Co (10.2)>Cd (0.32). The studied heavy metal median was higher than the same metal median from European soils. According to the Romanian legislation, the normal values are exceeding in 13% the samples for Zn, 73% for Cu, 93% for Ni, and in one case, the value of Ni exceeded the alert threshold (75 mg/kg). A direct relationship between Cu, Pb, Zn and Co was noticed, which may indicate a common source of these metals. The Pearson's coefficients of these elements were: Pb-Cu (0.863), Cu-Zn (0.826), Pb-Zn (0.74), Cu-Co (0.730), Pb-Co (0.703). According to the values of Pearson correlation coefficient, the significant positive correlations were identified between Cu, Zn, Pb, Co and clay content, while the same metals are highly negative correlated with* $CaCO_3$ *and fine sand contents. In case of Pb, Co, and Mn no influence of agricultural activities can be detected, wich suggestes that geogenic factors control the contents of these heavy metals. The Cu content exhibited a positive correlation with total Nitrogen, while the Cd content showed a positive significant relationship only with Km content. On the other hand, the Ni content exhibited correlation with both Pm and Km concentrations. The correlations of Cu-Nt, Cd-Km, Ni-Pm, and Ni-Km could be an index of the anthropogenic input of Cd, Cu, and Ni from the use of fertilizers.*

Keywords**:** *heavy metals, soil properties, agriculture*

1.Introduction

The quality of human life depends of the quality of the food, which is determined by the quality of the soil. The heavy metals soil contamination was identified as one of the restrictions for the soils of Europe [1]. Initially, the soil heavy metal contents are inherited from rocks through geochemical and pedochemical alteration of the materials that form the soil [2]. High levels of heavy metals can have natural or anthropic sources, but in most cases it is associated with human activity, which determined the accumulation of contaminants at high concentrations that can be a risk to the environment as well as to humans [3]. The application of the sewage sludge, manure, mineral fertilizers, fungicides, pesticides are known as sources of agricultural soil pollution with heavy metals [4- 7]. Due to long time application of mineral fertilizers, pesticides, fungicides, large area of land are contaminated [8]. There are differences between crop farms and animal farms according to the inputs of heavy metals to soils, because crop farms generate higher inputs compare with animal farms [9]. Over 50% of the heavy metal input (Zn, Mo, Ni, As, Cu, Hg) in agricultural soils from France are due to livestock manure application [10]. Also, livestock manure is an important contributor of heavy metals in farm soils from England and Wales and account for 35-40% of Zn, 8-17 of Cu [4]. Moreover, phosphate, nitrogen and potassium fertilizers are significant sources of Cd, Ni, Cu, and Pb [11].

High levels of soil contaminants have a negative effect on food security by decreasing the cropped area, crop diversity [12], crop production and by the production of unsafe crops for the humans and animals [3]. Soil quality assessment and monitoring are key issues for agricultural policy and environ-

**email: c.eftene@gmail.com*

-mental resources management [13]. For a sustainable use of soil and an environment protection policy, a soil quality monitoring system has been implemented [14]. However, among the studied indicators were also the selected heavy metals.

In this paper, the status of the selected heavy metals in some agricultural soils of Iasi County and the influence of agricultural practices such as mineral fertilization on the heavy metal contents were evaluated.

2. Materials and methods

Field studies were made across the Iasi County. The soils were sampled within a network (16 x16 km) according to the methodology presented in Order MADR 223/2002 [14]. Within the field stage, 15 soils have been studied. *Location of samples* was done according to the geographic (latitude, longitude) coordinates using a Global Positioning System (GPS). The soil samples were collected within an area of 400 m^2 (20x20 m) around the soil profile, from 0-20 cm depth. The sampling was performed with stainless-steel materials to avoid contamination. The samples were stored in tightly sealed plastic bags and properly numbered. All soil samples were air-dried, milled and sieved to 2 mm fractions to remove stones and other coarse fragments.

The pseudo-total concentrations of Cd, Co, Cu, Mn, Ni, Pb and Zn were determined using atomic absorption spectrometry after extraction by the *aqua regia* (*HCl:HNO₃ – 3:1*) – microwave digestion method. Microwave digestion was performed using 10 mL of *aqua regia* at 140°C for 30 min. A certified soil reference material (ERM–CC141) was used to ensure the accuracy of the analytical data. In addition, other parameters such as: particle size distribution, *p*H, $CaCO_3$ content, Sum of exchangeable cations (SB), Total exchange acidity (SH), Organic carbon content, total Nitrogen (Nt), availabale P and K (Pm, Km) (Table 1) were measured to determine their influence on the studied heavy metal contents.

Soil diagnosis was done in accordance with the Romanian Soil Taxonomy System-2012 [15] and WRB-SR-2014 [16]. In studied area, the identified soils according to WRB-SR-2014 were: Fluvisols (FL), Regosols (RS), Chernozems (CZ), Eutric Cambisols (EC), Haplic Luvisols (EL), Gleysols (GS).

The heavy metal assessment was made according to Romanian legislation [17]. Descriptive statistics including minimum, maximum, mean, median, standard deviation, variation coefficient were calculated for soil samples.

Table 1. The methods used for studied soil parameters determination

Soil parameters	***Method***
Particle size distribution	Romanian Standard STAS 7184/10-79 based on the wet and dry sieving, sedimentation procedure, pipette sampling, followed by chemical treatment with different dispersant agents (H_2O_2, HCl, $Na_4P_2O_7 \cdot 10H_2O$) according to organic matter and carbonate contents [18].
pH_{H2O}	Romanian Standard STAS 7184-13:2001 based on the Potentiometric method (1:2.5 w/v, soil : water).
$CaCO_3$	Romanian Standard STAS 7184/16-80 based on the Scheibler method.
Sum of exchangeable cations (SB)	Romanian Standard STAS 7184/12-88 based on the Kappen method (extraction of basic cations in 0.1N HCl solution).
Total exchange acidity (SH)	Romanian Standard STAS 7184/12-88 based on the percolation until exhaustion with a solution of Potassium acetate solution 1 N.
Degree of Base Saturation ($V_{pH8.3}$)	Calculation: $V_{pH8.3}$= SB/(SB+SH)*100.
Organic carbon content	Romanian Standard STAS 7184/ 21-82 based on the method Walkley-Black modified by Gogoașă.
Humus (Ht)	Calculation: the organic carbon content was multiplied by a conversion factor of 1,724.

Total Nitrogen (Nt),	Romanian Standard STAS 7184/2-85 based on the Kjeldahl method.
Available P	Romanian Standard STAS 7184/19-82 based on the Egner-Riehm-Domingo method.
Available K	Romanian Standard STAS 7184/18-80 based on the Egner-Riehm-Domingo method.

3.Results and discussions

On the studied area, the concentration range of Cu, Zn, Pb, Cd, Mn, Co, and Ni were: 15.4-36 mg/kg, 49-115 mg/kg, 7.4-20.4 mg/kg, 0.13-0.43 mg/kg, 463-798 mg/kg, 6.9-13.15 mg/kg, 25-100 mg/kg, with median values of 22.1 mg/kg, 67 mg/kg, 12.1 mg/kg, 0.32 mg/kg, 589 mg/kg, 10.2 mg/kg, and 46 mg/kg (table 2). The mean concentration of the heavy metals decreased in the next order: Mn (594) > Zn (76) > Ni (48.7) >Cu (24)>Pb (12.7)>Co (10.2)>Cd (0.3).

The Copper content lied between 15.4 mg/kg and 36 mg/kg, the median (22 mg/kg) was higher than the median reported for Romanian agricultural soils [18] and the mean is similar with the values recorded in the West Region [19] and below of the values from North East Region [20]. The Cu median was higher than the median values reported for European soils (13 mg/kg) and for European agricultural soils (15 mg/kg) [21, 22]. Over 73% of the samples slightly exceed the normal content limit (20 g/kg). Minimum content of Cu was recorded in Fluvisols (15.4 mg/kg) and maximum value was noticed in Gleysols (36 mg/kg) (Figure 1).

Table 2. The basic statistics of studied heavy metals (mg/kg) and the thresholds according to national legislation

	Cu	**Zn**	**Pb**	**Cd**	**Mn**	**Co**	**Ni**
Minimum	15.4	49	7.4	0.13	463	6.9	25
Mean	**24.0**	76	12.7	0.3	594	10.2	**48.7**
Median	22.1	67	12.1	0.32	589	10.2	46
Maximum	**36**	**115**	20.4	0.43	798	13.2	**100**
Standard deviation	6.2	21.4	3.2	0.10	83.3	2	19
Variation coefficient	25.7	28.2	24.8	28.7	14	19.9	38.7
Normal values*	20	100	20	1	900	15	20
Alert threshold*	100	300	50	3	1500	30	75
Intervation* threshold	200	600	100	5	2500	50	150

*) [16]

The Lead content ranged from 7.4 to 20.4 mg/kg and the mean (12.7 mg/kg) and median (12.1 mg/kg) were below the recorded values of country and regional levels [18-20]. Also, the Pb median (12.10 mg/kg) was lower than the median reported at the European levels [21, 22] Minimum content of Pb was recorded in Fluvisols (7.4 mg/kg) and maximum value was observed in Gleysols (20.4 mg/kg).

Total Zinc content ranged from 49 to 115 mg/kg, with an average of 76 mg/kg. Both the mean and the median values are below of the Romanian agricultural soil values [18-20]. The Zn median (67 mg/kg) was higher than the median reported for European soils (48 mg/kg) and for European agricultural soils (45 mg/kg). Most of the samples have normal Zn content (87 %) and only 13% of samples slightly exceed the normal limit (100 mg/kg). Minimum content of Zn was recorded in Eutric Cambisols (49 mg/kg) and the highest Zn value was recorded in Gleysols (115 mg/kg).

Total Cadmium content range between 0.13 and 0.43 mg/kg and the mean is 0.3 mg/kg. The average and median values are close to the founded values in the previous studied [18-20], but median was twice more than Cd median in European soil (0.15 mg/kg). Minimum content of Cd was recorded in Chernozems (0.13 mg/kg) and maximum value was observed in Haplic Luvisols (0.43 mg/kg).

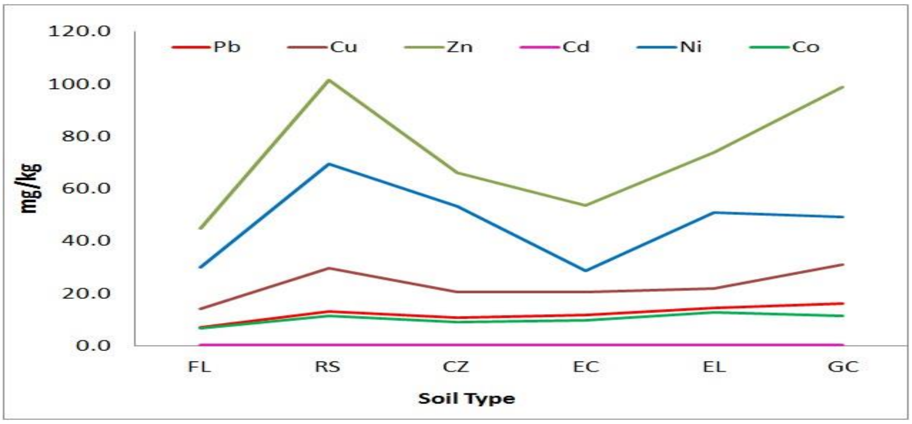

Figure 1. The mean content variations of studied heavy metals (mg/kg) with type of soils

Total Cobalt content lied between 6.9 and 13.15 mg/kg, with a mean of 10.2 mg/kg. The range, mean and median values are close to the values of previous results [18-20], but are higher than median of the European soils (7.5 mg/kg). Minimum content of Co was observed in Eutric Cambisols (6.9 mg/kg) and maximum value of Co was found in Gleysols (13.15 mg/kg).

Total Nickel content ranged from 25 and 100 mg/kg, with an average of 48.7 mg/kg. Both the mean and the median values are higher than the recorded values of the Romanian agricultural soils [18-20,23]. The Ni median (46 mg/kg) was three times higher than the median value reported for the European soils (14 mg/kg) and for the European agricultural soils (15 mg/kg). Most of the Ni values (93%) were between 20 mg/kg and 75 mg/kg, moreover one value exceeded the alert threshold for sensitive uses. Minimum content of Ni was recorded in Eutric Cambisols (25 mg/kg) and maximum value was noticed in Chernozems (36 mg/kg).

Mn total content varied from 463 to 798 mg/kg, with an average value of 593.5 mg/kg. The median value is higher compared with the recorded values in the Romanian and the European soils. Minimum content of Mn was recorded in Fluvisols (463 mg/kg) and maximum value was found in Eutric Cambisols (798 mg/kg).

Pearson correlation analysis

The correlation of soil heavy metals was evaluated by using the Pearson correlation matrix. The information on heavy metal sources were provide by “inter-element relationships’’ [24].

According to the Pearson correlation coefficient, a direct relationship between Cu, Pb, Zn, and Co was identified (Table 3). However, the correlation between heavy metals suggests similar physical and chemical factors that control the association of the elements in the parent material and during the soil formation processes [25].

Table 3. The heavy metal content correlation matrix of studied soils

	Pb	Cu	Zn	Cd	Mn	Ni	Co
Pb	1						
Cu	0.863***	1					
Zn	0.7398***	0.826***	1				
Cd	-0.01	-0.06	-0.03	1			
Mn	0.44	0.22	0.04	-0.1	1		
Ni	0.16	0.36	0.31	-0.3	-0.24	1	
Co	0.703***	0.727***	0.52	-0.2	0.565*	0.13	1.0

*Correlation is significant at the 0.05 level, ** Correlation is significant at the 0.01 level,
*** Correlation is significant at the 0.001 level

The Pearson's coefficients of these elements were: Pb-Cu (0.863), Cu-Zn (0.826), Pb-Zn (0.74), Cu-Co (0.727), Pb-Co (0.703). Also, direct relationships between Cu-Pb and Cu-Zn were reported in [26], but these correlations were low. However, the Mn content was correlated only with Co concentration (0.565), that is consistent with the result (0.537) obtained by other authors [13], while the contents of Cd and Ni exhibited no significant correlation with the rest of the metals, which indicate a different origins than Cu, Pb, Zn, and Co. Similarly, negative and no significant correlations between Cd and the other metals were noticed in other studies [27].

Correlation between heavy metals contents and the soil physicochemical properties

According to the Pearson correlation coefficients, a significant positive correlation was observed between Cu, Zn, Pb, and Co and clay content, while the same metals are highly negative correlated with $CaCO_3$ and fine sand contents (Table 4). The Pearson's coefficient of Cu, Zn, Pb, and Co with clay decreased in the next order: Cu-Clay (0. 935) > Zn-Clay (0.823) > Pb-Clay (0.782) > Co-Clay (0.668).

Table 4. Correlation coefficiens of heavy metal contents with some soil physical and chemical properties

Element	**Pb**	**Cu**	**Zn**	**Cd**	**Mn**	**Ni**	**Co**
pH	0.007	0.306	0.332	-0.08	-0.17	0.174	0.068
CaCO3	-0.899***	-0.868***	-0.729**	0.113	-0.41	-0.42	-0.522*
Ht	0.28	0.300	0.291	-0.24	0.170	-0.062	0.192
Nt	0.37	0.521*	0.418	-0.38	0.011	0.195	0.423
Pm	-0.173	-0.057	-0.085	-0.13	-0.11	0.640*	-0.166
Km	-0.065	0.155	0.205	-0.583*	-0.52	0.542*	0.029
V8,3	-0.008	0.132	0.314	-0.08	-0.46	0.099	-0.206
Coarse sand	0.095	0.232	0.022	0.138	-0.14	0.435	0.057
Fine sand	-0.869***	-0.932***	-0.851***	0.037	-0.26	-0.20	-0.708**
Silt	0.259	-0.029	0.099	0.106	0.479	-0.277	0.124
Clay	0.782***	0.935***	0.823***	-0.08	0.104	0.272	0.668**

*Correlation is significant at the 0.05 level, ** Correlation is significant at the 0.01 level,
*** Correlation is significant at the 0.001 level

The strong correlations between Cu, Zn, Co with clay content were also observed in [27]. In addition to the correlation with clay, $CaCO_3$ and fine sand, Cu content exhibited a positive correlation (0.521) with total Nitrogen (Nt), the nitrogen fertilizer application being knowing as an important source of Cu in the soil [11].

However, the Cd content showed a relationship only with Km content, while Ni concentration exhibited correlation with both Pm and Km concentrations, which may suggest that high Ni contents may come from the use of fertilizers. Moreover, the potassium fertilizers were found to be important sources of Cd [11], while the high Ni concentrations in the topsoil are due to anthropogenic input, such as the diffuse use of P-fertilizers [11, 28]. Instead, Mn is the only elements which show no relationship with soil properties. The pattern of correlation of Cu, Pb, Zn and Co contents with clay and soil reaction is the same. However, it can be observed that contents of Cu, Pb, Zn, and Co increased in the field of loamy to clay soils with 73, 52, 57 and 72%, respectively (Table 5).

Table 5. The heavy metals mean variations with the particle size distribution

Texture class (Clay < 0.002 mm)	**Cd**	**Co**	**Cu**	**Mn**	**Ni**	**Pb**	**Zn**
	mg/kg						
Loam (21-32)	0.31	8.6	19.0	555.6	48.9	10.4	57.6
Clay loam (33-45)	0.32	10.2	22.6	645.2	41.0	12.6	78.8
Clay (≥46)	0.27	12.0	32.8	570.7	59.8	15.9	101.5

The studied heavy metal contents were not correlated with soil reaction and similar results were noticed [29, 30]. The variations of metals with soil *p*H highlight that the contents of Co, Pb, and Zn increased in the field of slightly acid to neutral soils (Table 6). In clay soils, the adsorption of lead, copper, chrome, and nickel ions increase with increasing *p*H [31]. In the case of the Cd and Mn contents were observed no variation with the particle size distribution and *p*H soil.

Table 6. The heavy metals mean variations ($mg \cdot kg^{-1}$) with to *p*H soil

pH	Cd	Co	Cu	Mn	Ni	Pb	Zn
	mg/kg						
5.9-6.4	0.36	6.9	17.3	580	25	11.1	49
6.5-6.8	0.3805	11.15	21.9	614	55	13.45	70
6.9-7.2	0.2375	11.75	27.25	610.5	51.5	15.05	84.5
7.3-7.8	0.2623	9.7	23.2	589.22	51.4	12	78.33
7.9-8.4	0.345	10.66	28.22	570.4	49.2	13.46	84.4

4.Conclusions

The metals concentration range were: 15.4-36 mg/kg (Cu), 49-115 mg/kg (Zn), 7.4-20.4 mg/kg (Pb), 0.13-0.43 mg/kg (Cd), 6.9-13.15 mg/kg (Co), 25-100 mg/kg (Ni), and, respectively, 463-798mg/kg (Mn). According to the Romanian legislation, the normal values were exceeded in 73% of the sites for Cu; 93% for Ni and 13% Zn. In one sample, the value of Ni exceeded the alert threshold (75 mg/kg). The studied heavy metal median was higher than the same metal median from European soils and the median of Cd and Ni was twice more and three times higher, respectively, than the median reported for the European soils.

A direct relationship between Cu, Pb, Zn and Co was found, which may indicate a common source of these metals. The Pearson's coefficients of these elements were: Pb-Cu (0.863), Cu-Zn (0.826), Pb-Zn (0.74), Cu-Co (0.727), Pb-Co (0.703). The correlation coefficients of Cd and Ni with the other studied metals were not observed, which indicates a different origin.

According to the values of Pearson correlation coefficient, the significant positive correlations were found between Cu, Zn, Pb, Co and clay content, while the same metals are highly negative correlated with both $CaCO_3$ and fine sand contents. The highest values of Cu, Pb, Co, and Zn were found in Gleysols, these soils being known as having a fine texture.

In case of Pb, Co, Mn no influence of agricultural activities can be detected, wich suggestes that geogenic factors control the contents of these heavy metals.

The Cu content exhibited a positive correlation with total Nitrogen, while the Cd content showed a relationship only with Km content. On the other hand, the Ni content exhibited correlation with both Pm and Km concentrations. The correlations of Cu-Nt, Cd-Km, Ni-Pm, and Ni-Km could be an index of the anthropogenic input of Cd, Cu, Ni from the use of fertilizers.

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