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DISTRIBUTION OF NICKEL IN FRACTIONS EXTRACTED WITH THE BCR PROCEDURE FROM NICKEL-CONTAMINATED SOIL

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Abstract

The objective of the study was to evaluate the impact of liming and the application of waste organic material, i.e. sewage sludge, on the content and distribution of nickel in the fractions extracted with the BCR procedure from soil contaminated with this metal. The study was carried out on soil after a 3-year pot experiment, which included the following factors: I – nickel used in the incremental amounts 0, 50, 100 mg Ni kg^{-1} soil; II – liming (0 Ca and Ca according to 1 Hh of soil); and III – the addition of sewage sludge (with and without the addition of sewage sludge at the introducing dose of 2 g C kg^{-1} soil).

The test plant was cocksfoot harvested four times (four swaths) in each plant growing season. The total content of nickel was determined with ICP-AES and its fractions with the three-stage BCR procedure.

The introduction of nickel into the soil resulted in an increase in its total content and in all fractions as well as in its percentage in the exchangeable fraction. Liming reduced the mobility of nickel and decreased its content in the reducible fraction, whil inccreasing it in the residual fraction. The application of sewage sludge contributed to an increase in the total content of nickel in soil and its proportion in the oxidizable fraction. Liming and the application of sewage sludge reduced the mobility of nickel.

Lime and waste organic material (i.e. sewage sludge) were found to be suitable materials for reduction of the mobility of nickel in soil contaminated with this metal.

Key words: soil, liming, sewage sludge, nickel fractions.

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ROZMIESZCZENIE NIKLU WE FRAKCJACH WYDZIELONYCH WG PROCEDURY BCR W GLEBIE ZANIECZYSZCZONEJ TYM METALEM

Abstrakt

Celem pracy była ocena wpływu wapnowania i wprowadzenia do gleby odpadowego materiału organicznego – osadu ściekowego na zawartość i rozmieszczenie niklu we frakcjach wydzielonych wg procedury BCR w glebie zanieczyszczonej tym metalem. Badaniami objęto glebę po trzyletnim doświadczeniu wazonowym, w którym uwzględniono następujące czynniki: I – nikiel stosowany we wzrastających dawkach (0, 50, 100 mg Ni kg^{-1} gleby); II – wapnowanie (0 Ca i Ca wg 1 Hh gleby); III – dodatek osadu ściekowego (bez dodatku i z dodatkiem osadu ściekowego w dawce wprowadzającej 2 g C kg^{-1} gleby).

Rośliną testową była trawa kupkówka pospolita (*Dactylisglomerata* L.), zbierana każdego roku w czterech pokosach (odrostach). Ogólną zawartość niklu oznaczono metodą ICP-AES, a jego frakcje trzystopniową metodą BCR.

Wprowadzenie niklu do gleby spowodowało nie tylko wzrost jego zawartości ogólnej i we wszystkich frakcjach, a jednocześnie zwiększenie procentowego udziału we frakcji wymiennej. Wapnowanie gleby ograniczyło mobilność niklu, zmniejszył się jego udział we frakcji redukowalnej, zwiększył we frakcji rezydualnej. Zastosowany osad ściekowy spowodował zwiększenie ogólnej ilości niklu w glebie i jego udziału we frakcji utlenialnej. Wapnowanie i osad ściekowy ograniczyły mobilność niklu.

Wapnowanie oraz zastosowany w doświadczeniu odpadowy materiał organiczny – osad ściekowy okazały się dobrymi materiałami zmniejszającymi mobilność niklu na glebach zanieczyszczonych tym metalem.

Słowa kluczowe: gleba, wapnowanie, osad ściekowy, frakcje niklu.

INTRODUCTION

Excessive accumulation of toxic substances in soil, including heavy metals, is one of the side effects of the development of civilization (Wyszkowska et al. 2009) Heavy metals are a special type of contaminants, as they are elements which do not break down to basic compounds (Vondrackova et al. 2013). They are found in all types of soil, even in those which are regarded as non-polluted. Their content is low in most soil types, not exceeding several mg kg^{-1} of soil and sometimes even tenths or hundredths of that (Kucharski et al. 2009). The accumulation of heavy metals in soil induces negative changes in its physical, chemical and biological properties, which may have an impact on the availability of many macro- and microelements and, subsequently, on the soil yielding capacity and chemical composition (Wyszkowska et al. 2009). The group of heavy metals includes nickel, which - due to its toxicity and persistence in the environment as well as a high bioaccumulation factor – is classified as a List II substance in the European Commission Dangerous Substances Directive. The World Health Organization (WHO) classified nickel compounds into group I, i.e. carcinogenic substances, while metallic nickel was included in group IIB, i.e. potentially carcinogenic to humans (Nakonieczny 2007). Its presence in soil is associated with parent rocks and with industrial (paper, plating, chemical industry), urban and agricultural pollution, including wrong fertilization with organic waste materials (sewage sludge) (Filipek-Mazur et al. 1999, Cempel, Nikel 2006, Kalembasa, Kuziemska 2010).

Nickel is a heavy metal which is essential for the growth and development of living organisms, but in excess it can be toxic (Kuziemska, Kalembasa 2009). It is relatively easily absorbed by plants proportionally to its content in soil, until it reaches a threshold concentration in plant tissues. The toxicity of this metal to plants has been reported in regions with higher nickel concentrations in soil. The visible symptoms of nickel toxicity include chlorosis and necrosis of leaves, shorter root system, other morphological pathologies in the root system (Marschner 1995, Spiak et al. 2003). Diagnosis of risks to the natural environment through measuring the total concentration of metals, including nickel, in soil and in natural and organic fertilizers, omits many important factors that determine the mobility and bioavailability of elements (pH, content of organic matter, content of clay minerals). Reliable evaluation requires determination of individual forms and fractions which constitute a total content of a given metal (Świetlik, Trojanowska 2008, Pakuła, Kalembasa 2009). Among the numerous methods of eco-toxicological examination of soil, speciation of heavy metals is particularly important because it provides for making a reference to their bioavailability (Qvevauviller 2003, Kalembasa et al. 2009, Reid et al. 2011, Jaske, Gworek 2011, Jaremko, Kalembasa 2011).

The objective of the study was to determine the impact of liming and the application of waste organic material, i.e. sewage sludge, on the content and distribution of nickel in the fractions extracted with the BCR procedure from soil contaminated with this metal.

MATERIAL AND METHODS

The study was carried out on soil after a 3-year pot experiment conducted in a research station of the Siedlce University of Natural Sciences and Humanities. The pot trials were run in 2006-2008, in a randomized model with three replications.

The study included the following factors:

1) contamination of soil with nickel – 0, 50 i 100 mg Ni kg^{-1} soil;
2) liming – 0 Ca (without liming) and Ca (liming) at a dose calculated according to the soil hydrolytic acidity 1;
3) waste organic material – without sewage sludge and with sewage sludge, obtained from a sewage treatment plant in Siedlce, applied at a starter dose of 2 g C kg^{-1} soil.

Nickel in the form of $NiCl_2$ $6H_2O$ water solution, liming with $CaCO_3$ and sewage sludge were applied to the soil from April to May 2006.

The study material (Table 1) was collected from the 0-20 cm horizon of lessive soil, whose texture was typical of loamy sand (Kuziemska, Kalembasa 2008). The chemical composition of the sewage sludge is presented in Table 2.

The following determinations were made in the soil: hydrolytic acidity according to PN-R-04027:1997, pH by potentiometric method according to PN-ISO 10390:1997, the content of organic carbon by the oxidation-titration method according to PN-ISO 14235:2003, total nitrogen content by elemental analysis on a Perkin-Elmer Series II 2400 CHN analyzer with a thermal conductivity detector (TCD), the content of available phosphorus and potassium

Table 1

Some properties of soil used in the pot experiment

Hh cmol(+) kg^{-1}	pH 1 M KCl	$C_{org.}$	$N_{tot.}$	Available		Total
		g kg^{-1} of soil		P	K	Ni
				mg kg^{-1} of soil		mg kg^{-1} of soil
2.22	5.6	7.900	0.980	69.00	75.00	5.670

Table 2

Chemical composition of sewage sludge from Siedlce

Component	(g kg^{-1} d.m.)	Component	(m gkg^{-1}d.m.)
N	60.50	Cd	1.990
P	31.20	Pb	50.50
K	4.280	Ni	20.60
Ca	39.60	Fe	10850
Mg	8.420	Cu	137.7
$C_{org.}$	371.0	Zn	1276.8
Organic matter	640.0	Cr	30.14
Dry matter (g k g^{-1})		180.0	

by the Egner-Riehm method according to PN-R-040022:1996, and the total nickel content by atomic emission spectrometry with inductively coupled plasma (ICP-AES) on a Perkin-Elmer Optima 3200 RLspectrometer, after digestion in a mixture of perchloric acid and nitric acid (1:2) (Kopec, Gondek 2002).

The sludge used in the experiment was analyzed by the same methodology as in the analysis of soil. Additionally, the dry matter content was determined by the drying-weight method according to PN-EN 12880:2004, while the content of phosphorus, potassium, calcium, magnesium and heavy metals was aassessed by atomic emission spectrometry after dry digestion in a muffle furnace and dissolution of the ash in 10% HCl solution.

The test plant was cocksfoot (*Dactylisglomerata* L.) harvested four times (four swaths) in each plant growing season (every 30 days).

Table 3

A diagram of the BCR metal sequential extraction method

Fraction	Name of fractions	Extraction reagents	pH
F_1	exchangeable and acid soluble	0.1 M CH_3COOH	3.0
F_2	reducible	0.5 M NH_2OHHCl	1.5
F_3	oxidizable	8.8 M H_2O_2 + 1M CH_3COONH_4	2.0
F_4	residual	calculated as difference between total content and sum of three previously separated fractions	-

The soil was analyzed each year of the experiment, after the last grass swath. The total content of nickel was determined with the ICP-AES method and its fractions with 3-step sequential fractionating proposed by the Community Bureau of Reference – BCR (Rauret et al. 1999). The scheme of the procedure is given in Table 3.

RESULTS AND DISCUSSION

Evaluation of the status of nature by measuring the total content of metals in certain compartments of the natural environment (soil, natural and organic fertilizers) very often omits factors that determine the mobility and bioavailability of elements. A reliable assessment of risks to the environment requires determination of individual forms and fractions that constitute the total content of a given metal (Węglarzyk 2001, Wójcikowska-Kapusta, Niemczuk 2009).

Our studies demonstrated that the total content of selected metals in waste organic material used in the experiment (Table 2) did not exceed the permissible limits (The Regulation of the Minister of the Environment). The applied sewage sludge contained large amounts of organic matter and basic plant nutrients, which is why it should be used as fertilizer.

The total content of nickel, its distribution in the individual fractions and their percentages in soil sampled each year after cultivation of the test plant are presented in Tables 4-9. In all experimental years, the lowest total mean content of nickel was detected in the soil of control objects (without application of nickel) and the highest concentration was measured in the soil supplemented with 100 mg $Ni \cdot kg^{-1}$ soil.

The use of nickel in the mineral form contributed to an increase in its total content as well as in all determined fractions. This correlation was confirmed in the subsequent years of the experiment, when - in the objects treated with nickel – the nickel content in the exchangeable fraction (F_1) decreased while increasing in the residual fraction (F_4), which indicated its immobilization by different soil-associated factors. In the first year of the experiment,

Table 4

The content (mg kg^{-1} of soil) of nickel in fractions determined by the BCR method in the analyzed soil after the first year of cultivation

Fertilization	Fractions	Liming					
		0 Ca			Ca to 1 Hh		
		dose of nickel (mg kg^{-1} of soil)			dose of nickel (mg kg^{-1} of soil)		
		0	50	100	0	50	100
Without organic fertilization	F_1	0.530	39.20	73.90	0.400	31.00	57.20
	F_2	0.760	8.290	13.60	0.610	6.120	10.20
	F_3	1.120	3.980	10.80	1.010	4.220	9.840
	F_4	3.210	3.830	7.400	3.610	14.06	28.46
Sum of fractions	Σ	5.620	55.30	105.3	5.630	55.40	105.7
Sludge from Siedlce	F_1	0.380	30.30	54.60	0.300	25.30	46.80
	F_2	0.920	6.700	13.12	0.840	6.050	10.12
	F_3	2.000	11.20	20.42	1.820	9.020	14.92
	F_4	2.440	7.600	11.86	2.790	15.53	34.06
Sum of fractions	Σ	5.740	55.80	105.9	5.750	55.90	105.9
			F_1	F_2	F_3	F_4	Σ
$LSD_{(0.05)}$ for:							
doses of nickel			12.28	2.010	9.947	13.92	0.310
sludge from Siedlce			n.s.	1.545	n.s.	10.06	n.s.
liming			n.s.	n.s.	3.648	n.s.	0.228

n.s.–not significant

Table 5

The percentage share of the nickel fraction in the analyzedsoil after the first year of cultivation

Fertilization	Fractions	Liming					
		0 Ca			Ca to 1 Hh		
		dose of nickel (mg kg^{-1} of soil)			dose of nickel (mg kg^{-1} of soil)		
		0	50	100	0	50	100
Without organic fertilization	F_1	9.430	70.89	69.92	7.100	55.96	54.11
	F_2	13.52	14.99	12.87	10.83	11.05	9.650
	F_3	19.93	7.200	10.22	17.94	7.620	9.310
	F_4	57.12	6.920	6.990	64.13	25.37	26.93
Sum of fractions	Σ	100.0	100.0	100.0	100.0	100.0	100.0
Sludge from Siedlce	F_1	6.620	54.30	51.55	5.230	45.26	44.19
	F_2	16.03	12.01	12.39	14.61	10.82	9.560
	F_3	34.84	20.07	19.28	31.65	16.14	14.09
	F_4	42.51	13.62	16.78	48.51	27.78	32.16
Sum of fractions	Σ	100.0	100.0	100.0	100.0	100.0	100.0

Table 6

The content (mg kg-1 of soil) of nickel in fractions determined by the BCR method in the analyzed soil after the second year of cultivation

Fertilization	Fractions	Liming					
		0 Ca			Ca to 1 Hh		
		dose of nickel (mg kg-1 of soil)			dose of nickel (mg kg-1 of soil)		
		0	50	100	0	50	100
Without organic fertilization	F_1 F_2 F_3 F_4	0.620 0.830 1.180 2.850	36.20 9.020 4.420 5.260	66.80 11.21 9.890 17.30	0.430 0.900 1.000 3.170	25.12 4.980 5.520 19.38	48.10 10.28 11.08 35.44
Sum of fractions	Σ	5.480	54.90	105.2	5.500	55.00	104.9
Sludge from Siedlce	F_1 F_2 F_3 F_4	0.400 0.990 2.370 1.880	26.12 8.950 16.32 3.910	45.12 14.00 28.12 18.34	0.330 0.560 1.140 3.630	22.06 7.280 12.75 13.31	43.08 10.12 21.08 31.12
Sum of fractions	Σ	5.640	55.30	105.6	5.660	55.40	105.4
$LSD_{(0.05)}$ for:			F_1	F_2	F_3	F_4	Σ
doses of nickel			11.80	2.628	8.985	9.916	0.302
sludge from Siedlce			n.s.	n.s.	n.s.	7.436	n.s.
liming			n.s.	n.s.	7.162	n.s.	0.177

n.s.–not significant

Tabela 7

The percentage share of the nickel fractions in the analyzed soil after the second year of cultivation

Fertilization	Fractions	Liming					
		0 Ca			Ca to 1 Hh		
		dose of nickel (mg kg-1 of soil)			dose of nickel (mg kg-1 of soil)		
		0	50	100	0	50	100
Without organic fertilization	F_1 F_2 F_3 F_4	11.31 15.14 21.53 52.02	65.94 16.43 8.060 9.570	63.50 10.66 9.100 16.44	7.820 16.36 18.18 57.64	45.67 9.050 10.04 35.24	45.83 9.800 10.56 33.81
Sum of fractions	Σ	100.0	100.0	100.0	100.0	100.0	100.0
Sludge from Siedlce	F_1 F_2 F_3 F_4	7.090 17.55 42.02 33.34	47.23 16.18 29.51 7.080	42.72 13.26 26.64 17.38	5.830 9.890 20.14 64.14	39.81 13.14 23.01 24.04	40.87 9.60. 20.00 29.53
Sum of fractions	Σ	100.0	100.0	100.0	100.0	100.0	100.0

Table 8

The content (mg kg^{-1} of soil) of nickel in fractions determined by the BCR method in the analyzed soil after the third year of cultivation

Fertilization	Fractions	Liming					
		0 Ca			Ca to 1 Hh		
		dose of nickel (mg kg^{-1} of soil)			dose of nickel (mg kg^{-1} of soil)		
		0	50	100	0	50	100
Without organic fertilization	F_1 F_2 F_3 F_4	0.440 0.980 1.080 2.900	31.12 10.15 4.460 8.790	60.12 12.56 8.040 23.88	0.350 1.020 1.000 3.050	22.16 5.380 6.210 20.85	43.60 10.08 9.460 41.66
Sum of fractions	Σ	5.400	54.60	104.9	5.420	54.60	104.8
Sludge from Siedlce	F_1 F_2 F_3 F_4	0.320 1.060 2.510 1.670	23.68 10.21 14.12 6.990	42.80 12.84 23.10 26.36	0.300 0.640 1.390 3.250	20.09 6.080 10.86 17.97	36.68 9.860 18.96 39.50
Sum of fractions	Σ	5.560	55.00	105.1	5.580	55.00	105.0
			F_1	F_2	F_3	F_4	Σ
$LSD_{(0.05)}$ for:							
doses of nickel			10.87	2.136	7.315	17.43	0.180
sludge from Siedlce			n.s.	2.035	n.s.	n.s.	n.s.
liming			n.s.	n.s.	5.903	n.s.	0.121

n.s.–not significant

Table 9

The percentage share of the nickel fractions in the analyzed soil after the third year of cultivation

Fertilization	Fractions	Liming					
		0 Ca			Ca to 1 Hh		
		dose of nickel (mg kg^{-1} of soil)			dose of nickel (mg kg^{-1} of soil)		
		0	50	100	0	50	100
Without organic fertilization	F_1 F_2 F_3 F_4	8.150 18.15 20.00 53.70	57.00 18.59 8.170 16.24	57.31 11.97 7.660 23.06	6.460 18.82 18.45 56.27	40.59 9.860 11.37 38.18	41.60 9.620 9.030 39.75
Sum of fractions	Σ	100.0	100.0	100.0	100.0	100.0	100.0
Sludge from Siedlce	F_1 F_2 F_3 F_4	5.750 19.06 45.14 30.05	43.05 18.56 25.67 12.72	40.72 12.22 21.98 25.08	5.370 11.47 24.91 58.25	36.53 11.05 19.75 32.67	34.98 9.390 18.06 37.62
Sum of fractions	Σ	100.0	100.0	100.0	100.0	100.0	100.0

in the objects where nickel was applied at 50 mg Ni·kg^{-1} of soil, the average percentage of the exchangeable fraction (F_1) in its total content was 56.6%, in the second year it was 49.66%, whereas in the third year it was only 44.29%. In the objects treated with 100 mg Ni·kg^{-1} of soil, the values were 54.94%, 48.23% and 43.64% in the first, second and third year, respectively.The average percentage of nickel in its total content in the extracted fractions of soil into which this metal had not been introduced, in the subsequent years of the experiment, was arranged in the following order of decreasing values: $F_4>F_3>F_2>F_1$.

Throughout the study, the total content of nickel depended not only on its amount introduced into soil as $NiCl_2$ $6H_2O$, but also on the organic matter in added waste material. The application of sewage sludge resulted in a minor, yet statistically significant, increase in the total content of nickel in soil despite the fact that its concentration in the sewage sludge was only 20.6 mg Ni kg^{-1} d.m. (Table 2). Similar results related to the impact of sewage sludge on the concentration of nickel and other metals in soil were reported by Filipek-Mazur et al. (1999) and Iżewska et al. (2009).

Regardless of the time of sampling, the sewage sludge used in our study contributed to an increase in the total content of nickel and in its proportion in the oxidizable fraction (F_3), which confirmed the positive impact of organic matter on limiting its mobility and was consistent with the studies conducted by Gondek, Filipek-Mazur (2003) and Latosińska, Gawdzik (2011). Organic matter contained in the sludge created organometallic compounds with mobile forms of nickel. These compounds are difficult to mineralize and therefore not available for plants and do not cause pollution of soil (Jakubus 2012).

The study did not reveal any significant impact of liming on the total content of this metal, but in the first and third year of the experiment it caused some “immobilization of nickel”, which was confirmed by a decrease in its content in the reducible fraction (F_2) and an increase in its concentration in the residual fraction (F_4) in the first and second year of the experiment. The study did not show any significant impact of liming on the amount of nickel in the exchangeable fraction (F_1), but there was a tendency towards a decrease in the percentage of this fraction under the influence of this factor. This correlation was observed in each year of the experiment.

The values of the soil pH in three consecutive years are presented in Table 10. The limed soil had a significantly higher pH than the non-limed soil. The other two experimental factors did not have any significant impact on the discussed parameter. The analysis of correlation did not reveal any significant relationship between pH and distribution of nickel in the extracted fractions, regardless of the time of sampling. This relationship is not consistent with the work of other authors and previously conducted studies, which demonstrated a significant effect of soil acidification on the content of mobile forms of nickel (Chen, Wong 2006, Siebielec et al. 2007, Kucharski et al. 2009). This issue requires further study.

Table 10

pH of soil in 1 M KCl

Fertilization	Years	Liming					
		0 Ca			Ca to 1 Hh		
		dose of nickel (mg kg^{-1} of soil)			dose of nickel (mg kg^{-1} of soil)		
		0	50	100	0	50	100
Without organic fertilization	I II III	6.640 6.520 5.970	6.580 6.490 5.840	6.120 6.010 5.800	7.460 7.400 6.640	7.400 7.330 6.590	7.380 7.350 6.800
Sludge from Siedlce	I II III	6.780 6.710 5.990	6.690 6.520 5.800	6.680 6.530 5.800	7.480 7.450 6.600	7.460 7.260 6.710	7.420 7.260 6.720
		Years	I	II	III		
$LSD_{(0.05)}$ for:							
doses of nickel			n.s.	n.s.	n.s.		
sludge from Siedlce			0.184	0.352	0.165		
liming			n.s.	n.s.	n.s.		

n.s.–not significant

Summarizing the results of our study, it is concluded that the addition of nickel into the soil, liming and application of sewage sludge modified the total content of nickel in the soil and its proportion in the fractions extracted with the BCR procedure. The introduction of nickel into the soil resulted in an increase in its total content and, more importantly, in its percentage in the exchangeable fraction (F_1) directly absorbed by plants, which poses a risk to the growth and development of cultivated plants (Spiak et al. 2003, Kuziemska, Kalembasa 2008). It should be taken into consideration that the amount of nickel in fraction F_1 decreased in the consecutive years of the experiment, especially in soils enriched with nickel in doses of 50 and 100 mg kg^{-1}, which confirms its immobilization.Liming reduced the mobility of nickel by decreasing its content in the reducible fraction (F_2) and increasing its proportion in the residual fraction (F_4), which further confirms the data reported in numerous publications indicating that liming is one of the factors limiting the mobility of metals (Kalembasa, Kuziemska 2010).

The application of sewage sludge resulted in an increase in the total content of nickel in the soil and its proportion in the fraction bound to organic matter (F_3), which limited its availability to plants.

Similar correlations were reported by Gondek, Filipek-Mazur (2003) who found that nickel and other metals were bound by humus in soil polluted with urban contamination.

Lime and waste organic material (i.e. sewage sludge) were found to be effective in reducing the mobility of nickel in soil contaminated with this metal.

CONCLUSIONS

1. The total content of heavy metals in the sewage sludge used in the experiment did not exceed the permissible limits.

2. The application of $NiCl_2 6H_2O$ into the soil resulted in an increase in its total content and in the individual fractions as well as an increase in the percentage of the exchangeable fraction in its total content.

3. Liming contributed to an increase in the nickel content in the residual fraction (F_4) and reduced its proportion in the exchangeable fraction (F_1)

4. The sewage sludge limited the mobility of nickel by increasing its content in the oxidizable fraction (F_3).

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