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COPPER SPECIATION IN DIFFERENT-TYPE SOIL PROFILES

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Abstract

Determination of the total content of metals in soils does not give enough information about their mobility and potential uptake by plants. The influence of heavy metals on plants depends on the type and form of a metal as well as properties of soil.

The aim of the research was to evaluate the influence of soil type (Rendzic Leptosols, Haplic Luvisols, Haplic Arenosols) on the content and speciation of copper in soil profiles.

The research was carried out in two physiographical regions: Lublin Upland and Sandomierska Valley. Samples were collected once from individual genetics horizons, in total from 30 typological differentiated soil profiles, made from chalk marl, loess and sands.

Speciation analysis of copper was carried out with the use of a three-stage sequential method of fractionation, which can isolate four fractions with BCR: fraction I – forms soluble in water, exchangeable and bounded with calcium carbonate, extractable with CH_3COOH; fraction II – forms bound with free Fe and Mn oxides, extractable with NH_2OHHCl; fraction III – forms complexed with organic matter, hot extractable with 30% H_2O_2 and next the mineralization products reextractable with CH_3COONH_4; fraction IV – residual forms (residue), i.e. the difference between the total content and the sum of three fractions I – III.

The speciation analysis indicated that in all the examined soil types, the residual form showed the largest share of copper in its total content, followed by forms bounded with organic matter and, containing the smallest proportion of copper, the soluble, exchangeable and bound with calcium carbonate forms. In rendzinas and lessive soils, the content of fraction IV in the humus horizons was significantly higher than in the parent rock, whereas in Haplic Arenosols the host rock was richer in this copper form than the humus horizons.

Key words: rendzina, Haplic Arenosols, lessive soil, forms of copper.

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SPECJACJA MIEDZI W PROFILACH GLEB ZRÓŻNICOWANYCH TYPOLOGICZNIE

Abstrakt

Określenie całkowitej zawartości metali w glebie nie daje wystarczających informacji o ich ruchliwości i możliwości pobierania przez rośliny. Oddziaływanie metali ciężkich na rośliny zależy od rodzaju metalu, formy, w jakiej występuje, a także od właściwości gleby.

Celem pracy była ocena wpływu typu gleb (rędziny, płowe, rdzawe) na zawartość i specjację miedzi w ich profilach.

Badania prowadzono na obszarze obejmującym 2 regiony fizjograficzne: Wyżynę Lubelską i Kotlinę Sandomierską. Próbki pobrano jednorazowo z poszczególnych poziomów genetycznych, w sumie z 30 profili glebowych zróżnicowanych typologicznie, wytworzonych z margli kredowych, lessów i piasków.

Analizę specjacyjną miedzi przeprowadzono z wykorzystaniem trzystopniowej metody sekwencyjnego frakcjonowania umożliwiającej wyodrębnienie 4 frakcji metodą BCR: frakcja I – formy rozpuszczalne w wodzie, wymienne i związane z węglanami, ekstrahowane CH_3COOH; frakcja II – formy zasocjowane z wolnymi tlenkami Fe i Mn ekstrahowane NH_2OHHCl; frakcja III – formy związane z materią organiczną ekstrahowane 30% H_2O_2 na gorąco i następnie reekstrahowane produkty mineralizacji CH_3COONH_4; frakcja IV – formy rezydualne (pozostałość), różnica między całkowitą zawartością a sumą trzech frakcji.

Analiza specjacyjna wykazała, że we wszystkich typach badanych gleb, forma rezydualna miedzi stanowiła największy udział w jej całkowitej zawartości, następnie formy związane z materią organiczną, a najmniejszy formy rozpuszczalne, wymienne i związane z węglanami. W rędzinach i glebach płowych poziomy próchniczne zawierały istotnie więcej IV frakcji niż skały macierzyste, natomiast w glebach rdzawych skała macierzysta zwierała więcej tej formy miedzi niż poziomy próchniczne.

Słowa kluczowe: rędziny, gleby płowe, gleby rdzawe, formy miedzi.

INTRODUCTION

The average copper content in Polish soils is 6.6 mg·kg^{-1} (TERELAK et al. 1997). According to TERELAK et al. (1997), 96.5% of arable lands area in Poland shows natural copper concentrations, while 3.1% has elevated copper levels. Soils slightly, moderately and strongly contaminated with copper make up only 0.2% of the total area. While studying the soil abundance in microelements, CZUBA (1995) reported that the largest percentage of Polish soils showed boron and copper deficiencies.

Determination of the total content of metals in soil does not provide sufficient information on their mobility and potential uptake by plants. Interactions of heavy metals with plants depend on the type and form of a metal as well as soil properties (KUCHARZEWSKI et al. 2004). Methods for determinations of various metals in soil include sequential procedures, in which a variety of single agents are used for extraction. These methods are used for determining soil fractions and their sequence (KABATA-PENDIAS, SADURSKI 2003, KARCZEWSKA 2002). Methods of sequential extraction have some advatnages and disadvantages. Different chemicals are used to determine particular metal fractions, and their selection should depend on examined soils properties, including the level of heavy metal contamination.

The aim of the present study has been to evaluate the influence of soil types (Rendzic Leptosols, Haplic Arenosols, and Haplic Leptosols) on copper content and speciation in soil profiles.

MATERIAL AND METHODS

The study was carried out in two physiographic regions: Lubelska Upland and Sandomierska Valley. Samples were collected once from particular horizons, from different types of soil profiles developed from marls, sands, and loess. Ten profiles from every soil type (5 from forest and 5 from cultivated fields) were collected (Figure1).

Fig. 1. Location of the investigated area

Soils developed from marls were classified to proper rendzic soils, and according to WRB classification – to *Rendzic Leptosols,* while those developed from sands – to *Haplic Arenosols*, and developed from loess – to *Haplic Luvisols.*

Collected soil samples were subjected to determinations of granulometric composition by means of Bouyoucos's method with modifications by Casagrande and Prószyński (OSTROWSKA et al. 1991); pH in 1 mol $KCl \cdot dm^{-3}$ maintaining soil to water ratio for 1:2.5; organic carbon content applying Tiurin's method with modifications by Simakov (OSTROWSKA et al. 1991); as well as the total copper content – after digestion of soil samples in a HNO_3 and $HClO_4$ mixture (1:1) (OSTROWSKA et al. 1991) by means of the F-AAS technique.

The speciation analysis of copper was performed using the three-stage method of sequential fractionation, which makes it possible to separate 4 fractions by means of the BCR technique (URE et al. 1993, THOMAS et al. 1994): fraction I – water-soluble forms, exchangeable and bonded to carbonates, extractable with CH_3COOH at 0.11 $mol \cdot dm^{-3}$ concentration and pH=2; fraction II – forms associated with free Fe and Mn oxides, extractable with NH_2OHHCl at 0.1 $mol \cdot dm^{-3}$ concentration and pH=2; fraction III – forms bonded to organic matter, extractable with hot 30% H_2O_2 and then the mineralization products re-extracted with CH_3COONH_4 at 1 $mol \cdot dm^{-3}$ concentration and pH=2; fraction IV – residual forms (remains), the difference between the total copper content and sum of the above three fractions. All the four fractions were determined by means of the atomic absorption spectrometry technique using a Varian Spectra-250 Plus system.

The results of these determinations were presented as arithmetic mean values, and in order to evaluate the variability of the tested characteristics, the minimum, maximum and standard deviation values were calculated. Statistical processing was made using Statistica 6.0 (StatSoft) software.

RESULTS AND DISCUSSION

Rendzic soils were characterized by the granulometric composition of heavy loams: 4-32% (mean 15%) of fraction 1-0.1 mm; 12-31% (mean 19%) of fraction 0.1-0.02 mm; 52-85% (mean 66%) of fraction <0.02 mm; haplic arenosols were loose sands containing: 83-96% (mean 91%) of fraction 1-0.1 mm, 1-13% (mean 6%) of fraction 0.1-0.02 mm; 0-7% (mean 3%) of fraction <0.02 mm; haplic luvisols were characterized as dust and contained: 0-20% (mean 3%) of fraction 1-0.1 mm; 40-65% (mean 55%) of fraction 0.1-0.02 mm; 22-60% (mean 39%) of fraction <0.02 mm.

Humus and transitional horizons of the examined rendzic soils had neutral acidity (pH 6.0-7.1), while the mother rock developed from marls was basic (pH 7.2-7.7). Haplic arenosols were strongly acidic and acidic with pH from 3.6 to 5.6. The pH value of haplic luvisols varied from strongly acidic to basic. In humus horizons, pH value was within the range of 3.8-6.7, in elution horizons (Eet) 4.0-5.0, in enrichment horizons (Bt) 4.0-7.1, and in mother rock 6.9-7.6.

The organic carbon content in humus horizons of rendzic soils ranged from 12.6 to 45.2 $g \cdot kg^{-1}$, haplic arenosols 4.2-20.4 $g \cdot kg^{-1}$, and in haplic luvisols 8.4-25.3 $g \cdot kg^{-1}$.

Total average copper content in humus horizons of rendzic soils amounted to 10.73 $mg \cdot kg^{-1}$, in transitional horizons 8.74 $mg \cdot kg^{-1}$, and in mother rock 6.44 $mg \cdot kg^{-1}$ (Table 1). In haplic arenosols, mean copper concentration did not exceed 3 $mg \cdot kg^{-1}$, while in haplic luvisols, mean copper content in humus horizons was 6.49 $mg \cdot kg^{-1}$. These were values corresponding to natural not contaminated levels (KABATA-PENDIAS et al. 1993). Low copper concentrations in haplic arenosols might be associated not only with the mother rock (KABATA-PENDIAS, PENDIAS 1999) that is typically deficient in the metal, but also a strong acidity of that soil type. Under such conditions, copper compounds are easily soluble and may migrate along with soil solution inside the soil profile; they are also faster taken by plants (GANCARCZYK-GOLA, PALOWSKI 2005).

In the analyzed soil types, copper distribution varied in particular horizons. In rendzic soils, the copper content decreased with the profile depth, whereas in haplic arenosols, particular horizons were characterized by simi-

Table 1

Total content of copper in examined soil profiles ($mg \cdot kg^{-1}$)

Genetic horizon	Minimum	Maximum	Mean
Rendzinas			
Aca	8.25	13.20	10.73
ACca	5.25	13.35	8.74
Cca	3.75	9.90	6.44
Arenosols			
A	2.00	4.25	2.96
Bv	1.25	4.25	2.35
C	0.90	3.90	2.03
Lessive soils			
A	4.45	8.00	6.49
Eet	3.40	8.00	5.98
Bt	6.10	14.75	9.31
Cca	6.50	11.60	8.03

lar copper levels. UZIAK et al. (2001) reported similar dependencies in different types of soils developed from sands in eastern part of Poland. SZYMAŃSKA (1996), who studied podzolic soils, found the largest copper accumulation in surface layers, revealing a decreasing tendency with the soil depth. In haplic luvisols, the elution levels (Eet) were the most deficient in copper, while enrichment levels (Bt) were characterized by its accumulation. Mean copper content in the mother rock of haplic luvisols was higher than in humus horizons. UZIAK et al. (2001) recorded similar distribution of total copper contents in profiles of soils developed from dusts. Also JAWORSKA (1996), in studies upon haplic luvisols developed from dusts and loams in Ziemia Dobrzyńska, found two centers of copper accumulation in profiles: humus and illuvial horizons.

Copper fraction I (water-soluble, exchangeable and bonded to carbonates forms) in humus horizons of rendzic soils was within the range from 0.09 to 0.32 $mg \cdot kg^{-1}$ – 0.19 $mg \cdot kg^{-1}$, on average (Table 2). Transitional horizon and mother rock contained higher amounts of that fraction as compared to humus horizon. In haplic arenosols, the fraction in all horizons was at similar trace levels – 0.09 $mg \cdot kg^{-1}$, on average (Table 3). In haplic luvisols, the mean content of fraction I in particular horizons varied from 0.07 to 0.17 $mg \cdot kg^{-1}$, and the mother rock contained the largest quantities of that

Table 2

Content of copper fractions in rendzina profiles ($mg \cdot kg^{-1}$)

Genetic horizon	Fraction	Minimum	Maximum	Mean	Standard deviation
A	I	0.09	0.32	0.19	0.07
	II	0.004	0.36	0.22	0.11
	III	0.26	3.01	1.54	0.86
	IV	4.80	12.81	8.78	2.64
ACca	I	0.14	0.42	0.31	0.09
	II	0.07	0.39	0.30	0.10
	III	0.46	2.79	1.56	0.66
	IV	2.85	12.68	6.58	3.02
Cca	I	0.08	0.43	0.33	0.12
	II	0.04	0.49	0.30	0.14
	III	0.35	2.08	1.61	0.54
	IV	1.03	9.03	4.20	2.71

Explanations: I – forms soluble in water, exchangable and bound to calcium carbonate; II – forms associated with free Fe and Mn oxides; III – forms bound to organic matter; IV – residual forms

Table 3

Content of copper fraction in Haplic Arenosol profiles (mg·kg^{-1})

Genetic horizon	Fraction	Minimum	Maximum	Mean	Standard deviation
A	I	0.01	0.13	0.09	0.04
	II	0.02	0.16	0.08	0.05
	III	0.28	0.89	0.52	0.19
	IV	1.35	3.57	2.27	0.84
Bv	I	0.06	0.16	0.10	0.03
	II	0.01	0.20	0.08	0.06
	III	0.18	0.34	0.26	0.06
	IV	0.66	3.81	1.91	1.03
C	I	0.03	0.18	0.09	0.05
	II	0.03	0.15	0.06	0.04
	III	0.04	0.44	0.24	0.12
	IV	0.13	3.46	1.64	1.13

Explanations as in Table 2

fraction (Table 4). For all the studied soil types, this fraction – the most available one for plants – was a small percentage of the total copper content (in humus horizons of the soils: haplic luvisols 1.26%, haplic arenosols 2.97%, rendzic soils 1.79%). Taking into account all the horizons, mother rocks of all the studied soils were characterized by the highest content of this copper fraction.

Copper fraction II (associated with iron and manganese oxides), like fraction I, was less than 3% of the total copper quantity in all the studied soil types. McLaren, Crawford (1973) reported that free manganese oxides play a very important role in binding copper in soil. This finding was not confirmed in the soil types we assayed, analogously to chernozems analyzed by Dąbkowska-Naskręt, Kędzia (1996).

The content of copper fraction III (bonded to humus) in rendzic soils and Haplic Luvisols was much higher than in Haplic Arenosols. In Haplic Arenosols and Luvisols, humus horizons were more abundant in this copper form as compared to the other horizons. Copper fraction III made up from 17.6% to 21.8% of the total metal content. The results confirmed strong copper affinity to form complexes with functional groups of humic and fulvic acids, which affects the mobility, thus availability of the element in the soil (McLaren, Crawford 1973, Kabata-Pendias, Pendias 1999). In rendzic soils, the content of copper bonded to humus was similar in all horizons and amounted to 14.3% of the total copper quantity. It may have resulted from imperfections of the method for extraction of the organic fraction from soils at high

Table 4

Content of copper fraction in lessive soil profiles (mg·kg^{-1})

Genetic horizon	Fraction	Minimum	Maximum	Mean	Standard deviation
A	I	0.02	0.17	0.08	0.05
	II	0.08	0.19	0.14	0.04
	III	0.51	2.14	1.42	0.60
	IV	3.23	6.69	4.85	1.26
Eet	I	0.01	0.10	0.07	0.04
	II	0.11	0.17	0.14	0.03
	III	0.49	2.36	1.25	0.73
	IV	2.30	7.26	4.52	1.79
Bt	I	0.05	0.20	0.11	0.05
	II	0.10	0.22	0.17	0.04
	III	0.33	2.50	1.01	0.57
	IV	4.52	13.92	8.02	2.46
Cca	I	0.09	0.33	0.17	0.07
	II	0.01	0.30	0.16	0.07
	III	0.35	1.45	0.79	0.31
	IV	4.75	10.81	6.90	2.12

Explanations as in Table 2

pH because under such conditions strong oxidizers make additional metal precipitation from other fractions (KARCZEWSKA 2002).

Fraction IV (residual) of copper made up the largest percentage in its total content in all the studied soil types. Aslo GONDEK and FILIPEK-MAZUR (2005), when determining the copper concentrations in soil by means of Zeinen and Brümmer's sequential extraction, recorded the highest levels of copper in a fraction bonded to organic matter and the residual one. Studies performed by DĄBKOWSKA-NASKRĘT, KĘDZIA (1996) on chernozems also revealed the highest amounts of the form bonded to organic matter and the residual fraction. Similar results were achieved by CHAO et al. (2007) for cultivated soils in China. Such fraction distribution in profiles depended on a soil type. In Haplic Arenosols, the content of the fraction was the highest in humus horizons and gradually decreased with the depth, falling two-fold in the mother rock. Also in Haplic Arenosols, the content of the residual fraction decreased along with the depth, but not to the same extent in rendzic soils. That fraction was most accumulated in enrichment horizons (Bt) of haplic luvisols (Table 4). In particular horizons, the residual fraction contents could be lined up in a following sequence: Bt>Cca>A>Eet.

CONCLUSIONS

1. Humus horizons of studied soils were characterized by varied natural copper contents. Rendzic soils were the most abundant in copper, haplic luvisols contained less copper, while haplic arenosols the least.

2. The speciation analysis revealed that for all soil types, the residual form of copper made up the largest percentage in its total content, then forms bonded to organic matter, and soluble, exchangeable, and bonded to carbonates.

3. The low contents of mobile copper forms indicated its poor migration and bio-availability.

4. Speciation analysis revealed a significant role of humus in binding potentially mobile copper fractions in studied soil types.

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8. Tabele i rysunki należy załączyć w oddzielnych plikach. U góry, po prawej stronie tabeli należy napisać Tabela i numer cyfrą arabską, również w języku angielskim, następnie tytuł tabeli w języku polskim i angielskim wyrównany do środka akapitu. Ewentualne objaśnienia pod tabelą oraz opisy tabel powinny być podane w języku polskim i angielskim. Wartości liczbowe powinny być podane jako zapis złożony z 5 znaków pisarskich (np. 346,5; 46,53; 6,534; 0,653).
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