
EFFECT OF SULFUR FORMS ON CONCENTRATIONS OF CADMIUM AND NICKEL SOLUBLE IN 1 MOL HCl dm⁻³ IN ARABLE SOILS

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Abstract

Heavy metal contamination of arable soils remains one of the world's most serious environmental problems. The mobility of heavy metals in soil is determined by soil conditions, environmental factors and human activity. The immediate and residual effects of sulfur fertilization on the concentrations of heavy metals in soil are an important consideration when monitoring changes in environmental conditions in agricultural areas.

The objective of this study was to determine the effect of increasing doses of sulfate and elemental sulfur on changes in the concentrations of cadmium and nickel soluble in 1 mol HCl dm⁻³ in soil samples collected at a depth of 0-40 and 40-80 cm. A three-year field experiment was conducted on Dystric Cambisols (FAO) of the texture of heavy loamy sand. Soil samples were collected from each plot, prior to the establishment of the trials, after each harvest and before sowing the consecutive crop. The soil samples were used to determine the concentrations of Cd and Ni in soil (extractions with 1 mol HCl dm⁻³, the ratio between soil and extraction - 1:10) by the AAS method using a Shimadzu AA apparatus.

Irregular changes in soluble cadmium concentrations were observed in soil fertilized with sulfate and elemental sulfur. No distinct trends were noticed in response to sulfate and elemental sulfur fertilization. The effect of sulfur was noticeable only in the third year of the experiment. Sulfur application contributed to a decrease in the soluble nickel content of soil. Sulfate and elemental sulfur fertilization did not increase the levels of cadmium and nickel soluble in 1 mol HCl dm⁻³. The soil can be used for growing high-quality horticultural and agricultural crops safe for human consumption.

Key words: fertilizer, sulfate sulfur, elemental sulfur, cadmium, nickel, soil.

OCENA WPŁYWU FORM SIARKI NA ZMIANY ZAWARTOŚCI KADMU I NIKLU ROZPUSZCZALNYCH W 1 MOL HCl dm⁻³ W GLEBIE UŻYTKOWANEJ ROLNICZO

Abstrakt

Zanieczyszczenie metalami ciężkimi gleb użytkowanych rolniczo to jeden z problemów środowiskowych w wielu częściach świata. Określenie możliwości uruchamiania metali ciężkich jest procesem złożonym zależnym od czynników glebowych, środowiskowych i działalności człowieka. Zbadanie bezpośrednich i następczych efektów nawożenia siarką na zmiany zawartości metali ciężkich w glebie ma znaczenie dla monitorowania zmian przyrodniczych warunków na obszarach rolniczych.

Celem badań była ocena wpływu nawożenia wzrastającymi dawkami siarki siarczanowej i elementarnej na zmiany zawartości form kadmu i niklu rozpuszczalnych w 1 mol HCl dm⁻³ w dwóch poziomach gleby: 0-40 i 40-80 cm. Trzyletnie doświadczenie polowe założono na glebie brunatnej, kwaśnej o składzie granulometrycznym piasku gliniastego mocnego. Glebę do analiz chemicznych pobierano wiosną i jesienią. Formy rozpuszczalne kadmu i niklu ekstrahowano z gleby roztworem 1 mol HCl dm⁻³, a ich zawartość oznaczono metodą absorpcyjnej spektrometrii atomowej. Wyniki analiz chemicznych gleby opracowano statystycznie metodą analizy wariancji.

Po nawożeniu siarką siarczanową i elementarną zmiany zawartości form rozpuszczalnych kadmu w glebie przebiegały nieregularnie. Nie uwidoczniła się żadna tendencja w zmianie zawartości omawianej formy pierwiastka. Działanie siarki uwidoczniło się dopiero w trzecim roku trwania eksperymentu. Zastosowanie siarki wpłynęło na zmniejszenie zawartości form rozpuszczalnych niklu w glebie. Nawożenie siarką siarczanową i elementarną nie spowodowało zwiększenia zawartości form kadmu i niklu rozpuszczalnych w 1 mol HCl dm⁻³. Gleba nadaje się do uprawy żywności o wysokim stopniu bezpieczeństwa. Może być zagospodarowana pod uprawę roślin ogrodniczych i rolniczych.

Słowa kluczowe: nawożenie, siarka siarczanowa, siarka elementarna, Cd, Ni, gleba.

INTRODUCTION

The mobility of heavy metals in soil is determined by soil conditions, environmental factors and human activity. The bioavailability and desorption of heavy metals are affected by soil properties, including organic matter content, pH, iron oxide content, and redox conditions. Soil pH usually exerts the strongest effect due to the solubility and speciation of heavy metals in the soil solution (MÜHLBACHOVÁ et al. 2005, JIAN-LING et al. 2010).

Soil is a non-renewable resource and it should be adequately protected and used rationally to produce high quality crops (KAYA et al. 2009), which is often ignored when industrialization and agricultural development become the top priorities. According to NEDERLOF and RIEMSOLIJK (1995), TEMMINGHOFF et al. (1997), FÄSSLER et al. (2010a,b) and VIOLANTE et al. (2010), the toxicity of heavy metals and their availability to plants increase due to soil acidification caused by sulfur deposition. KAYSER et al. (2000) demonstrated that the application of elemental sulfur and a decrease in soil pH increased heavy

metal solubility in soil. In the Province of Warmia and Mazury, average heavy metal concentrations in arable soils are considerably lower than in other regions of Poland. The above soils have a natural (0°) heavy metal content (TERELAK et al. 2001).

Previous research findings into the residual effects of sulfur on agricultural ecosystems have been inconsistent and contradictory, which prompted us to carry out the present experiment. The objective of this study was to determine the effect of increasing doses of sulfate and elemental sulfur on changes in the concentrations of cadmium and nickel soluble in 1 mol HCl dm^{-3} in soil samples collected at a depth of 0-40 and 40-80 cm.

MATERIAL AND METHODS

A three-year field experiment was conducted in North-East Poland from 2000 to 2002. The exact location was far from larger industrial plants emitting sulfur compounds or any big cities. The soil content of sulfur in the soil was not caused by human activity.

The trial was set up on Dystric Cambisols (FAO) of the texture of heavy loamy sand. Initially, the soil had the following properties: $\text{pH}_{(\text{KCl})} = 5.30$, mineral nitrogen 24.0, sulfate sulfur 4.10, available phosphorus 34.5 and potassium 110.0 mg kg^{-1} of soil. The annual rates of sulfate sulfur ($\text{SO}_4^{2-}\text{-S}$) and elemental sulfur ($\text{S}^0\text{-S}$) were: $\text{S}_1 - 40$, $\text{S}_2 - 80$ and $\text{S}_3 - 120 \text{ kg ha}^{-1}$. Air-dry soil was passed through a 1 mm mesh sieve.

A permanent experiment was established in a random block design and consisted of eight fertilization treatments with four replications: 1) unfertilized control, 2) NPK, 3) NPK + $\text{S}_1\text{-SO}_4$, 4) NPK + $\text{S}_2\text{-SO}_4$, 5) NPK + $\text{S}_3\text{-SO}_4$, 6) NPK + $\text{S}_1\text{-S}^0$, 7) NPK + $\text{S}_2\text{-S}^0$, 8) NPK + $\text{S}_3\text{-S}^0$. The following compounds were applied to introduce given elements: ammonium nitrate (34% N) or ammonium sulphate (20.5% N), triple superphosphate (20.1% P), potassium salt (49.8% K) or potassium sulphate (43.7% K), potassium sulphate (17% S), ammonium sulphate (24% S) or elemental sulfur. The NPK rates (Table 1) depended on the crop species and soil fertility. No heavy metals were added to the soil during the experiment.

Table 1

Applied doses of NPK in the experiment

Year	kg (ha^{-1})		
	N	P	P
2000	200.0	52.5	180.0
2001	160.0	60.0	183.0
2002	90.0	80.0	111.0

Soil samples were collected from each plot at 0-40 and 40-80 cm depths prior to the onset of the trials, after each harvest and before sowing the consecutive crop. Air-dry soil was passed through a 1 mm mesh sieve. The soil samples were used to determine the concentrations of Cd and Ni in soil (extractions with 1 mol HCl dm⁻³, the ratio between soil and extraction – 1:10) by the AAS method using a Shimadzu AA apparatus.

The results of the yields and chemical analysis of soil were processed statistically with the analysis of variance for a two-factorial experiment in a random block design, using the form of sulfur as factor *a* and rate of sulfur as factor *b*.

RESULTS AND DISCUSSION

A three-year field experiment was carried out to determine the effect of fertilization with sulfate or elemental sulfur at doses of 40, 80 and 120 kg ha⁻¹ on concentrations of cadmium and nickel soluble in 1 mol HCl dm⁻³ in soil samples collected from 0-40 cm and 40-80 cm soil horizons.

Changes in the soil pH resulting from sulfate and elemental sulfur fertilization have been described in detail by SKWIERAWSKA et al. (2008). Over the three-year experimental period, the application of sulfate and elemental sulfur significantly affected soil pH in the 0-40 cm horizon. When applied in a dose of 120 kg, S-SO₄²⁻ and S-S⁰ caused a significant decrease in the soil pH in comparison with the control treatment. In the deeper soil layer, pH values remained relatively stable and lower than in the topsoil in the corresponding treatments. The results of Duncan's test at a significance level of 0.05 show that during the three years the average soil pH was the lowest in the treatment with a triple sulfate dose, and the differences relative to the other doses were statistically significant.

Cadmium is one of the most toxic and mobile metallic elements (BASTA et al. 2005, ZHAO, MASAIHIKO 2007). Before the experiment, the content of cadmium soluble in 1 mol HCl dm⁻³ in both sampled soil horizons (0-40 cm and 40-80 cm) was comparable, but slightly lower in the deeper layer than in the topsoil (Tables 2 and 3). Similar trends were observed in the first and second year of the study, when neither the sulfur form nor its dose exerted a significant effect on changes in cadmium concentrations in the soil. The impact of sulfur was not observed until the third year. At the end of the experiment, the soluble cadmium content of the 0-40 cm soil layer increased in the treatments fertilized with sulfate and elemental sulfur at a dose of 40 kg ha⁻¹. In the 40-80 cm horizon, sulfate and elemental sulfur had no significant effect on soluble cadmium levels in soil (Table 3). Before sowing spring barley, the cadmium content of soil increased insignificantly, particularly in the treatment fertilized with 120 kg S-SO₄²⁻ ha⁻¹. After three years

Table 2

Effect of different rates and forms of sulfur on the content of cadmium soluble
in 1 mol HCl dm⁻³ in soil at 0-40 cm depth (mg Cd kg⁻¹ soil)

Treatments	Before experiment	After cabbage harvest	Before onion sowing	After onion harvest	Before barley sowing	After barley harvest
0	0.080	0.090	0.054	0.072	0.055	0.096
NPK	0.085	0.095	0.055	0.072	0.094	0.119
NPK+ S ₁ -SO ₄ ²⁻	0.080	0.087	0.072	0.078	0.113	0.132
NPK+ S ₂ -SO ₄ ²⁻	0.089	0.098	0.048	0.063	0.115	0.105
NPK+ S ₃ -SO ₄ ²⁻	0.077	0.087	0.058	0.061	0.053	0.116
NPK+S ₁ -S ⁰	0.069	0.072	0.047	0.076	0.095	0.120
NPK+S ₂ -S ⁰	0.089	0.091	0.062	0.045	0.064	0.093
NPK+S ₃ -S ⁰	0.096	0.111	0.039	0.077	0.070	0.119
LSD _{0.05}						
<i>a</i>	n.s.	n.s.	n.s.	n.s.	0.0163	n.s.
<i>b</i>	n.s.	n.s.	n.s.	n.s.	0.0230	n.s.
<i>a x b</i>	n.s.	n.s.	n.s.	n.s.	0.0326	0.0274

SO₄²⁻ – sulfate sulfur; S⁰ – elementary sulfur; S₁ – 40 kg ha⁻¹, S₂ – 80 kg ha⁻¹,
S₃ – 120 kg ha⁻¹, *a* – form of sulfur, *b* – dose of sulfur,

a x b interaction,

*n.s. – non-significant difference

Table 3

Effect of different rates and forms of sulfur on the content of cadmium soluble
in 1 mol HCl dm⁻³ in soil at 40-80 cm depth (mg Cd kg⁻¹ soil)

Treatments	Before experiment	After cabbage harvest	Before onion sowing	After onion harvest	Before barley sowing	After barley harvest
0	0.037	0.047	-	0.070	0.045	0.013
NPK	0.036	0.046	-	0.072	0.127	0.029
NPK+ S ₁ -SO ₄ ²⁻	0.038	0.038	-	0.078	0.082	0.033
NPK+ S ₂ -SO ₄ ²⁻	0.039	0.042	-	0.063	0.046	0.021
NPK+ S ₃ -SO ₄ ²⁻	0.032	0.032	-	0.061	0.113	0.030
NPK+S ₁ -S ⁰	0.036	0.028	-	0.076	0.067	0.039
NPK+S ₂ -S ⁰	0.035	0.035	-	0.045	0.092	0.017
NPK+S ₃ -S ⁰	0.031	0.033	-	0.077	0.079	0.021
LSD _{0.05}						
<i>a</i>	n.s.	n.s.	-	n.s.	n.s.	n.s.
<i>b</i>	n.s.	n.s.	-	n.s.	n.s.	n.s.
<i>a x b</i>	n.s.	n.s.	-	n.s.	n.s.	n.s.

Key: cf. Table 2

of sulfate and elemental sulfur fertilization, cadmium concentrations decreased, which could have been due to the cadmium uptake by plants. FÄSSLER et al. (2010A) amended contaminated soil with elemental sulfur and ammonium sulfate to increase heavy metal bioavailability. As expected, elemental sulfur decreased the soil pH and increased its cadmium content. In the long term, sulfur fertilization contributed to an increased cadmium uptake by tobacco plants. Due to its high mobility, cadmium may be easily absorbed by plants (GONDEK 2010). As demonstrated by McLAUGHLIN et al. (1998), an increase in Na_2SO_4 doses is followed by an increase in the concentrations of active cadmium forms in the soil solution. Such an increase was also noted by KAYSER et al. (2001) after soil amendment with elemental sulfur.

Before the experiment, the content of nickel soluble in 1 mol HCl dm^{-3} in the 0-40 cm soil layer varied from 0.68 to 1.45 mg kg soil (Table 4).

Table 4

Effect of different rates and forms of sulfur on the content of nickel soluble in 1 mol HCl dm^{-3} in soil at 0-40 cm depth (mg Ni kg^{-1} soil)

Treatments	Before experiment	After cabbage harvest	Before onion sowing	After onion harvest	Before barley sowing	After barley harvest
0	1.34	1.44	0.78	0.95	0.80	0.367
NPK	0.89	0.93	1.01	1.15	0.56	0.592
NPK+ S ₁ -SO ₄ ²⁻	0.68	0.68	1.29	1.28	0.55	0.505
NPK+ S ₂ -SO ₄ ²⁻	1.35	1.35	1.13	1.11	0.82	0.556
NPK+ S ₃ -SO ₄ ²⁻	1.45	1.65	0.80	0.89	0.92	0.501
NPK+S ₁ -S ⁰	0.83	0.85	1.36	0.93	0.65	0.539
NPK+S ₂ -S ⁰	1.09	1.11	0.73	0.99	0.57	0.470
NPK+S ₃ -S ⁰	1.00	1.09	1.51	0.97	0.76	0.516
LSD _{0.05}						
a	n.s	n.s.	n.s.	0.1305	n.s.	n.s
b	0.2191	0.2191	0.2460	n.s.	0.0962	0.0974
a x b	0.3098	0.3098	0.3479	n.s.	n.s.	n.s

Key: cf. Table 2

In the first year of the study, the nickel content of the 0-40 cm horizon increased with increasing sulfate doses. The experimental factors had no significant influence on changes in nickel concentrations in the 40-80 cm soil layer (Table 5).

In the spring, in the second year of the study, sulfur fertilization at all applied doses contributed to an increase in the soluble nickel content of the 0-40 cm soil layer. Sulfur form had no significant effect on nickel concentra-

Table 5

Effect of different rates and forms of sulfur on the content of nickel soluble in 1 mol HCl dm⁻³ in soil at 40-80 cm depth (mg Ni kg⁻¹ soil)

Treatments	Before experiment	After cabbage harvest	Before onion sowing	After onion harvest	Before barley sowing	After barley harvest
0	0.80	0.90	-	0.31	0.39	0.178
NPK	1.13	1.14	-	0.34	0.30	0.413
NPK+ S ₁ -SO ₄ ²⁻	1.35	1.35	-	0.64	0.35	0.306
NPK+ S ₂ -SO ₄ ²⁻	1.09	1.11	-	0.63	0.17	0.290
NPK+ S ₃ -SO ₄ ²⁻	1.01	1.18	-	0.64	0.32	0.265
NPK+S ₁ -S ⁰	1.30	1.37	-	0.48	0.29	0.433
NPK+S ₂ -S ⁰	0.98	0.99	-	0.71	0.30	0.162
NPK+S ₃ -S ⁰	1.01	1.01	-	0.66	0.19	0.178
LSD _{0.05}						
<i>a</i>	n.s.	n.s.	-	n.s.	n.s.	n.s.
<i>b</i>	n.s.	n.s.		n.s.	n.s.	n.s.
<i>a</i> x <i>b</i>	n.s.	n.s.		n.s.	n.s.	n.s.

Key: cf. Table 2

tions in soil. After onion harvest, the application of sulfate sulfur resulted in an increase in nickel concentrations in the 0-40 cm horizon, compared with elemental sulfur. Increasing doses of sulfate sulfur led to a minor decrease in the concentrations of nickel soluble in 1 mol HCl dm⁻³ in soil. Changes in the nickel content of the 40-80 cm soil layer were not affected by sulfur dose or form.

In the third year, before sowing spring barley, the concentrations of nickel soluble in 1 mol HCl dm⁻³ in the 0-40 cm horizon were in most cases lower than in the corresponding treatments in the previous two years. The application of 120 kg ha⁻¹ of both sulfur forms led to an increase in the nickel content of soil in comparison with the other sulfur doses. This could have resulted from the drop in the soil pH, which increased the nickel content and bioavailability. Sulfur decreases the pH of soil and increases the solubility, availability and mobility of heavy metals (MARTINEZ et al. 2000, CUI et al. 2004).

Sulfur fertilization generally increased soluble nickel concentrations in the soil, relative to the NPK treatment. In the deeper soil layer, neither the sulfur form nor its dose had a significant effect on changes in nickel concentrations.

At the end of the experiment, the soluble nickel content of the 0-40 cm soil layer ranged from 0.367 to 0.592 mg kg soil. A decrease in soluble nickel levels was observed in comparison with the previous two years. The ap-

plication of elemental sulfur at a dose of 80 kg led to a decrease in the concentration of nickel soluble in 1 mol HCl dm⁻³ compared with the NPK treatment, probably due to an increased nickel uptake by the tested plants. Our results corroborate the findings of SUMMERS et al. (2003) and HOLAH et al. (2010) in this respect. Sulfur fertilization had no significant influence on changes in soluble nickel concentrations in the 40-80 cm horizon (Table 5). However, a steady decrease in the concentrations of nickel soluble in 1 mol HCl dm⁻³ in soil was noted, compared with the corresponding treatments in the past two years.

CONCLUSIONS

1. Irregular changes in the concentrations of cadmium soluble in 1 mol HCl dm⁻³ were observed in soil fertilized with sulfate and elemental sulfur. The effect of sulfur was noticeable only in the third year of the experiment.

2. Sulfur application contributed to a decrease in the soluble nickel content of soil.

3. The applied sulfur doses had no significant effect on the concentrations of cadmium and nickel soluble in 1 mol HCl dm⁻³ in soil. The analyzed soil can be used for growing high-quality horticultural and agricultural crops safe for human consumption, particularly for infants and children.

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