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POST-MINE SULFUROUS SOIL CHEMISTRY IN LEACHING EXPERIMENT UNDER CONTROLLED CONDITIONS AT DIFFERENT TREE SPECIES LITTER ADDITION

Streszczenie

Impact of organic matter on soil chemistry of former Jeziórko sulfur mine was investigated. In research two types of substrate were used: less contaminated (mean 5,090 mg kg^{-1} S) and high contaminated (42,500 mg kg^{-1} S). Soil substrates were tested with addition of birch or pine litter. The composites (soil substrate + tree litter) were rinsed with water during 12 weeks of experiment. In the obtained leachate pH, EC, DOC (dissolved organic carbon), N, Ca, Mg and S were determined. The results indicate that the rate and amount of leached elements did not depend on litter, but firstly depend on level of sulfur contamination.

Słowa kluczowe: remediation, sulfur contamination, organic matter, leaching

INTRODUCTION

Sulfur is necessary for proper growth of plants [Marcshner 2012]. However, excessive concentrations of sulfur in soil could have a negative impact on plants by damaging their root system, foliage, deformation of trees and reduced their growth [Tomlinson 1983]. The problem of excessive sulfur concentration occurs especially in open-strip lignite mining sites with high carbon content also on areas with processing plants and in the former sites of mineral mining with the Frasch method. One of the largest sulfur deposits in the world is former Jeziórko sulfur mine located in southern Poland. There sulfur was extracted by Frasch method. To 2010 year, 1,179 hectares (from 2,140 hectares occupied by Jeziórko sulfur mine) were reclaimed [Likus-Cieślik et al. 2015]. Despite reclamation treatments

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(mainly consisting of isolation and neutralization using post-flotation lime), spots with high sulfur content and acid soil were found in this area [Likus-Cieślik et al. 2017]. Sulfur in the soil reached even up to 4% in the 0-20 cm horizon [Likus-Cieślik et al. 2017]. Such areas are unique in environment on the word.

In the paper we presented the results received in experiment conducted under controlled conditions. The aims of experiment was: i) determining the soil solution chemistry especially the amount of sulfur leached from soil substrates characterized by heavy sulfur contamination; ii) to verify the impact of organic matter (birch and pine litter) application on the dynamics of soil solution chemistry.

MATERIAL AND METHODS

Experiment design and laboratory analyses

Substrate samples for the experiment came from the former Jeziórko sulfur mine area (Southern Poland, 50°32'34 N, 21°47'46 E) from sulfur contaminated soils with no vegetation cover (so called hot spots) [Likus-Cieślik et al. 2015]. In the experiment used two kinds of substrates which differed sulfur concentration: i) LS – lower sulfur concentration (average concentration (n=4) 5,090 mg kg^{-1} S, ranging from 4,471 to 5,606 mg kg^{-1}); and ii) HS, with higher sulfur concentration (mean 42,500 mg kg^{-1} S, ranging from 39,477 to 45,959 mg kg^{-1}).

Also two types of litter were used in the experiment: B – birch (Betula pendula Roth), and P – pine (Pinus sylvestris L.). Tree litter were collected under the canopy of tree stands in managed forests with no mining influence.

For the purpose of the experiment composites with substrate (5 cm thickness) and air-dried litter samples (20 g) were created in PCV cylinders (10×15 cm). The composites were created in the following combinations: LS-B and LS-P – substrate with low sulfur concentration and addition of birch or pine litter, respectively; HS-B and HS-P – substrate with high sulfur concentration and addition of birch or pine litter, as well as control substrate containing only substrate LS-c, HS-c (Fig.1). The experiment was conducted over a 12 week-period, during which the cylinders were percolated with 200 ml distilled H2O, twice a week.

In laboratory pH in 1 M KCl (soil:liquid ratio 1:5 for litter and 1:2.5 for soil samples); organic carbon (C), total nitrogen (Nt), and total sulfur (St) contents were measured using LECO TruMac® CNS analyzer and total Ca and Mg contents after digestion in a mixture of HNO3 and 60% HClO4 acid at a ratio of 3:1 (litter) and 1:3 (soil samples) by ICP-OES in the litter and soil samples were measured. C:N ratio was calculated.

The soil solutions were measured once a week for 12 weeks. In soil solution pH and electrical conductivity (EC), dissolved organic carbon (DOC) and total nitrogen content (Nt) were determined in filtered soil solution extracts with a Shimadzu TOC-VCPH Total Organic Carbon Analyzer and the concentrations of Ca, Mg, and St by ICP-OES.

Based on the following equation the total sulfur load (TSL) leached from the composites in the experiment was calculated.

$$TSL_{(12\,weeks)} = \sum_{i=1}^{12} (S_i \cdot WD_i)$$

Si – amount of sulfur leached in a subsequent week of the experiment [mg l^{-1}]; WDi – amount of distilled water used to rinse the composites in a week – constant 0.2 l.

The soil substrates and litter were analyzed at the beginning and at the end (after 12 weeks) of the experiment.

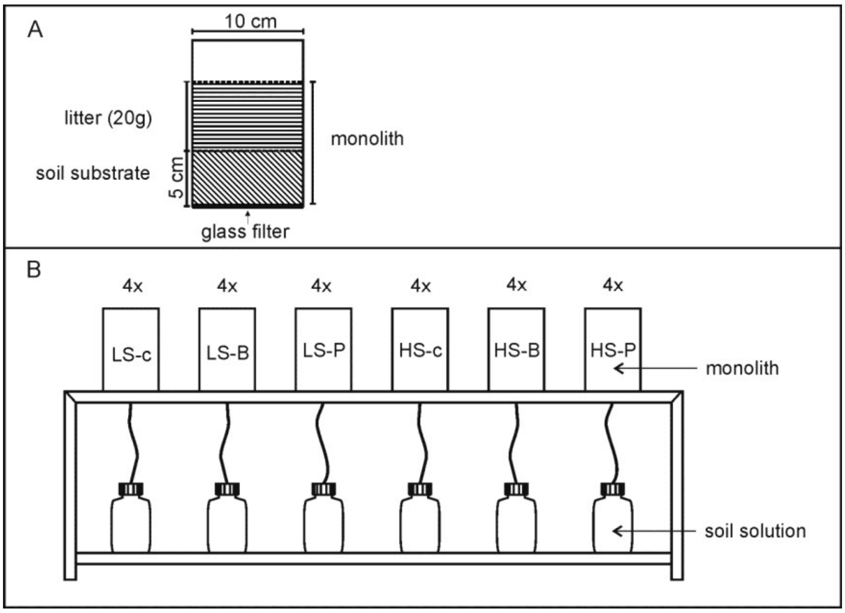

Fig. 1. a – composite scheme; b – general scheme of leaching experiment with different litterfall application (birch or pine litter) to substrate with varied sulfur concentration and control (substrate with no litter application)

Statistical analyses

Statistica software [StatSoft Inc. Software 2011] was used for statistical analysis. The significance of differences in average values of soil characteristics and soil solution between the tested variants of substrate with tree litter were tested by an ANOVA test. The ANOVA test was followed by multiple pairwise comparisons using Fisher's LSD (least significant difference) post-hoc test. Based on the results of Fisher's LSD test, homogeneous subsets were distinguished.

RESULTS

Change of litter parameters

After 12 weeks of experiment, a significant increase of pH was observed in birch litter with low sulfur contamination (LS-B) from 5.3 to 5.8. In pine litter pH increased significantly from 4.2 to 5.6 (LS-P) and to 5.4 (HS-P). Litter pH in the HS-B remained at a similar level as at the beginning of the experiment (Tab. 1).

Tab. 1. Changes in birch and pine litter chemical parameters during a 12-week experiment under controlled conditions.

Properties	pH	St [mg kg^{-1}]	C [g kg^{-1}]	Nt [g kg^{-1}]	Ca [g kg^{-1}]	Mg [g kg^{-1}]	C:N
B†	5.3^{a} ±0.1‡	1415^{a} ±29	475.87^{ac} ±11.72	17.20^{a} ±0.19	8.43^{a} ±.25	0.47^{a} ±0.02	28^{a}
LS-B	5.8^{b} ±0.2	1913^{a} ±380	415.03^{b} ±45.51	18.91^{b} ±2.16	14.49^{b} ±2.22	0.91^{bc} ±0.14	22^{b}
HS-B	5.2^{a} ±0.3	5236^{b} ±1388	414.33^{b} ±23.53	18.73^{ab} ±1.01	14.21^{b} ±1.92	0.84^{b} ±0.10	22^{b}
P	4.2^{c} ±0.0	877^{a} ±40	499.47^{c} ±2.29	6.45^{c} ±0.35	14.64^{b} ±0.24	1.01^{c} ±0.04	77^{c}
LS-P	5.6^{d} ±0.1	1301^{a} ±266	427.55^{b} ±49.63	6.01^{c} ±0.80	9.01^{ac} ±0.40	0.45^{a} ±0.03	68^{d}
HS-P	5.4^{ad} ±0.1	4386^{b} ±1215	438.90^{ab} ±23.12	6.44^{c} ±0.17	10.29^{c} ±0.65	0.50^{a} ±0.03	71^{d}

B – birch litter before the experiment; **LS-B** – birch litter after the experiment on substrate with 5,090 mg·kg^{-1} S; **HS-B** – birch litter after the experiment on substrate with 42,500 mg·kg^{-1} S; **P** – birch litter before the experiment; **LS-P** – pine litter after the experiment on substrate with 5,090 mg·kg^{-1} S; **HS-P** – pine litter after the experiment on substrate with 42,500 mg·kg^{-1} S; §n.d. – no data; ‡a,b – mean values with the same letter are not significantly different at p=0.5, 2.53±0.04 – mean and SD.

The significant increase of St concentration was observed in HS substrates with birch litter i.e. from 1,415 (B) to 5,236 mg kg^{-1} (HS-B) and pine litter i.e. from 877 (P) to 4386 mg kg^{-1} (HS-P) (Tab. 1). In LS substrate significant changes of St was not observed.

Nt content in birch litter increased significantly in LS-B from 17.20 (at the beginning of experiment) to 18.91 g kg^{-1} in the LS-B, and in HS-B to 18.73 g kg^{1}.

In case of pine litter, Nt content was not significant changes in case of LS nor HS substrate (Tab. 1).

In both types of litter C content and C:N ratio decreased significantly (Tab. 1).

An significant increase in Ca and Mg content in birch litter was found in case of LS and HS substrate. However, differences between types of subtract (LS or HS) were not found (Tab. 1).

Changes of mineral soil substrate parameters

Increase of pH was observed in composites with LS substrate, but deacidification of soil occurred only in LS-P (under the pine litter influence). In composites with HS substrate increased of pH was not observed (Tab. 2). The pH of the all substrates during the experiment was still phytotoxic, i.e. below 3.0, in all samples.

In the case of HS substrates rinsing significantly reduced St concentration from over 42,52 to 35,63 (HS-c), 33,25 (HS-B) and 34,16 mg kg^{-1} (HS-P) however, the type of litter was not significant (Tab. 2, Fig. 2).

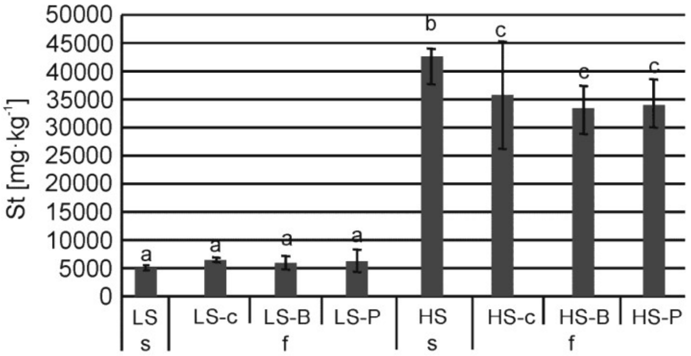

Fig. 2. Sulfur content (St) in the substrate at the beginning and at the end of the experiment under controlled conditions

SOC was at constant levels throughout the experiment (i.e. from 2.89 to 3.22 g kg^{-1} in LS substrates and from 7.20 to 7.80 g kg^{-1} in HS substrate) in HS and LS substrate. (Tab. 2).

The significant increase of Nt after 12 weeks of experiment from 0.16 to 0.22 g kg^{-1} were observed only in HS substrate (Tab. 2).

After 12 weeks of rinsing in LS and HS substrate, Ca content decreased several-fold, but influence of litter addition was not observed (Tab. 2).

Mg content after 12 weeks in all the investigated combinations of LS decreased significantly, but influence of litter addition was not observed (Tab. 2). In case of HS substrate only in the HS-c the Mg content decreased significantly (from 0.11 to 0.08 g kg^{-1}, Tab. 2).

Tab. 2. Chemical parameter changes of soil-substrates during a 12-week experiment under controlled conditions.

	pH	St [mg kg^{-1}]	SOC [g kg^{-1}]	Nt [g kg^{-1}]	Ca [g kg^{-1}]	Mg [g kg^{-1}]
LS†	2.1^{a} ±0.0‡	5090^{a} ±483	3.22^{a} ±0.09	0.03^{a} ±0.03	7.06^{a} ±0.50	0.09^{ac} ±0.01
LS-c	2.6^{bc} ±0.0	6447^{a} ±397	2.89^{a} ±0.32	0.02^{a} ±0.01	0.07^{b} ±0.05	0.04^{b} ±0.01
LS-B	2.5^{bd} ±0.1	5947^{a} ±1319	2.97^{a} ±0.16	0.03^{a} ±0.01	0.06^{b} ±0.00	0.04^{b} ±0.00
LS-P	2.9^{e} ±0.3	6178^{a} ±1870	3.09^{a} ±0.20	0.02^{a} ±0.00	0.38^{b} ±0.36	0.05^{b} ±0.00
HS	2.5^{bc} ±0.0	42521^{b} ±2670	7.78^{b} ±0.13	0.16^{b} ±0.02	11.68^{c} ±1.56	0.11^{a} ±0.00
HS-c	2.6^{c} ±0.0	35634^{c} ±9568	7.23^{b} ±0.34	0.19^{bc} ±0.04	4.21^{de} ±2.25	0.08^{c} ±0.04
HS-B	2.3^{d} ±0.0	33247^{c} ±4033	7.80b ±1.14	0.22^{c} ±0.04	2.95^{d} ±0.73	0.11^{a} ±0.01
HS-P	2.5^{bc} ±0.1	34157^{c} ±4291	7.20^{b} ±0.44	0.18^{b} ±0.01	5.99^{de} ±2.23	0.11^{a} ±0.01

†**LS** – soil substrate with 5,090 mg·kg^{-1} S before the experiment; **LS-c** – control sample of soil substrate with 5,090 mg·kg^{-1} S after the experiment; **LS-B** birch litter after the experiment on substrate with 5,090 mg·kg^{-1} S; **LS-P** – pine litter after the experiment on substrate with 5,090 mg·kg^{-1} S; **HS-b** –soil substrate with 42,500 mg·kg^{-1} S before the experiment; **HS-c** – control sample of soil substrate with 42500 mg·kg^{-1} S after the experiment; **HS-B** –birch litter after the experiment on substrate with 42,500 mg·kg^{-1} S; **HS-P** – pine litter after the experiment on substrate with 42,500 mg·kg^{-1} S; §n.d. – no data; ‡a,b – mean values with the same letter are not significantly different at p=0.5, 2.53±0.04 – mean and SD.

Dynamics of soil dissolution under leaching

On the beginning of experiment (first week), the solutions in both substrate variants and in variants with litter were acidic (pH LS-c 1.8, HS-c 2.2, LS-B 1.8 LS-P 1.8, HS-B 2.2 HS-P 2.3) (Fig. 3a). During the experiment, all variants showed initial increase followed by stabilization of pH value (Fig. 3a). At the end of the experiment, higher pHs were in solutions of composites containing pine litter, especially LS-P (3.8) (Tab. 3).

The highest EC values were observed in first and second week in all researched variants, what indicating the heaviest salt leaching in this time. Next, stabilization of EC was observed (Fig. 3b). Greater dynamics and a faster rate of leaching decrease were observed in LS substrate (Fig. 3b). After 12 weeks of experiment, the leachate EC from HS substrates was significantly higher than from LS substrates (Tab. 3). Birch litter addition influenced at leaching EC only in case of HS substrate – more salt was leaching under influence of birch litter in

HS substrate. In LS substrates the salt leaching impact (EC change) was significant without litter addition.

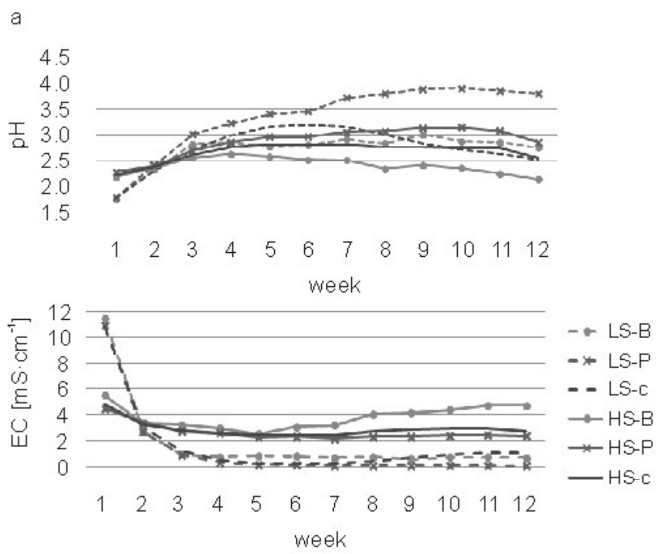

Fig. 3. Changes in pH (a) and EC (b) of the solutions during the experiment under controlled conditions

In the first week significantly higher St were observed in the solutions from LS than HS substrate (Fig. 4, Tab. 3). In LS substrates dynamic decline of St were observed to third week. After this time stabilization of St leaching was observed (Fig. 4). In HS substrate St changes were permanent (Fig. 4). At the end of experiment St concentrations in the solutions from the LS substrate were significantly lower than at the beginning (Tab. 3). It was found that the leaching effect did not increase significantly after application of the litter (Tab. 3).

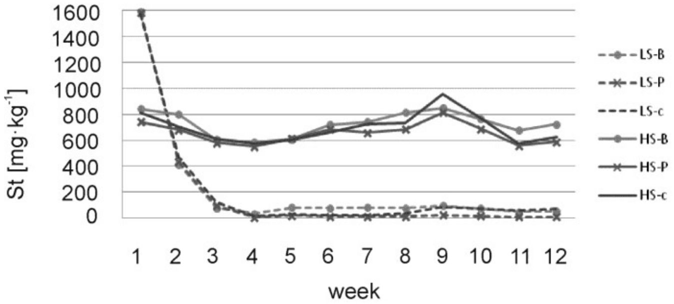

Fig. 4. Changes in sulfur content (St) in solutions during the experiment under controlled conditions

Total sulfur load (TSL) leached from the LS substrates was significantly lower than in HS substrate. It was not found impact of litter application on the amount of sulfur leached after 12 weeks of leaching (Fig. 5).

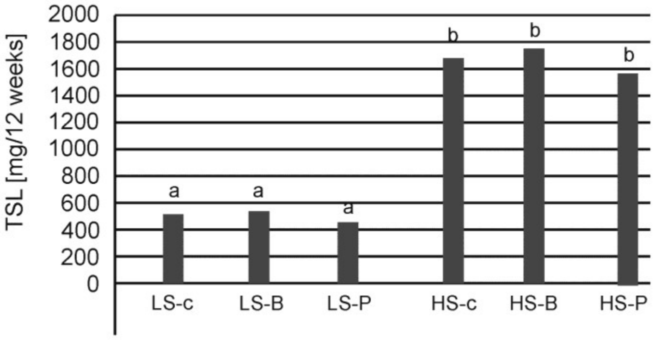

Fig. 5. Load of sulfur leached (TSL) from substrates in various combinations of composites used in the experiment

In case of DOC, it was found that significantly more leaching of DOC were observed in composites containing litter, but the type of added litter did not affect to amount of leached DOC (Tab. 3).

In the first 2-3 weeks of the experiment, greater leaching of Nt was observed in the leachate from LS substrate. After this period Nt content stabilized (Fig. 6b). After the experiment, significantly higher Nt leaching was observed in the leachate from HS substrate with birch litter (Tab. 3).

At the beginning of the experiment Ca in HS were higher than in LS solutions. Ca leaching from HS substrate composites was constantly high but at the end it was significantly lower than at the beginning of the experiment (Fig. 6c). Ca leaching from LS substrates was intensive in the first three weeks of the experiment, and next it stabilized (Fig. 6c). Ca concentrations in leachate from LS composites at the end of the experiment were significantly lower than at the beginning of the experiment (Tab. 3).

At the beginning of the experiment, Mg in solutions from HS substrates was significantly lower than from LS substrate (Tab. 3). At the end of the experiment, Mg content in solutions from HS substrate was significantly higher than at the beginning in the case of birch litter addition (Tab. 3). Mg concentration in the solutions from LS substrate was significantly lower at the end of the experiment than at the beginning in each case (Tab. 3). Mg leached the most from composites with birch litter.

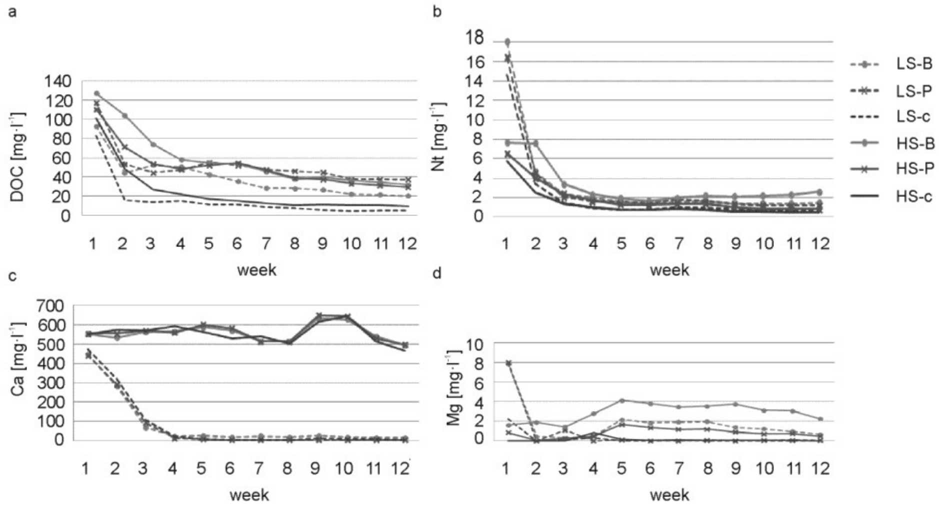

Fig. 6. Changes in the DOC (a), Nt (b), Ca (c), Mg (d) content in solutions during the experiment under controlled conditions

Tab. 3. Changes in pH, EC and St concentration in soil solutions after 12 weeks of leaching through soil substrates and composites in controlled conditions.

		LS-c	LS-B	LS-P	HS-c	HS-B	HS-P
pH	s[†]	$1.8^{b§}$ ±0.04	1.8^{b} ±0.02	1.8^{b} ±0.10	2.2^{b} ±0.03	2.2 ±0.10	2.3^{b} ±0.10
	f[‡]	2.5^{a} ±0.08	2.8^{a} ±0.12	3.8^{a} ±0.26	2.6^{a} ±0.18	2.2 ±0.22	2.9^{a} ±0.26
EC [mS·cm^{-1}]	s	10.80^{a} ±1.38	11.48^{a} ±0.42	10.91^{a} ±3.07	4.85^{a} ±0.29	5.52 ±0.77	4.51^{a} ±0.49
	f	1.12^{b} ±0.32	0.71^{b} ±0.23	0.10^{b} ±0.03	2.74^{b} ±0.34	4.75 ±1.00	2.40^{b} ±0.36
St [mg·kg^{-1}]	s	1570.8^{a} ±156.5	1585.9^{a} ±100.1	1583.3^{a} ±428.6	814.3^{a} ±31.4	841.4 ±73.6	741.0^{a} ±74.4
	f	71.0^{b} ±20.3	53.7^{b} ±17.2	9.6^{b} ±4.6	625.7^{b} ±36.6	725.4 ±63.6	588.0^{b} ±24.0
DOC [mg·l^{-1}]	s	82.84^{a} ±8.95	92.97^{a} ±9.38	117.13^{a} ±18.80	101.41^{a} ±9.54	127.00^{a} ±33.11	110.37^{a} ±15.38
	f	5.08^{b} ±0.33	20.47^{b} ±3.17	37.20^{b} ±10.72	9.78^{b} ±1.65	32.00^{b} ±4.03	29.56^{b} ±2.48
Nt [mg·l^{-1}]	s	14.65^{a} ±1.97	18.05^{a} ±1.02	16.42^{a} ±4.11	5.72^{a} ±0.31	7.68^{a} ±1.55	6.54^{a} ±0.69
	f	0.53^{b} ±0.04	1.46^{b} ±0.17	1.17^{b} ±0.33	0.43^{b} ±0.02	2.58^{b} ±0.67	0.82^{b} ±0.05

Ca [mg·l^{-1}]	s	474.41^{a} ±39.07	445.30^{a} ±26.10	442.78^{a} ±33.14	550.23^{a} ±17.79	552.79^{a} ±14.18	553.38 ±50.04
	f	5.84^{b} ±1.28	13.66^{b} ±4.08	5.20^{b} ±6.11	467.27^{b} ±24.03	494.78^{b} ±21.99	495.56 ±17.26
Mg [mg·l^{-1}]	s	2.21^{a} ±1.73	7.85^{a} ±1.48	8.01^{a} ±3.14	0.01^{c} ±0.00	1.62^{ad} ±0.96	0.85^{d} ±1.70
	f	0.01^{b} ±0.00	0.68^{b} ±0.12	0.07^{b} ±0.14	0.01^{c} ±0.03	2.24^{a} ±0.49	0.48^{d} ±0.09

†s –at the beginning of experiment; ‡f – after 12 weeks; §different letters indicate significant differences in the measured values at the beginning and at the end of the experiment (p=0.05, pairwise t-test)

DISCUSSION AND CONCLUSIONS

The rate and amount of leached elements did not depend on litter, as it was hypotyzed. The results indicate that chemistry of leachate depend on the amount of sulfur concentration in the substrate (LS or HS substrate). For example: a significant reduction of sulfur content was observed in HS composites after rinsing, but no effect of litter addition was found. Organic matter, especially pine litter had impact on deacidification (increase pH value). Although pH of the substrates increased during 12 weeks of the experiment, it was still phytotoxic. Low soil pH may be related to litter decomposition when organic acids are released and decrease of soil pH [Augusto et al. 2002]. Acidification and low pH in the soil are usually associated with high sulfur concentrations [Benison, Bowen 2013]. In this case the main cause of acidification of substrates and soil solutions could be high sulfur concentration. It was also observed that despite significant reduction of St content in the HS composites, the degree of sulfur contamination was still very high. According to the values reported by Polish Institute of Crop and Soil Fertilization (IUNG), sulfur concentration was exceeded by 35-fold more than very high sulfur content (heavy contamination), i.e. above 1,000 mg kg^{-1} [Kabata-Pendias et al. 1995]. Kabata-Pendias [2011] claimed that elemental sulfur application (at a rate of 300 μmol kg^{-1}) can decreases soil pH by 0.5 unit. In our experiment it was observed that birch litter increased Nt and Mg leaching from the substrate with HS and Mg in LS. At the beginning of the experiment birch litter contained more Nt and Mg than pine litter. In similar experiment conducted by Woś and Pietrzykowski [2015], showed a significant impact of birch litter on the intensification of soil-forming processes and increased leaching of DOC, Nt and Mg. Some species affected soil pH, DOC and Mg [Menyailo et al. 2002]. Menyailo et al. [2002] did not observe the impact of species on Ca and Nt content in soils. Chodak and Niklińska [2010] found that the tree species had a significant impact on the chemical and microbial properties of mine soils, mainly through pH changes with SOC and Nt content.

During decomposition of organic matter, its mass decreases while the concentration of these elements in organic matter increases. This is the reason for that St Nt, Ca and Mg increased in litter. Elevated St content in the litter on LS and HS substrates may also be partly due to litter contamination with these materials during the experiment, e.g. by suction and soaking from the contaminated substrate (residual layer of the composite mineral). This may be one of the factors explaining an increase in element leaching from soil substrates through greater solubility [You et al. 1999, Kalbitz et al. 2000].

The results show that tree litter in areas where soils is most contaminated with sulfur, is not sufficient for detoxification. In mine areas where the neutralization was not effective or inaccurate, it would be necessary to make neutralization again, using higher doses of flotation lime [Gołda 2003, Likus-Cieślik et al. 2017]. In the next step, it would be possible to introduce afforestation, which will introduce litterfall and organic matter. Litterfall and organic matter are very important in soil-forming processes [Woś, Pietrzykowski 2015]. Soil organic matter, plays an important role in the nutrient balance in newly developed ecosystems on reclaimed and afforested post-industrial, especially post-mine sites [Pietrzykowski, Krzaklewski 2007, Urbanová et al. 2014]. It has been proven that under influence of decaying organic matter and biochemical processes produce more intensive leaching and displacement of macronutrients into the soil profile and soil development will occur. However, without neutralization it will be a very long-term process.

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CHEMIZM POKOPALNIANEJ GLEBY SULFUROZOWEJ W DOŚWIADCZENIU W WARUNKACH KONTROLOWANYCH

Streszczenie

Zbadano wpływ materii organicznej na chemizm gleby byłej kopalni siarki Jeziórko. W badaniach wykorzystano dwa rodzaje substratu: mniej zanieczyszczony (średnio 5090 mg kg-1 S) i wysoce zanieczyszczo-ny (42 500 mg kg -1 S). Podłoża glebowe badano z dodatkiem ściółki brzozowej lub sosnowej. Kompozyty (podłoże gleby + ściółka drzewna) płukano wodą w ciągu 12 tygodni eksperymentu. W otrzymanym odcieku oznaczono EC, DOC (rozpuszczony węgiel organiczny), N, Ca, Mg i S. Wyniki wskazują, że szybkość i ilość wyługowanych pierwiastków nie za-leżą od ściółki, ale zależą od poziomu zanieczyszczenia siarką.

Słowa kluczowe: remediacja, zanieczyszczenie siarką, materia organiczna, ługowanie