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DISTRIBIUTION OF NICKEL FRACTIONS IN FOREST LUVISOLS IN THE SOUTH PODLASIE LOWLAND

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Abstract

Sequential extraction methods enable identification of chemical fractions of heavy metals in soil environment as well as evaluation of their availability and potential toxicity to biotic elements of a trophic chain. The study aimed at separating nickel fractions from particular genetic horizons of forest Luvisols by means of three sequential extraction methods (modified Tessier's, Zeien and Brümmer's as well as Hedley's with Tiessen and Moir's modifications methods), and to compare the metal content in four fractions: easily soluble, exchangeable, organic, and residual, along with their distribution within studied soils' profiles. Nickel concentrations in the examined fractions varied: the largest amounts of the heavy metal (regardless of the analytical procedure applied) were found in residual fraction F_{resid} (mineral horizons) and organic fraction F_{org} (forest litter horizons – Ol), while the smallest ones occurred in easily soluble fraction F1 (all genetic horizons). Statistical processing revealed significant dependences between the four nickel fractions as well as between the fractions and selected properties of analyzed soils (except soil pH and total nickel content Ni_t).

Keywords: sequential extraction, nickel fractions, Luvisols, forest.

ROZMIESZCZENIE FRAKCJI NIKLU W LEŚNYCH GLEBACH PŁOWYCH NA NIZINIE POŁUDNIOWOPODLASKIEJ

Abstrakt

Metody ekstrakcji sekwencyjnej umożliwiają identyfikację frakcji chemicznych metali ciężkich w środowisku glebowym, a także ocenę ich dostępności i potencjalnej toksyczności dla biotycznych elementów łańcucha troficznego. Celem pracy było wydzielenie frakcji ni-

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klu w poszczególnych poziomach genetycznych leśnych gleb płowych trzema metodami ekstrakcji sekwencyjnej (zmodyfikowanej metody Tessiera, metody Zeiena i Brümmera oraz metody Hedleya w modyfikacji Tiessena i Moira), a także porównanie zawartości tego metalu w czterech frakcjach: łatwo rozpuszczalnej, wymiennej, organicznej i rezydualnej oraz ocena ich rozmieszczenia w profilu badanych gleb. Zawartość niklu w badanych frakcjach była zróżnicowana. Najwięcej tego metalu, niezależnie od zastosowanej procedury analitycznej, stwierdzono we frakcji rezydualnej – F_{resid} (poziomy mineralne) oraz organicznej – F_{org} (poziomy ściółki leśnej – Ol), a najmniej we frakcji łatwo rozpuszczalnej – F1 (wszystkie poziomy genetyczne). Obliczenia statystyczne wykazały, że badane cztery frakcje niklu były wysoko istotnie zależne między sobą oraz wybranymi właściwościami analizowanych gleb (z wyjątkiem pH gleby i zawartości ogólnej Ni_t).

Słowa kluczowe: ekstrakcja sekwencyjna, frakcje niklu, gleby płowe, las.

INTRODUCTION

Environmental pollution may contribute to the accumulation of heavy metals, which is an important link in bio-geochemical cycles of elements. Nickel is a heavy metal which is essential for the growth and development of living organisms, but in excess can be toxic (Kabata-Pendias, Pendias 1999). Different (regarding their solubility) chemical fractions (forms), the percentage of which depends on the soil properties (pH value, content of organic and mineral colloids, organic matter, redox potential, and parent rock's abundance), make up the total content of this metal in soil. Sequential extraction methods enable identification of chemical fractions of metals, their mobility within soil environment as well as their availability and potential toxicity to biotic elements of a trophic chain (Kalembkiewicz, Sočo 2005).

The research was aimed at examining the nickel content in four fractions extracted by means of three sequential extraction methods as well as at comparing their distribution within the profile of forest Luvisols.

MATERIAL AND METHODS

Luvisols developed from loams and localized within three forest complexes in the South Podlasie Lowland were studied. The following were determined in air-dried soil samples collected from particular genetic horizons of the three profiles: granulometric composition of mineral horizons by aerometric method (according to PN-R-04033); pH in 1 mol $KCl \cdot dm^{-3}$ (pH_{KCl}) by potentiometry; soil's sorption capacity (T = CEC) on the basis of hydrolytic acidity value (Hh) and sum of basic cations (S) determined by means of Kappen's method; organic carbon content (Corg) by the oxidation-titrimetric method (Kalembasa, Kalembasa 1992); total nickel content (Ni_t) by the ICP-EAS technique after wet digestion in concentrated nitric acid in a microwave system. Nickel sequential fractionation was made applying three analytical procedures (Table 1): modified Tessier's method (own modifications consisted in adjusting the extraction solutions to determinations by means of ICP-EAS) (Tessier et al. 1979, Kalembasa, Pakuła 2006), Zeien and Brümmer's method (Zeien, Brümmer 1989), as well as Hedley's method with Ties-

Table 1

Nickel sequential extraction methods used in the examined forest Luvisols

Fraction	Extraction reagent and condition
Tessier et al. method (in own modification)	
F1 easily soluble	20 cm^3 deionized H_2O, pH=7, shake for 1 h
F2 exchangeable	20 cm^3 1 mol $NH_4Cl \cdot dm^{-3}$, pH=5.5, shake for 1 h
F3 bound to carbonates	20 cm^3 1 mol $CH_3COOH \cdot dm^{-3}$, pH=3, shake for 5 h
F4 bound to Fe-Mn oxides	40 cm^3 0.2 mol $(NH_4)_2C_2O_4 \cdot dm^{-3}$ + 0.2 mol $H_2C_2O^4 \cdot dm^{-3}$, pH=3, shake for 4 h
F_{org} bound to organic matter	40 cm^3 0.1 mol $NaOH \cdot dm^{-3}$, pH=12.5, shake for 3 h
F_{resid} residual*	
Zeien and Brümmer's method	
F1 easily soluble	20 cm^3 unbuffered 1 mol $NH_4NO_3 \cdot dm^{-3}$, shake for 24 h
F2 exchangeable	20 cm^3 1 mol $CH_3COONH_4 \cdot dm^{-3}$, pH=6, shake for 24 h
F3 bound to MnOx	20 cm^3 1 mol $NH_2OH \cdot HCl \cdot dm^{-3}$ + 1 mol $CH_3COONH_4 \cdot$ $\cdot dm^{-3}$, pH=6, shake for 0.5 h
F_{org} bound to organic matter	20 cm^3 0.025 mol $C_{10}H_{22}N_4O_8 \cdot dm^{-3}$, pH=4.6, shake for 1.5 h
F5 bound to amorphic FeOx	40 cm^3 0.2 mol $(NH_4)_2C_2O_4 \cdot dm^{-3}$+0.2 mol $H_2C_2O_4 \cdot dm^{-3}$, pH=3, shake for 4 h
F6 bound to crystalline FeOx	40 cm^3 0.2 mol $(NH_4)_2C_2O_4 \cdot dm^{-3}$+0.2 mol $H_2C_2O_4 \cdot dm^{-3}$ + 0.1 mol $C_6H_8O_6 \cdot dm^{-3}$, pH=3, 0.5 h in boiling water
F_{resid} residual*	
Hedley's method modified by Tiessen and Moir	
F1 easily soluble	20 cm^3 deionized H_2O, pH=7, shake for 1 h
F2 exchangeable	20 cm3 0.5 mol $NaHCO_3 \cdot dm^{-3}$, pH=8.5, shake for 16 h
F_{org} bound to organic matter	20 cm^3 0.1 mol $NaOH \cdot dm^{-3}$, pH=12.5, shake for 16 h
F4 bound to carbonates	20 cm^3 1 mol $HCl \cdot dm^{-3}$, shake for 16 h
F5 bound to stable organic-min. and mineral compounds	10 cm^3 conc. HCl in a water bath at 80°C for 10 min
F_{resid} residual*	

* Calculation as difference between total content of nickel and sum of the above determined fractions.

sen and Moir's modifications (referred to as Hedley's method in further parts of present paper) (Tiessen, Moir 1993).

Soil samples (1 g) were subjected to extraction with sequentially replaced extraction solutions. After centrifuging, particular nickel fractions were determined in the extracts by means of the ICP-EAS technique. The accuracy of the analytical procedures was confirmed by applying the standard addition method to every analyzed sample. Control samples consisting of used solutions were also included in the analyses.

In order to compare the applied sequential extraction schemes, four fractions defined as easily soluble (water-soluble F1), exchangeable (F2), bonded to organic matter (organic fraction F_{org}), and residual (F_{resid}) fractions were selected from six fractions extracted with Tessier's and Hedley's methods, and from seven fractions obtained with Zeien and Brümmer's method. The four fractions were selected because they could be directly separated in particular analytical procedures and because the percentage of their total amount in the soil best represents a diverse character of the analyzed bindings with solid phase components in the soil, from the most labile (fractions F1 and F2), through potentially labile (F_{org}), to very stable mineral and organic-mineral complexes (F_{resid}).

The following values were calculated for particular genetic horizons of soils: nickel enrichment coefficient (WW) in relation to its content in the mother rock, and mobility index (WM) as the percentage of the sum of F1 and F2 fractions, which enables evaluation of the current bio-availability of nickel.

Dependencies between total nickel content and its quantities in particular fractions as well as selected soil properties were evaluated by means of linear correlation.

RESULTS AND DISCUSSION

The physical, physicochemical and chemical properties of soils chosen for the study are characteristic for forest Luvisols developed from material deposited during the Middle-Polish glaciation (Table 2). The total nickel content ranged from 4.75 to 20.2 $mg \cdot kg^{-1}$ in the soils, which did not exceed the geochemical background level for boulder loam deposits (Czarnowska 1996). The differences in the metal concentration were probably caused by the dual structure of the soil profile (surface horizons consisted of sandy deposits, while enrichment horizons and parent rock were made of loamy deposits). Particular genetic horizons were were ordered according to the total nickel content as floows: IIBt > IIC > Ol > Ah > Eet. The distribution of nickel in the soil profile resulted from a great abundance of this metal in the parent rock (low values of enrichment coefficient WW in sandy horizons Ah

Table 2

Some properties (mean* and ranges** for three soil profiles) of the forest Luvisols

Genetic horizon	Sand 2-0.05	Silt 0.05-0.002	Clay <0.002	pH_{KCl}	CEC***	Corg	Ni_t****	WW*****
	% fraction of diameter in mm				mmol(+)·kg^{-1}	g·kg^{-1}	mg·kg^{-1}	
Ol	-	-	-	-	631.0	458.0	17.8	0.99
	-	-	-	4.81-5.16	545.0-700.0	443.0- 472.0	14.5-19.2	0.74-1.18
Ah	72*	23	5	-	89.4	16.1	5.70	0.32
	69-76**	19-25	4-6	3.08-3.36	75.2-107.0	12.0-20.3	4.98-6.35	0.25-0.39
Eet	77	19	4	-	48.9	3.67	4.75	0.26
	73-80	17-22	3-5	3.55-3.65	43.6-54.0	2.30-5.00	3.76-5.87	0.19-0.36
	44	26	30	-	170.0	1.83	20.2	1.12
IIBt	37-53	23-31	24-32	3.32-3.48	146.0-187.0	1.60-2.00	18.3-21.8	1.11-1.13
	43	31	26	-	289.0	1.42	18.0	-
IIC	35-53	25-37	22-28	6.81-7.43	238.0-388.0	1.30-1.60	16.2-19.6	-

***CEC – Cation Exchenge Capacity
****total content of nickel
*****enrichment coefficient values

and Eet) and soil-forming lessivage processes (in mineral horizons), as well as biological accumulation (in organic horizons), which was confirmed by the highest values of WW index in Bt and Ol horizons. Kwasowski et al. (2000), and Uziak et al. (2001) reported similar nickel distribution in forest Luvisols. Kabata-Pendias and Pendias (1999) as well as Konecka-Betley et al. (1999) underline that nickel occurrence in a soil profile corresponds to its content in parent rocks (C), whereas its elevated concentrations in surface horizons (Ol) result from biological accumulation and anthropopression.

Different shares of the nickel fractions determined by means of the three sequential extraction methods were recorded in the soils (Table 3). The mean percentage of the nickel fractions in its total content for particular genetic horizons can be presented in a form of the following sequences:

Ol: organic (30.2) > residual (25.5) > exchangeable (13.8) > easily soluble (12.1);

Ah: residual (42.5) > organic (6.93) > exchangeable (5.88) > easily soluble (2.34);

Eet: residual (48.6) > exchangeable (5.60) > organic (5.44) > easily soluble (1.70);

IIBt: residual (63.4) > exchangeable (1.19) > organic (0.75) > easily soluble (0.61);

IIC: residual (71.5) > organic (0.51) > exchangeable (0.39) > easily soluble (0.29).

In the mineral horizons of the analyzed soils, nickel dominated in the residual fraction (F_{resid}) – Table 3. Most of the nickel in this fraction was separated by means of Tessier's method (73.59 %), while the least applying

Table 3

The mean percentage contribution of selected nickel fractions in the soils

Genetic horizon	Fraction				WM*
	F1	F2	F_{org}	F_{resid}	(%)
Tessier et al. method					
Ol	9.38	13.90	32.21	25.36	23.28
Ah	1.56	6.14	7.02	53.10	7.70
Eet	1.13	5.99	5.44	65.80	7.12
IIBt	0.43	1.19	0.72	85.24	1.62
IIC	0.25	0.38	0.52	90.20	0.63
Zeien and Brümmer method					
Ol	17.70	15.10	23.10	35.63	32.80
Ah	3.92	6.53	5.22	43.47	10.45
Eet	2.85	6.21	4.38	46.10	9.06
IIBt	0.97	1.54	0.58	65.43	2.51
IIC	0.39	0.48	0.41	77.70	0.87
Hedley method in modification by Tiessen and Moir					
Ol	9.28	12.50	35.20	15.57	21.78
Ah	1.55	4.98	8.55	30.90	6.53
Eet	1.13	4.59	6.51	33.80	5.72
IIBt	0.43	0.84	0.94	39.47	1.27
IIC	0.24	0.31	0.61	46.60	0.55

Fraction: F1– easy soluble, F2 – exchangeable, F_{org} — ound to organic matter, F_{resid} — esidual;
*mobility coefficient values

Hedley's method (37.69%, on average). Lower contribution of nickel in the residual fraction (Hedley's method) resulted from the extraction of the previous fraction (F5) using concentrated HCl, which made the metal release from more eroded minerals and less durable organic-mineral bindings.

The lowest amount of nickel from the residual fraction was recorded in organic horizons (Ol) (from 15.57 to 35.65 %), and its contribution increased with the profile's depth, reaching the maximum values in the parent rock. A similar profile distribution of this nickel fraction in soils was also observed by Abollino et al. (2002) as well as Kaasalainen, Yli-Halla (2003). Kabata--Pendias, Pendias (1999) reported that nickel in mineral horizons is characterized by great affinity to clay minerals, while in organic horizons, it is chelated by organic matter. Palumbo et al. (2000), Andersen et al. (2002) and Królak (2004) determined from 60.0 to 84.0 % of the residual fraction of nickel in Luvisols (agricultural and forest).

In the organic fraction (F_{org}), the largest percentage of nickel was determined in forest litter horizons (Ol); the largest amounts were separated with Hedley's method (35.20 %), while the smallest ones were obtained with Zeien and Brümmer's method (23.10 %) – Table 3. IThis confirmed a more selective action of NH_4-EDTA (Zeien and Brümmer's method) compared to

NaOH solution (applied in the other two methods), which may destabilize relatively strong organic-mineral bindings, including bonds with iron and manganese oxides (Hlavay et al. 2004). Distribution of nickel in the organic fraction of the soil profile, regardless of the applied analytical procedure, decreased with the depth, which was also observed by Palumbo et al. (2000) and Abollino et al. (2002), who determined the largest quantities of organic nickel bindings in surface horizons: 14.0% and 1.56%, respectively. In organic horizons of acidic soils, organic nickel forms prevail. They may be transported inside the soil profile, where they are mineralized (Andersen et al. 2002).

The highest nickel contribution (9.28-17.70%) in the bio-available fractions, easily soluble (F1) and exchangeable (F2), were found in the organic horizons (Ol), while the lowest ones (0.24-0.48%) were in the parent rock (C) – Table 3. The lowest amounts of nickel were extracted by means of Tessier's and Hedley's method, while the highest ones – with Zeien and Brümmer's method, which was confirmed by the calculated values of mobility index (WM). Hlavay et al. (2004) as well as Kalembkiewicz, Sočo (2005) also reported higher efficiencies of NH_4NO_3 and CH_3COONH_4 solutions for separating easily soluble and exchangeable fractions as compared to chlorides (NH_4Cl) and de-ionized water. Królak (2004) found about 12.0% available nickel fractions in humus horizons of arable soils, while Palumbo et al. (2000) from 1.2 to 2.9% its total content. Kaasalainen and Yli-Halla (2003) recorded 11.0-16.0% of nickel in F1 and F2 fractions of surface horizons of contaminated soils. Decrease of easily soluble and exchangeable fractions with the depth was observed by Abollino et al. (2002) and Andersen et al. (2002).

On the basis of the linear correlation coefficients (Table 4), highly significant dependence was verified between nickel content in particular fractions (extracted using the three analytical procedures): easily soluble, exchangeable, organic (positive) as well as residual (negative) in the examined forest Luvisols. Statistical analysis revealed highly significant negative influence of organic carbon content (C_{org}) and sorption capacity – CEC (except Zeien and Brümmer's methods) as well as a positive effect of clay fraction ($\check{r} < 0.002$ mm) on nickel concentration in the residual fraction. Stable and non-exchangeable nickel bindings with the mineral part of soil (colloidal particle size) determine the contribution of F_{resid} in the total amount of the metal in soil. Nickel levels in F1, F2, and F_{org} fractions were correlated with C_{org} and CEC values (positively) as well as clay amount (negatively). Susceptibility of nickel towards chelate bindings with organic matter determines great mobility of the heavy metal. Bio-available fractions can be exchangeably adsorbed on soil colloids. Palumbo et al. (2000) as well as Kaasalainen and Yli-Halla (2003) reported significant influence of the soil properties (organic carbon content, sorption capacity, and clay fraction < 0.002 mm content) on nickel speciation in the soil environment. No effects of the total nickel content (Ni_t) and soil pH on the metal speciation in the examined soils were found.

Table 4

The coefficient values of the correlation between the fractions of nickel and some properties of the soils

Specification	F1	F2	F_{org}	F_{resid}	Ni_t	pH_{KCl}	C_{org}	CEC	Ø < 0.002
Tessier et al. method									
F1	x	0.927*	0.996*	-0.892*	0.215	0.133	0.993*	0.837*	-0.564*
F2		x	0.954*	-0.985*	-0.150	-0.113	0.879*	0.602*	-0.801*
F_{org}			x	-0.925*	0.134	0.094	0.981*	0.799*	-0.628*
F_{resid}				x	0.201	0.173	-0.836*	-0.561*	0.808*
Zeien and Brümmer method									
F1	x	0.956*	0.999*	-0.726*	0.160	0.089	0.984*	0.806*	-0.605*
F2		x	0.967*	-0.889*	-0.120	-0.106	0.892*	0.622*	-0.783*
F_{org}			x	-0.746*	0.118	0.085	0.977*	0.790*	-0.640*
F_{resid}				x	0.380	0.383	-0.598*	-0.221	0.861*
Hedley method in modification by Tiessen and Moir									
F1	x	0.951*	0.994*	-0.915*	0.213	0.131	0.993*	0.836*	-0.565*
F2		x	0.978*	-0.981*	-0.087	-0.065	0.909*	0.653*	-0.769*
F_{org}			x	-0.945*	0.112	0.077	0.975*	0.786*	-0.643*
F_{resid}				x	0.109	0.234	-0.866*	-0.575*	0.736*

* significant at $\alpha = 0.01$

CONCLUSIONS

1. In the examined forest Luvisols, the total nickel content did not exceed the geochemical background level for boulder loam deposits in Poland. Particular genetic horizons of these soils can be lined up in the following sequence for mean nickel concentrations: IIBt > IIC > Ol > Ah > Eet.

2. Sequential fractionation of nickel in particular genetic horizons by means of Tessier's, Zeien and Brümmer's, and Hedley's methods revealed that its content in the separated fractions varied. Organic horizons of the forest litter (Ol) contained the largest amounts of easily soluble, exchangeable, and organic nickel fractions, while parent rock horizons (C) were the richest in the residual fraction.

3. Chemical analyses revealed that the largest amounts of nickel were contained in easily soluble and exchangeable fractions separated applying Zeien and Brümmer's method; in organic fraction – Hedley's method; and in stable bindings of residual fraction – Tessier's method.

4. Statistical processing revealed that nickel concentration in the four analyzed fractions was highly significantly correlated (positively or negatively) between those fractions as well as with organic carbon content (C_{org}), sorption capacity and clay fraction level in forest Luvisols.

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