The effect of different sulfur doses and forms on changes of soil heavy metals

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ABSTRACT

A three-year field experiment was conducted in North-East Poland. Each year three sulfur fertilization rates in the form of sulphate (S-SO$_4^{2-}$) and pure (S–S^0) sulfur were applied: 40, 80, and 120 kg/ha. After the third year of the study, the application of sulfate and elemental sulfur decreased the zinc content of 0–40 and 40–80 cm soil layers, as compared with soil sampled before the experiment. Over the entire experimental period, sulfur fertilization had no significant effect on changes in the natural copper content of soil at a depth of 0–40 and 40–80 cm. Manganese concentrations remained at a similar level at a soil depth of 0–40 cm. The manganese content of the 40–80 cm soil layer was substantially lower than in the 0–40 cm horizon. An insignificant increase in the lead content of soil was observed. The applied doses of sulfate and elemental sulfur led to an increase in the cadmium content of soil depth of 0–40 cm. Sulfur fertilization contributed to a decrease in the nickel content of soil. The applied doses of sulfate and elemental sulfur exerted a stronger effect in the 0–40 cm soil layer than in the 40–80 cm horizon.

Keywords: fertilizer; S-SO$_4^{2-}$; S-elemental; available forms; zinc; copper; manganese; lead; cadmium; nickel; interaction

The concentrations of soluble forms of heavy metals in soil are mostly determined by their total content and soil processes. Soil organic matter and pH are the key factors affecting the content and mobility of heavy metals in soil (Soliman et al. 1992, Martinez and Motto 2000, Borůvka and Drábek 2004, Šichorová et al. 2004). Another important consideration is human activity which contributes to soil contamination in some regions of Poland, thus leading to changes in the natural heavy metals content. In the 1980's SO$_2$ emissions in Poland were at about Gg/year, which provided an average of 132.1 kg of SO$_2$/ha during the year. In 2006, SO$_2$ emissions were reduced to 1195 Gg/year, i.e. 38.3 kg of SO$_2$/ha CSO (2008).

According to many authors (Nederlof and Riemsolijk 1995, Temminghoff et al. 1997), the toxicity of heavy metals and their availability to plants increase due to soil acidification caused by sulfur deposition. In a study by Kayser et al. (2000), the application of elemental sulfur and a decrease in soil pH increased the solubility of heavy metals.

Some elements, including zinc and copper, precipitate as sulfides and sulfates, to produce forms that are relatively immobile in the soil profile (Kabata-Pendias and Pendias 1992). Cadmium, on the other hand, can form inorganic ligands with S-SO$_4^{2-}$ (McLaughlin et al. 1996).

Direct and residual effect of sulfur fertilization on changes in the heavy metal content of soil has to be taken into account in environmental analyses in agricultural areas, including environmental impact assessments and predictions.

The purpose of the present study was to determine the effect of fertilization with increasing rates of sulfur applied in the form of sulphates and as elemental sulfur on the natural content of heavy metals in soil at the depth 0–40 cm and 40–80 cm.

MATERIAL AND METHODS

A three-year field experiment was conducted from 2000 to 2002, in North-East Poland. The

village is distant from larger industrial plants which emit sulfur compounds and lies far from any big cities. The concentration of sulfur in the soil was not caused by human activity.

The trial was set up on Dystric Cambisols (FAO), of the granulometric composition of heavy loamy sand. The initial soil had the following properties: $pH_{KCl} = 5.30$, mineral nitrogen 24.0, sulphate sulfur 4.10, available phosphorus 34.5 and potassium 110.0 mg/kg of soil. The annual rates of sulphate sulfur ($S-SO_4^{2-}-S$) and elemental sulfur (S^0-S) were: $S_1 - 40$, $S_2 - 80$ and $S_3 - 120$ kg/ha. Air-dry soil was passed through a 1 mm mesh sieve. The soil samples were used to determine soil pH in 1 mol KCl (the ratio between soil and extraction 1:2.5); total sulfur (Butters and Chenery 1959) and $S-SO_4^{2-}$ with the turbidimetric method (the ratio between soil and extraction 1:3); $N-NO_3^-$ by colorimetry using phenyl disulphonic acid (the ratio between soil and extraction 1:5); $N-NH_4^+$ was determined using the Nessler's reagent (the ratio between soil and extraction 1:5); available phosphorus and potassium was determined with the Enger Riehm's method (DL) – (the ratio between soil and extraction 1:50) – (Panak 1997).

The 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 + S_1-SO_4; (4) NPK + S_2-SO_4; (5) NPK + S_3-SO_4; (6) NPK + S_1-S^0; (7) NPK + S_2-S^0; (8) NPK + S_3-S^0.

Nitrogen in the form of ammonium nitrate or ammonium sulphate, phosphorus in the form of triple superphosphate, potassium in the form of potassium salt of 60% or in the form of potassium sulphate, sulfur in the form of potassium sulphate and ammonium sulphate supplementation as well as in the form of elemental sulfur. The NPK rates (Table 1) depended on the crop species and soil fertility.

Soil samples were collected from each plot, at 0–40 and 40–80 cm depths, prior to the establishment 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 samg ples were used to determine the concentrations: Zn, Cu, Mn, Pb, Cd, and Ni in soil (extractions with 1 mol HCl/dm^3, the ratio between soil and extraction – 1:10) was determined by AAS method using Schimadzu AA apparatus (model AA-6800, Kyoto, Japan).

The results of the yields and chemical analysis of soil were processed statistically with the analysis of variance for a two-factor experiment in a random block design, using the form of sulfur as factor a and rate of sulfur as factor b. Additional statistical analyses were performed with the software package Statistica 6.0 PL, to carry out analysis of regression with the Duncan's tests with an aim of determining statistical differences between sets of data.

RESULTS AND DISCUSSION

Soil pH. Results of soil reaction changes after sulfur fertilization are presented in the publication by Skwierawska et al. (2008a).

During the three years of the experiment, sulphate and elementary sulphur addition had a significant effect on changes in the soil pH at the depth of 0–40 cm. The rate of 120 kg $S-SO_4^{2-}$/ha and $S-S^0$ caused a significant decline in the soil pH versus the control object. At the deeper layer of soil (40–80 cm) the soil pH was in general more even and lower than in the top soil layer in the analogous objects.

Rate 120 kg $S-SO_4^{2-}$/ha and $S-S^0$ depressed soil pH in the 0–40 cm soil horizon. Acidification of soil caused by $S-SO_4^{2-}$ became evident already in the first year of the trials, while that produced by $S-S^0$ did not occur until the third year. The influence of sulphur on soil pH at the depth of 40–80 cm was irregular.

Zinc. Changes in the natural heavy metal content of soil fertilized with sulfate and elemental sulfur at a dose of 40, 80, and 120 kg/ha were studied over a three-year period (Table 2). Before the experiment, zinc concentrations ranged from 15.65 to 17.00 mg/kg in the 0–40 cm soil layer, and from 5.80 to 7.60 mg/kg in the 40–80 cm horizon.

At the end of the experiment, zinc concentrations in the 0–40 cm soil layer ranged from 2.92 to 3.99 mg/kg, irrespective of sulfur doses, and they were generally considerably lower than in the corresponding treatments in the first and second year of the study. This could be due to increased bioavailability of zinc. Kayser et al. (2001) demonstrated that the application of elemental sulfur increased zinc solubility in the soil and

Table 1. NPK rates applied in the trials

Crops	Year	N	P	K
		(kg/ha)		
Head cabbage	2000	200.0	52.5	180.0
Common onion	2001	160.0	60.0	183.0
Spring barley	2002	90.0	80.0	111.0

Table 2. Effect of different rates and forms of sulphur on heavy metals content in the soil layers at 0–40 and 40–80 cm depths, before and after experiment (mg/kg soil)

Treatments	Before experiment						After experiment					
	Zn	Cu	Mn	Pb	Cd	Ni	Zn	Cu	Mn	Pb	Cd	Ni
Horizon 0–40 cm												
0	17.00	1.80	89.00	5.26	0.080	1.34	2.92	1.72	85.75	5.50	0.096	0.367
NPK	16.18	1.90	106.25	5.00	0.085	0.89	2.95	1.63	88.66	5.59	0.119	0.592
NPK + S_1-SO_4^{2-}	16.61	1.90	109.00	5.43	0.080	0.68	3.16	1.57	107.41	5.99	0.132	0.505
NPK + S_2-SO_4^{2-}	15.65	1.80	101.74	5.16	0.089	1.35	3.44	1.78	91.32	4.66	0.105	0.556
NPK + S_3-SO_4^{2-}	16.15	1.76	106.32	5.55	0.077	1.45	3.94	1.56	93.44	5.95	0.116	0.501
NPK + S_1-S^0	15.89	2.00	101.89	4.78	0.069	0.83	3.69	1.53	93.19	6.11	0.120	0.539
NPK + S_2-S^0	16.12	1.50	101.15	4.91	0.089	1.09	3.30	1.58	90.16	5.81	0.093	0.470
NPK + S_3-S^0	15.82	1.60	99.74	4.29	0.096	1.00	3.99	1.72	91.80	6.05	0.119	0.516
$LSD_{0.05}$ a	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	0.2754	n.s.	n.s.	0.295	n.s	n.s.
b	n.s.	n.s.	n.s.	n.s.	n.s.	0.2191	n.s.	n.s.	6.776	n.s.	n.s.	0.0974
a × b	n.s.	0.299	n.s.	n.s.	n.s.	0.3098	n.s.	n.s.	9.583	0.591	0.0274	n.s.
Horizon 40–80 cm												
0	6.72	1.20	41.64	2.11	0.037	0.80	1.94	0.767	40.02	2.18	0.013	0.178
NPK	6.51	1.00	41.32	3.52	0.036	1.13	2.32	1.157	32.27	2.89	0.029	0.413
NPK + S_1-SO_4^{2-}	6.12	1.11	45.87	2.41	0.038	1.35	2.08	0.764	41.31	3.14	0.033	0.306
NPK + S_2-SO_4^{2-}	5.99	1.15	43.75	2.84	0.039	1.09	1.75	0.713	28.96	3.18	0.021	0.290
NPK + S_3-SO_4^{2-}	5.80	1.20	46.30	2.78	0.032	1.01	1.69	0.891	41.53	2.38	0.030	0.265
NPK + S_1-S^0	7.50	1.26	33.86	2.52	0.036	1.30	1.57	1.130	35.45	2.62	0.039	0.433
NPK + S_2-S^0	6.30	1.04	39.80	1.64	0.035	0.98	1.79	0.708	29.80	2.65	0.017	0.162
NPK + S_3-S^0	7.60	1.00	39.45	2.30	0.031	1.01	1.65	0.761	26.07	3.14	0.021	0.178
$LSD_{0.05}$ a	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	0.210	n.s.	n.s.	n.s.	n.s.	n.s.
b	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	7.9925	0.572	n.s.	n.s.
a × b	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.

a – form of sulphur; b – dose of sulphur; a × b – interaction, n.s. – no significant difference

utilization by plants. Kaya et al. (2009) reported that the application of sulfur and sulfur-containing waste resulted in decrease in soil pH, but it also increased the concentrations of nutrients available to plants, such as Zn, Cu and Mn. Different results were obtained by Abdou et al. (2011) who did not observe an increase in zinc availability to plants as a result of elemental sulfur fertilization. In the present study, zinc concentrations were lower in the 40–80 cm soil layer than in the 0–40 cm horizon, and they were significantly affected only by sulfur form. Elemental sulfur contributed to a higher decrease in zinc levels in the soil.

Copper. Before the experiment, the copper content of the 0–40 cm soil layer remained at a stable level in all treatments (Table 2). Copper concentrations tended to increase in the treatment with a single dose of elemental sulfur. In the 40–80 cm horizon, copper content ranged from 1.00 to 1.26 mg/kg soil.

In the first year of the study, sulfur fertilization had no significant effect on changes in the copper content of soil. In the second year, copper concentrations increased in the 0–40 cm soil layer, relative to the NPK treatment, in particular after sulfate application. This could result from changes in soil pH. Sulfur decreases soil pH and increases the solubility, availability and mobility of heavy metals (Tichý et al. 1997, Seidel et al. 1998, Kayser et al. 2000, Martinez et al. 2000, Cui

et al. 2004). In a study by Takáč et al. (2009), the content of mobile copper forms in the soil was not significantly affected by soil pH.

In the third year of the study, at the end of the experiment, copper concentrations in the 0–40 cm soil layer were comparable, regardless of sulfur forms and doses. A decrease in copper content was noted, compared with soil samples collected before the experiment. A similar trend was observed in the 40–80 cm horizon, which could be due to copper uptake by plants (Skwierawska et al. 2008b). Kaya et al. (2009) reported that increased application of sulfur and sulfur-containing waste led to a significant increase in the average copper content of plants. In the present study, over the entire experimental period, sulfur fertilization had no significant effect on changes in the copper content of soil at a depth of 0–40 and 40–80 cm.

Manganese. One of the adverse effects of sulfur contamination is an increase in manganese solubility and the mobilization of heavy metals from both natural and anthropogenic sources (James and Riha 1984). Before the experiment, the manganese content of both soil horizons remained stable (Table 2). Manganese concentrations were substantially lower in the 40–80 cm horizon than in the top layer.

In the autumn, after the third year of the study, the manganese content of the 0–40 cm soil layer ranged from 85.75 to 107.41 mg/kg. Form of sulfur had no effect on changes in manganese concentrations in the soil. The application of 40 kg sulfur contributed to an increase in manganese content, compared with higher sulfur doses. Sulfate and elemental sulfur fertilization increased manganese concentrations in the soil, in comparison with the control treatments. The manganese content of the 40–80 cm horizon was considerably lower than in the surface layer, and it was not significantly affected by sulfur form. Manganese depletion was observed, relative to the corresponding treatments before the experiment. In a study by Erdal et al. (2004), the application of elemental sulfur in combination with nitrogen fertilizers substantially increased the bioavailability soil heavy metals, in particular manganese. Lošák et al. (2011) describe that individual nitrogen fertilization did not reduce the content of manganese in the plant or grain of maize. According to Abdou et al. (2011), sulfur fertilization has a minor effect on manganese availability.

Over a three-year period, manganese concentrations in the 0–40 cm soil layer remained stable, but were lower than in soil samples collected before the experiment. The only exception was the treatment fertilized with 40 kg $S\text{-}SO_4^{2-}\text{-}S$, where manganese content was higher than in the other treatments. This trend was maintained throughout the study. In the 40–80 cm layer, manganese concentrations were considerably lower than in the 0–40 cm horizon. The highest manganese content was observed in the treatment with a triple sulfate dose whose effect was noticeable over the entire experimental period. This could result from change in soil pH from acidic to highly acidic (Skwierawska et al. 2008a), which increased the content of readily available manganese.

Lead. Before the experiment, the lead content of soil samples collected at a depth of 0–40 cm and 40–80 cm was similar (Table 2).

In the second year of the study, sulfur fertilization had no significant effect on changes in the lead content of both sampled horizons (0–40 cm and 40–80 cm). The lead content of the 40–80 cm soil layer was considerably lower than in the 0–40 cm horizon, in the corresponding treatments. Similar results were obtained by Šichorová et al. (2004), in the cited study, lead concentrations decreased with increasing soil depth.

At the end of the experiment, the lead content of the 0–40 cm horizon ranged from 4.66 to 6.11 mg/kg soil, and it was significantly affected by sulfur form. A minor increase in lead concentrations was observed. Terelak et al. (1996) found that sulfur-contaminated soils are characterized by increased bioavailability and accumulation of Mn, Cd, and Pb in crops. Similar findings were reported by Holoah et al. (2010) – increasing doses of elemental sulfur caused a significant increase in lead content and uptake by plants. In our study, lead concentrations were lower in the 40–80 cm layer than in the 0–40 cm horizon, and they were significantly affected only by sulfur dose.

Cadmium. Cadmium is one of the most toxic and mobile metallic elements (Basta et al. 2005, Zhao and Masaihiko 2007). Before the experiment, the cadmium content of both sampled soil horizons (0–40 cm and 40–80 cm) was comparable, but it was slightly lower in the deeper layer than in the topsoil (Table 2). Similar trends were observed in the first and second year of the study, when neither sulfur form nor dose exerted a significant effect on changes in cadmium concentrations in the soil. The effect of sulfur was noted as late as in the third year. At the end of the experiment, the cadmium content of the 0–40 cm soil layer increased in treatments fertilized with sulfur, in particular at a dose of 40 kg S. Changes in the

natural cadmium content of soil, observed during a three-year period, were irregular. No distinct trends were noted in response to sulfate and elemental sulfur fertilization. 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) as a result of elemental sulfur application.

Nickel. Before the experiment, the nickel content of the 0–40 cm soil layer varied widely, from 0.68 to 1.45 mg/kg. In the first year of the study, the nickel content of the 0–40 cm horizon increased with increasing sulfate doses (Table 2). Sulfate sulfur exerted a stronger effect than elemental sulfur. The experimental factors had no significant influence on changes in nickel concentrations in the 40–80 cm soil layer.

At the end of the experiment, the nickel content of the 0–40 cm soil layer ranged from 0.367 to 0.592 mg/kg. Nickel depletion was observed, in comparison with the previous two years, probably due to increased nickel uptake by plants. Our results corroborate the findings of Holah et al. (2010). Sulfur fertilization had no significant influence on changes in nickel concentrations in the 40–80 cm horizon. However, a steady decrease in the nickel content of soil was noted, compared with the corresponding treatments in the past two years.

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