

DEPTH DISTRIBUTION OF AVAILABLE MICRONUTRIENTS IN CULTIVATED SOIL

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Abstract: This paper presents a study of the profile distribution of available micronutrients Fe, Mn, Cu and Zn and radionuclide ¹³⁷Cs in cultivated soil at the experimental field “Radmilovac” (property of Agricultural Faculty, Belgrade University) in the vicinity of Vinča Institute of Nuclear Sciences. The soil belongs to the anthrosol class of anthropogenic soils according to FAO (2006). At first, the deep plowing was performed while preparing soil for planting peach trees followed by cultivation of soil for 12 years. All agricultural treatments at the experimental field ceased for three years. After that period, soil sampling was carried out. Contents of DTPA-extracted Fe, Mn, Cu and Zn were in the range of (mg kg⁻¹): 5.8–41.6; 9.2–34.2; 1–7.6 and 0.2–1.3, respectively. Detected activity concentration (Bq kg⁻¹) for ¹³⁷Cs ranged from 1.8 to 35. It was noticed that distribution patterns of ¹³⁷Cs radionuclide and available Cu and Zn along soil depth were very similar and they were analyzed by simple linear regression; mutual affinity for the soil organic matter might affect their distribution in soil. Contents of available Fe and Mn exhibited different, more constant distribution within a soil horizon.

Key words: cultivated soil, ¹³⁷Cs, available Fe, Mn, Cu and Zn.

Introduction

Trace elements and heavy metals occur naturally in the earth's crust, and their content is resulting in spatial variations of background concentrations (Bowen, 1979). Heavy metals enter the soil by natural processes (like mineral weathering of parent material) and by different anthropogenic processes, one of them including a direct application of phosphate fertilizers to soil (Alloway, 2013). This is an important source of heavy metals in soil and its continuous use is connected with

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the tendency of metals to bioaccumulate. Generally, heavy metals might be included as impurities in minerals and natural materials, which is why they can be present in phosphate fertilizers or other chemical fertilizers. In production process of phosphate fertilizers, one of the key factors on deciding the quality of phosphate resources is heavy metal content, which does not have any standard permissible limit because maximum allowable content depends on soil characteristics, irrigation water quality, crop type, etc. (Aydin et al., 2010).

Heavy metals essential for plants such as zinc, copper, manganese and iron are the most important micronutrients required in a very small quantity for proper plant growth. Only the soluble, exchangeable, and chelated metal species in the soils are labile fractions available to plants, which is why it is accepted that the behaviour of micronutrients in soils cannot be assessed by measuring only their total concentration (Kabata-Pendias, 1993; Buccolieri et al., 2010). There is no universal extracting solution that can be used to estimate the micronutrient availability to plant because of the complexity of interactions between plant and environmental factors on the metal bioavailability in the soil system. Determined levels of microelements content and their critical levels (boundary values of available micronutrients below which deficiency is expected under the field conditions) in the soil depend on the extractant used and on the soil type and parent material (Alloway, 2013; Maqueda et al., 2011).

Soil properties are important factors influencing distribution of available Fe, Mn, Cu and Zn in cultivated soil (Yi et al., 2012; Belanović et al., 2012; Kumar et al., 2011; Milivojević et al., 2002). It was often confirmed that the DTPA extractable Cu, Zn, Mn and Fe were significantly and positively correlated among themselves and with soil organic matter (OM) content. The availability of micronutrients is increasing significantly with the increase of organic matter because it supplies soluble chelating agents which increase the solubility of micronutrients (Sharma et al., 2003). Maqueda et al. (2011) reported that higher levels of Cu extracted in the first 0–20-cm layer repeatedly fertilized are due to the formation of organo – Cu complexes and that soil OM has the effect of creating reducing conditions in soil with fine texture which will favour Mn solubilisation. Some authors concluded that main soil parameter which controls micronutrient availability in soil is organic carbon content (Bassirani et al., 2011; Yadov, 2011).

Anthropogenic radionuclide ^{137}Cs can be assumed to be derived from the radioactive fallout of nuclear fission products from Chernobyl accident in 1986 and from the global fallout from atmospheric weapon tests. ^{137}Cs has a physical half-life of 30.23 years but ecological half-life in organisms of food chain is generally longer (Larsson, 2008). It is known that ^{137}Cs will be immobilised in soils containing high quantity of clay minerals, free carbonates and small amounts of organic matter if pH values are between 4 and 7 (Koblinger-Bokori et al., 2000).

In undisturbed soil profiles, most of the cesium is concentrated up to 20 cm of the soil depth and this high activity at the upper part of the soil gradually decreases with soil depth (Fujiyoshi et al., 2004; Hrachowitz et al., 2005). Radiocesium in soil profiles from undisturbed locations one year after Fukushima nuclear accident was examined and the majority of ^{137}Cs remained in the upper 5 cm of soil layer and in the aboveground vegetation (or litter layer) (Matsunga et al., 2013) and its restricted migration was due to its strong affinities for humic substances and clay minerals (Tanaka et al., 2012). Similarly, in cultivated (disturbed) soil profiles, radiocesium is not uniformly distributed. If tillage operations are applied, soil is perturbed and radiocesium is mechanically redistributed. During cultivation, radiocesium would be distributed deeper (>20 cm) within the plow layer that would depend on the extent of soil mixing in the plow layer (Du et al., 1998, Soto and Navas, 2008). If tillage is performed regularly over several years, it will cause a uniform ^{137}Cs depth distribution down to the plow depth (Hrachowitz et al., 2005).

Mutual relationships between the total content of heavy metals and natural radioactive isotopes including fallout radiocesium in a soil were investigated in western Serbia (Dugalić et al., 2010) and in Belgrade (Dragović et al., 2012) and Zlatibor (Dragović et al., 2008) region. Strong positive correlations were found between ^{137}Cs and total soil Zn and these elements were both associated with the soil organic fraction. In agricultural soils of southern Italy, the most noticeable correlations were found between a pair of total soil Fe - Mn, which suggested that they may have a similar origin in soils probably simultaneously contaminated by nearby industries, and between a pair of total soil Cu - Zn which was attributed to the pollution from agricultural practices (Buccolieri et al., 2010).

Only a bioavailable fraction of a given trace element is important for plant uptake and this fraction is different in every soil layer along depth and it is strongly dependent on the soil properties. This work is a study towards determination of available Fe, Mn, Cu, Zn contents and activity concentration of radiocesium in cultivated soil around the fruit trees. The objective is to investigate: a) patterns of their depth distribution in soil and b) relationships among each other and with soil organic matter in order to better understand trace element behaviour within the soil environment where a significant intervention by humans has been encountered.

Materials and Methods

Soil sampling was carried out at the experimental field "Radmilovac" (property of Agricultural Faculty, Belgrade University), in the vicinity of Vinča Institute of Nuclear Sciences located near Belgrade. The soil of this field, according to FAO (2006) classification, belongs to the anthrosol class of anthropogenic soils. Soil used for growing peach trees is significantly altered by deep plowing before planting and then by annual plowing, irrigation and treatment with phosphate

mineral fertilizers for a period of 12 years. After a three-year pause, soil structure was sufficiently stable to determine the soil properties of the obtained anthrosol. The representative soil samples were collected from Ap horizon depth of 80 cm with a step of 20 cm (NRCS, 2004). Three soil profiles (P₁, P₂ and P₃) were taken from near the root of three randomly selected peach trees to examine if the distribution of ¹³⁷Cs was even across the plantation. The fourth profile (P₄) was taken from a soil area covered with grass. A detailed description of soil formation and soil properties can be found in our previous article (Vukašinović et al., 2009) and there has been reported that soil organic matter ranged from 0.46 to 2.80% and decreased by about 40% with the depth of the each soil profile.

Air-dry soil samples were crushed and sieved through a 2-mm sieve. Twenty grams of each sample were extracted with 40 ml of DTPA for 2 hours, using the 200 rpm shaker. Samples were filtered through a medium fine (Whatman No. 2) filter paper and analysed by atomic absorption spectrophotometry (AAS) method on Varian SpectrAA 250 plus (Lindsay and Norvell, 1978).

The measurement of ¹³⁷Cs activity concentration in soil samples was done by using HPGe detector (CANBERRA) with 20% relative efficiency. The detector was calibrated using a standard soil sample (MIX-OMH-SZ, National Office of Measures, Budapest) in the same geometry as measured samples (Marinelli beaker, 500 ml). The activity of ¹³⁷Cs was determined from 661.66 keV γ – line. Counting time was around 70,000 s and combined standard uncertainty was approximately 10%.

Results and Discussion

In the studied soil profiles (P₁, P₂, P₃ and P₄), the results of the measured available micronutrients and ¹³⁷Cs activity concentration at each of the 20-cm depth intervals are presented in Table 1. Results of radiocesium measurements were retrieved from our previous article (Vukašinović et al., 2013).

Contents of available Fe, Mn, Cu and Zn were in the range of (mg kg⁻¹): 5.8–41.6; 9.2–34.2; 1.0–7.6 and 0.2–1.3, respectively. The critical levels of contents (mg kg⁻¹) of DTPA extracted micronutrients are considered to be 4.5 for Fe; 2.0 for Mn; 0.2 for Cu and 0.6 for Zn (Lindsay and Norvell, 1978) and all soils indicate to be adequate in available micronutrients except critically low content of available Zn in the 40–80-cm soil layer. Considering the distribution of ¹³⁷Cs in the 20-cm depth intervals, radioisotope activities (Bq kg⁻¹) lie in the range of 1.8 to 35 and those are within the range expressed by regional levels for radiocesium (Popović et al., 2009).

It could be noticed that distribution of available Fe and Mn in soil profiles along 0–80-cm depth is more constant compared to available Cu and Zn. According to their coefficients of variation (CV) along profiles, the average

variability with depth of available Fe and Mn is 17% and 28%, respectively while for both available Cu and Zn, it is approximately ~ 60%. Variability of ^{137}Cs within the profiles is high (up to 80%) due to a considerable difference in the level of radiocesium contamination of particular soil layer. ^{137}Cs distribution patterns depended on the extent of soil mixing in the plow layer and during the period of cultivation radiocesium was distributed deeper in soil, up to ~ 60 cm (Vukašinić et al., 2013).

Table 1. Available (DTPA-extractable) micronutrients and ^{137}Cs activity concentration at 20-cm depth intervals of anthrosol soil.

Soil depth (cm)	Fe (mg kg ⁻¹)	Mn (mg kg ⁻¹)	Cu (mg kg ⁻¹)	Zn (mg kg ⁻¹)	^{137}Cs (Bq kg ⁻¹)
Profile 1 (P ₁)					
0–20	23.60	29.20	6.80	1.10	34 ± 3
20–40	19.80	15.60	5.80	0.90	29 ± 3
40–60	21.20	13.20	3.60	0.40	21 ± 2
60–80	22.50	13.40	1.80	0.30	4.9 ± 0.7
Profile 2 (P ₂)					
0–20	7.20	16.40	3.80	0.50	20 ± 2
20–40	7.80	14.80	5.00	0.60	28 ± 2
40–60	7.00	11.60	3.60	0.40	20 ± 2
60–80	5.80	9.20	1.20	0.20	1.8 ± 0.4
Profile 3 (P ₃)					
0–20	30.10	34.20	7.60	1.20	35 ± 4
20–40	41.60	24.20	6.20	0.90	27 ± 3
40–60	32.10	17.00	2.20	0.50	8.0 ± 1.0
60–80	28.60	17.40	1.80	0.40	5.0 ± 0.6
Profile 4 (P ₄)					
0–20	25.40	14.60	5.40	1.30	27 ± 3
20–40	15.40	11.40	3.80	1.20	19 ± 2
40–60	13.80	10.00	1.20	0.30	2.3 ± 0.4
60–80	14.60	11.60	1.00	0.20	<1.5

One-way analysis of variance test was also performed to indicate the main differences of trace element contents along the soil depth or between profiles themselves. Test results showed no statistically significant differences between means of contents for Fe and Mn in the soil layers of 0–20-cm, 20–40-cm, 40–60-

cm and 60–80-cm depths. But, in the case of Cu ($p < 0.001$), Zn ($p < 0.01$) and ^{137}Cs ($p < 0.001$), there were statistically significant differences between their means in the 0–20-cm layer in regard to the deeper layers investigated. In the topsoil layer, mean values of contents were the highest, 5.90 and 1.03 mg kg⁻¹, respectively for available Cu and Zn, and 29.0 Bq kg⁻¹ for activity concentration of ^{137}Cs . Mean values of contents of all trace elements were not statistically different among soil profiles (P₁–P₄) except mean Fe contents which were in order: P₃ > P₁ > P₄ > P₂.

Garcia et al. (2014) found higher values of available Fe and Mn in the 0–5-cm soil layer and a homogeneous level in the 5–30-cm layer and concluded that those levels were only slightly affected by the tillage system due to plowing under conventional tillage. Similar soil-profile distributions with depth of available Zn and Cu like ours, that were higher near the soil surface and decreased gradually (Zn) or sharply (Cu) with depth of (up to 90 cm) were found by Franzluebbers et al. (1996).

Typical micronutrient soil-profile distribution (higher levels in the surface relative to subsurface soils) was likely a result of greater decomposition of soil organic matter and crop residues that contribute to micronutrient accumulation to the surface layers. Secondly, root distributions and rooting depth play an important role in shaping micronutrient profiles because nutrients taken up by deep roots are transported into the above-ground parts and re-deposited on the soil surface through stemflow and throughfall (Garcia et al., 2014; Jiang et al., 2009; Franzluebbers et al., 1996).

The effect of measured soil OM on available Fe, Mn, Cu, Zn and on activity concentration of ^{137}Cs and their mutual relationships were analyzed by simple linear regression (Table 2). All trace elements were highly positively correlated, except available Fe that was moderately related only with available Mn. It was commonly reported that the DTPA-extractable Cu, Zn, Mn and Fe in cultivated soils were significantly and positively correlated among themselves (Yi et al., 2012; Yadov, 2011; Kumar et al., 2011) confirming their similar origin in soil.

Variance of soil OM content was of importance for available Cu and the ^{137}Cs distributions (Table 2) along the soil depth with a medium correlation ($r \sim 0.50$) that explained about 25% of their variability ($p < 0.05$). For available Zn, a much higher correlation with soil OM explained 56% of its variations ($p < 0.001$). In the 0–80-cm layer of anthrosol, soil OM content appears to influence the availability of Cu and Zn, and on the other hand, to contribute to immobilization of radiocesium. Soil OM has a great capacity for sorption of trace elements and depending on the solubility of the organic ligand, it can play a dual role, it immobilizes trace elements by the formation of insoluble complexes while dissolved organic matter forms strong soluble complexes increasing trace element solubility (Alloway, 2013).

Cu and Zn might form inner-sphere complexes (creating chelate rings) with soil organic matter. Generally, the more electronegative the metal ion is ($\text{Cu}^{2+} > \text{Zn}^{2+}$), the stronger is the bond formed with organic matter implying that stability of chelates with Cu is higher than with Zn (Petrović et al., 1999). This could be the reason why Zn was not strongly retained by the solid soil surfaces. It became available down the soil rather than Cu that is reflected on their profile distributions. In the case of ^{137}Cs , soil OM retained this radionuclide that is supported by the fact that humic substances in clayey soils facilitate interactions between the soluble radiocesium and the soil particles (Kruyts and Delvaux, 2002). Simple batch measurements showed that “95% of radiocesium was irreversibly sorbed onto the soil even under conditions that theoretically increase its release” (Tamponnet et al., 2008). Tsukada et al. (2007) determined that approximately 10 and 20% of ^{137}Cs contents in cultivated soil are in exchangeable and bound to organic matter fractions, respectively and about 70% are in the residual (strongly bound) fraction of soil.

Table 2. Correlation coefficient values between ^{137}Cs , available micronutrients Fe, Mn, Cu, Zn and soil organic matter.

	^{137}Cs	Cu	Zn	Mn	Fe	SOM
^{137}Cs	1	0.98 ^a	0.80 ^a	0.66 ^b	<i>ns</i>	0.51 (27%)
Cu		1	0.84 ^a	0.76 ^a	<i>ns</i>	0.50 (25%)
Zn			1	0.59 ^b	<i>ns</i>	0.75 ^a (56%)
Mn				1	0.60	<i>ns</i>
Fe					1	<i>ns</i>
SOM						1

Significance levels: ^a $p \leq 0.001$; ^b $p \leq 0.01$; $p \leq 0.05$; *ns* – not significant.

In the previous study of the same anthrosol (Tomić et al., 2011), illite was dominant mineral of the clay fraction with proportion of approximately 50% which should be taken into account as an important additional fact considering trace metal distribution with soil depth. It is well documented that frayed edge sites of illite minerals strongly fix the radiocesium ions in soil (Larsson, 2008). In the study of Sipos et al. (2008), the correlation between the sorbed Cu and Zn amounts was found to be generally strong in different clay mineral particles investigated and the strongest for illite minerals, indicating that these metals are immobilized on the same particles. In investigated soil profiles, results of simple linear regression confirmed that available Mn ($p < 0.01$), Cu ($p < 0.05$), Zn ($p < 0.01$) and radionuclide ^{137}Cs ($p < 0.05$) are positively and highly correlated with the percentages of illite minerals, which could be one more reason to explain a high mutual correlation among these trace elements (Table 2).

Conclusion

The objective of this study was to investigate depth distribution of available micronutrients Fe, Mn, Cu and Zn and fallout ^{137}Cs radionuclide in cultivated soil used for growing fruit trees. Results of simple linear regression analysis showed a high and positive correlation among available Mn, Cu, Zn and radionuclide ^{137}Cs . An exception was available Fe that was related only with available Mn. The distribution of radiocesium and available Cu and Zn in the 0–80-cm layer of anthrosol soil profiles was found to be connected to soil organic matter content. Available Fe and Mn were more evenly distributed with soil depth that is probably a result of their similar origin in soil.

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RASPODELA PRISTUPAČNIH SADRŽAJA MIKROELEMENATA
PO DUBINI KULTIVISANOG ZEMLJIŠTA

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R e z i m e

U ovom radu ispitivana je raspodela sadržaja pristupačnih mikroelemenata Fe, Mn, Cu i Zn i koncentracije aktivnosti proizvedenog radionuklida ¹³⁷Cs u profilima zemljišta (dubine 0–80 cm) sakupljenih sa voćnjaka pod zasalom breskvi na oglednom školskom poljoprivrednom dobru „Radmilovac” (Poljoprivredni fakultet, Univerzitet u Beogradu) u neposrednoj blizini Instituta za nuklearne nauke „Vinča“. Zemljište pripada klasi antrosol antropogenih zemljišta prema međunarodnoj FAO (2006) klasifikaciji. Priprema zemljišta za sadnju bresaka izvršena je rigolovanjem, posle čega je usledila 12 godina duga nega voćnjaka. Uzorkovanje zemljišta izvršeno je tri godine nakon prestanka svih poljoprivrednih tretmana zemljišta na oglednom polju. Sadržaji pristupačnih mikroelemenata Fe, Mn, Cu i Zn (dobijeni ekstrakcijom sa rastvorom 0,005 M DTPA) nalazili su se u opsegu (mg kg⁻¹): 5,8–41,6; 9,2–34,2; 1–7,6 odnosno 0,2–1,3. Detektovana koncentracija aktivnosti ¹³⁷Cs u zemljištu (merena metodom gama-spektrometrije korišćenjem koaksijalnog HPGe-detektora) nalazila se u intervalu (Bq kg⁻¹): 1,8–35. Uočeno je da su obrasci distribucije po dubini profila pristupačnih oblika Cu i Zn i radionuklida ¹³⁷Cs bili veoma slični, a rezultati proste linearne regresione analize su pokazali da je uzajamni afinitet prema organskoj materiji zemljišta mogao da utiče na takvu njihovu distribuciju. Sadržaji pristupačnih mikroelemenata Fe i Mn, pokazali su da imaju drugačiju, uniformniju distribuciju po dubini ispitivanih profila zemljišta.

Cljučne reči: obradivo zemljište, ¹³⁷Cs, raspoloživi Fe, Mn, Cu i Zn.

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