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The influence of Chemical Works “Police” on chemical composition of *Pinus sylvestris* needles, *Pleurozium schreberi* and soil samples

Wpływ oddziaływania Zakładów Chemicznych „Police” na skład chemiczny igieł *Pinus sylvestris*, *Pleurozium schreberi* i gleby

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Słowa kluczowe: metale ciężkie, bioakumulacja, *Pinus sylvestris*, siarka, skażenie gleby, bioindykatory

Abstract

The aim of this study was an evaluation of the influence of Chemical Works “Police” (“Grupa Azoty”) on total S, N, C, Mg, K, Ca, Na, Hg, Pb, Cd, Cu, Co, Cr, Mn, Ni and Zn concentrations in 11-year-old needles of *Pinus sylvestris* L., *Pleurozium schreberi* (Brid.) Mitt. and soil samples. The research material was collected in spring 2013 in the coniferous habitats. The total Hg content was determined by Mercury Analyzer AMA 254. The total content of S, N and C was analysed by COSTECH CHNS elementary analyser. The total content of other elements was determined using atomic absorption spectrometer ASA iCE 3000. The highest mercury content was similar in pine needles ($0.06 \text{ mg} \cdot \text{kg}^{-1} \text{ d. w.}$) and in moss ($0.07 \text{ mg} \cdot \text{kg}^{-1} \text{ d. w.}$). The soil samples pH value was typical for rusty soils, in which the obtained pH–KCl value was 3.13–4.39 and pH–H₂O was 3.69–5.14 (extreme acid soils). The Zn, Pb, Ni, Hg, Fe, Cu and Cr concentrations were higher in moss than in needles. The content of Pb in *Pleurozium schreberi* ($3.13 \text{ mg} \cdot \text{kg}^{-1} \text{ d. w.}$) was few times higher than in *Pinus sylvestris* needles ($0.073\text{--}0.817 \text{ mg} \cdot \text{kg}^{-1} \text{ d. w.}$). The soil contamination with heavy metals was not observed. There has been almost a double reduction of sulphur content in pine needles and moss over last 20 years.

Streszczenie

Celem pracy była ocena wpływu Zakładów Chemicznych „Police” S. A. (Grupa Azoty) na zawartość S, N, C, Mg, K, Ca, Na, Hg, Pb, Cd, Cu, Co, Cr, Mn, Ni i Zn ogółem w drugorocznych szpilkach sosny zwyczajnej (*Pinus sylvestris*), w rokitniku pospolitym (*Pleurozium schreberi*) oraz w glebie. Próbkę pozyskano wiosną 2013 roku na siedliskach borowych. Całkowitą zawartość Hg oznaczono za pomocą analizatora rtęci AMA 254. Zawartość S, N i C ogółem oznaczono analizatorem elementarnym CHNS firmy COSTECH. Zawartość pozostałych pierwiastków oznaczono spektrometrem absorpcji atomowej ASA iCE 3000. Najwyższa zawartość rtęci w szpilkach sosny ($0,06 \text{ mg} \cdot \text{kg}^{-1}$ suchej masy) i w mchu ($0,07 \text{ mg} \cdot \text{kg}^{-1}$ suchej masy) była podobna. Wartości pH gleby były charakterystyczne dla gleb rdzawych i wynosiły: pH–KCl 3,13–4,39; pH–H₂O 3,69–5,14 (gleby silnie kwaśne). Zawartość Zn, Pb, Ni, Hg, Fe, Cu i Cr była wyższa w mchu niż w szpilkach sosny. Zawartość Pb w mchu ($3,13 \text{ mg} \cdot \text{kg}^{-1} \text{ s. m.}$) była kilka razy wyższa niż w szpilkach *Pinus sylvestris* ($0,073\text{--}0,817 \text{ mg} \cdot \text{kg}^{-1} \text{ s.m.}$). Nie stwierdzono zanieczyszczenia gleb metalami ciężkimi. Stwierdzono prawie dwukrotną redukcję w akumulacji siarki w szpilkach sosny oraz w mchu na przestrzeni ostatnich 20 lat.

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1. INTRODUCTION

The Chemical Works “Police” (currently “Grupa Azoty”) are involved in the production of mineral fertilisers including multi-mineral fertilisers, phosphoric fertilisers, nitric fertilisers and nitric–sulphuric fertilisers. Despite implementation of the Environmental Management System according to ISO 14001:2004 and new technologies of gases and particulate matter reduction in the last 10 years, the factory remains one of the main West Pomeranian environment pollutants [www.grupaazoty.com; Raport...2010-2011]. The emission of sulphur oxides, nitrogen oxides, fluorine, volatile organic compounds and particulate matter (i. e. heavy metals) may have a negative impact on flora living in the pollutant area [Bell and Treshow, 2004]. Heavy metals are particularly dangerous for the environment because of their ability to accumulate in several ways. They cannot be decomposed by microorganisms, but are transported to other parts

of the environment. Unfortunately, heavy metals accumulate in plants or animals organisms, which are the elements of the food chain, and they can slowly damage the internal organs [Manahan, 2013].

Pinus sylvestris belongs to the group of plants vulnerable to the SO₂, O₃ and F pollution, what can be seen in affected needles necrosis and progressive defoliation of trees. Mosses are good bioindicators of air pollution with heavy metals, primarily due to the absorption of atmospheric pollutants by the entire surface of the thallus and bioaccumulation potential over many years [Zimny, 2006].

The main purpose of this study is to evaluate the impact of Chemical Works “Police” on the chemical composition of *Pinus sylvestris* L. 11-year-old needles, *Pleurozium schreberi* (Brid.) Mitt. moss and soil samples collected from coniferous habitats within the factory affected area.

2. MATERIAL AND METHODS

The samples of *Pinus sylvestris* 11-year-old needles, soil and *Pleurozium schreberi* were collected in spring 2013 from Chemical Works “Police” influence area, which is one of the main Polish producer of fertilisers. Namely, seven pooled needles samples, consisting of at least 300 needles and obtained from five trees in each point, were collected in forest sites of Wienkowo, Witorza, Trzeszczyn, Jasienica and Tanowo villages and two samples were collected in Police town (Fig. 1). The needles were collected from bottom branches of the trees, located on the outer wall of the forest. The soil samples were also collected in every point of research using Egner’s sampling stick (approximately 100 g from 0 to 20 cm depth – O, A or O, AE horizons). The soil substrate was characterised as mineral. The *Pleurozium schreberi* samples were collected from the adjacent area of every research point.

Pinus sylvestris needles, moss and soil samples were dried in temperature up to 40°C and pulverised in laboratory mill or mortar. The sub-samples of needles and moss (1.0 g) were cold-treated with oxi-acid mixture of 65% HNO₃ and 30% H₂O₂ (4 and 1 ml, respectively) and after at least 12 hours digestion put in Milestone™ microwave oven. Whereas, soil samples (0.5 g) were cold-treated with 6 ml mixture of 5:1 concentrated acids HNO₃:HClO₄ (65:60%, respectively) and also mineralised with Milestone™ microwave oven. Mineralised samples were finally diluted to 25 ml with deionised water. The content of Cd, Cu, Cr, Mn, Ni, Co, Pb, Mg, Na, Ca, Fe, Zn and K in dry weight (d. w.) of needles, moss and soil samples was determined using atomic absorption spectrometer ASA ICE 3000 series. The determination process was validated with certified reference materials: tea leaves (INCT–TL–1) produced by Institute of Nuclear Chemistry and Technology Warsaw, Poland and loamy sand (CRM 036-050).

The content of Hg was determined directly in dried samples using AMA 254 Mercury Analyzer. The content of P was determined with spectrometric molybdenum blue method (690 nm wave length).

The pH in 1M KCl and H₂O, and electrical conductivity of soil samples were determined using automatic conductivity meter.

The limits of detection (mg•kg⁻¹) for determined elements were as follows: Cd 0.013; Cu 0.005; Cr 0.005; Mn 0.002; Ni 0.008; Co 0.010; Pb 0.073; Mg 0.002; Na 0.004; Ca 0.009; Fe 0.052; Zn 0.003 and K 0.001.

Statistical analysis of the obtained data was performed using Statistica software (Statsoft Inc., version 10.0. MR1). At first, data were investigated to determine their distribution using the Shapiro–Wilks’ W-test. The basic statistical analysis included arithmetic mean, standard deviation and range values (minimum and maximum values). Bioaccumulation of metals concentrations between investigated species were compared by the Mann–Whitney U test ($p \leq 0.05$). The Spearman correlation coefficients ($p \leq 0.05$) between metals accumulation in *Pinus sylvestris* needles were also analysed.

3. RESULTS AND DISCUSSION

The data of soil pH in 1 M KCl, pH in H₂O and electrical conductivity are given in Table 1. The arithmetical mean, standard deviation and range (minimum and maximum values) of determined heavy metals (Pb, Hg and Cd) and trace elements (Co, Cu, Cr, Fe, Mn, Ni and Zn) in d. w. with bioaccumulation factors are presented in Table 2. Macroelement results (Mg, Na, Ca, K, P, N, C, S) are presented in Table 3. The analysis of Spearman correlation coefficients between elements in *Pinus sylvestris* needles are presented in Table 4.

The samples of *Pinus sylvestris* needles, moss and soil were collected from coniferous forests habitat. Investigated soils could be classified as Brunic Arenosol or Albic Brunic Arenosol [IUSS...2006]. The soil pH (in 1 M KCl and H₂O) was typical for the coniferous habitats and the range was 3.13–4.39 in KCl (acidic habitats) and 3.69–5.14 in H₂O (extreme acidic and acidic habitats). The electrical conductivity of tested soils ranged from 22.1 to 246.8 $\mu\text{S}\cdot\text{cm}^{-1}$, wherein the highest values were observed in soil samples from Police town. Borowiec [1990] performed soil and

Fig. 1. Sampling area

Table 1. The pH and electrical conductivity values of soil samples

Localisation and distance from the main source of emissions [km]	Number	pHKCl	pHH ₂ O	EC
				μS • cm ⁻¹
Wierkowo – 2.6	1	4.39	5.14	26.21
Witorza – 5.0	2	3.82	4.66	22.13
Trzeszczyn – 3.4	3	3.25	3.79	76.93
Jasienica – 2.7	4	3.99	4.54	31.03
Tanowo – 6.6	5	3.83	4.68	50.52
Police – 3.3	6	3.26	3.83	246.8
Police – 3.8	7	3.13	3.69	115.74

pine needles experiment in 1977 and 1988 in the Chemical Works "Police" area in two points – approximately 15 and 3 km from the emitter. The average soil pH_{H₂O} in 1977 and 1988 was 3.4, while currently the range is about 3.69–5.14, which shows a decrease in soil acidification.

Comparison of metals bioaccumulation in needles and moss revealed that Zn, Pb, Ni, Hg, Fe, Cu and Cr concentrations were higher in moss than in needles. However, the opposite situation was observed with Co and Mn contents – they were higher in needles. The concentrations of Pb, Fe, Cr and Co were higher in soil samples than in plants, which can be explained by the way of elements absorption on these organisms (mosses by the entire surface of thallus from water and air, directly or by the stomatal apparatus in *Pinus sylvestris* needles). The Cd concentration in all of determined samples was under detection limit (<0.013 mg • kg⁻¹ d. w.).

Iron content was markedly higher in needles and in soil, which is borne out by bioaccumulation factors value (BCFN/S=10.51). It is noteworthy that Co and Mn were accumulated 11.03 and 9.75 times, respectively, more than their concentrations in soil.

Soil quality standards excess of any metal concentration, according to the Polish laws [Rozporządzenie...2002], (Cr 50.0; Zn 100.0; Cd 1.0; Co 20.0; Cu 30.0; Ni 35.0; Pb 50.0; Hg 0.5 mg • kg⁻¹ d. w.), was not observed in this study. Referring to the Geochemical Atlas of Poland [www.mapgeochem.pgi.gov.pl], the average content of sulphur in examined material (0.004%) was lower than average content of S in total soils (0.017%), industrial areas (0.038%) and forest soils (0.012%) of Poland.

There were also no statistically significant (Mann–Whitney U Test; p≤0.05) differences between mosses and needles bioaccumulation. Nevertheless, few statistically positive significant Spearman correlation coefficients (p≤0.05) between metals bioaccumulation in needles were observed, e.g.: Co–Mn (r=0.666), Fe–Cr (r=0.800) and Cu–Ni (r=0.700).

Similar studies of metals accumulation in *Pinus sylvestris* needles and *Pleurozium schreberi* were performed by many authors [Świercz, 2003; Parzych and Jonczak, 2013; Borowiec and Zabłocki, 1984; Borowiec et al., 2005; Gałuszka, 2006; Dołęgowska et al., 2013].

Table 2. The content of heavy metals and trace elements in *Pinus sylvestris* needles, *Pleurozium schreberi* and soil samples

Object	n=7	Co	Cr	Cu	Fe	Mn	Ni	Pb	Zn	Hg
		mg • kg ⁻¹ d. w.								
<i>Pinus sylvestris</i> needles	AM	0.016	3.19	4.16	120.7	504.6	2.51	0.073	46.7	0.041
	SD	0.279	4.36	0.81	58.1	299.8	2.64	0.665	10.3	0.011
	Min	0.010	0.94	3.48	58.8	105.9	0.84	0.013	37.5	0.025
	Max	0.656	14.64	6.03	236.1	900.2	9.40	0.817	68.1	0.062
<i>Pleurozium schreberi</i>	AM	0.010	12.33	7.59	528.2	345.2	7.62	3.13	50.5	0.052
	SD	0.283	16.43	2.31	207.2	228.6	9.37	1.32	3.5	0.013
	Min	0.010	1.62	4.69	246.1	102.8	1.86	1.97	46.5	0.039
	Max	0.366	41.43	10.33	800.0	671.2	24.26	4.94	54.3	0.071
Soil	AM	0.071	3.98	3.05	3260.6	63.1	1.78	9.63	16.5	0.020
	SD	0.389	1.21	1.03	849.8	54.0	0.80	12.84	10.7	0.015
	Min	0.010	2.52	1.82	1677.7	26.3	0.44	2.32	6.2	0.007
	Max	0.821	5.74	5.24	4334.9	201.9	2.69	43.34	38.4	0.053
BCFN/S	AM	2.37	0.98	0.04	10.51	1.73	0.02	3.65	3.26	2.37
	SD	5.02	1.60	0.02	7.89	1.65	0.04	1.57	2.59	5.02
BCFM/S	AM	11.03	4.39	3.27	0.21	9.75	5.49	0.63	5.00	4.96
	SD	15.85	6.76	1.90	0.16	9.53	7.86	0.47	2.46	3.35

Cd<0.013; d. w.–dry weight; AM–arithmetical mean; SD–standard deviation; Min–minimum value; Max–maximum value; BCFN/S–bioaccumulation factor between *Pinus sylvestris* needles and soil; BCFM/S–bioaccumulation factor between *Pleurozium schreberi* and soil

Table 3. The content of macroelements in *Pinus sylvestris* needles, *Pleurozium schreberi* and soil samples

Object	n=7	Mg	Na	Ca	K	P	N	C	S
		g•kg ⁻¹ d. w.							
<i>Pinus sylvestris</i> needles	AM	0.876	0.266	5.06	5.56	2.87	16.0	446.6	0.532
	SD	0.198	0.155	1.78	1.46	0.722	2.8	148.8	0.266
	Min	0.592	0.059	2.96	3.69	1.32	11.7	50.4	0.305
	Max	1.152	0.502	8.11	8.10	3.93	21.2	511.3	1.184
<i>Pleurozium schreberi</i>	AM	1.063	0.143	1.68	7.32	4.54	15.1	428.3	0.736
	SD	0.306	0.043	0.177	1.77	1.15	6.0	3.8	0.292
	Min	0.728	0.096	1.40	5.46	3.07	10.0	424.2	0.533
	Max	1.378	0.208	1.83	9.09	6.19	24.8	434.1	1.249
Soil	AM	0.222	0.578	0.168	0.601	0.005	0.636	11.40	0.044
	SD	0.059	0.015	0.064	0.164	0.005	0.460	7.07	0.075
	Min	0.119	0.047	0.109	0.369	0.001	0.278	5.93	0.005
	Max	0.294	0.094	0.291	0.896	0.017	1.8	29.58	0.241
BCFN/S	AM	4.14	4.64	33.21	9.52	3034.4	31.23	48.37	49.26
	SD	1.21	2.62	16.06	2.26	872.5	12.46	24.60	72.46
BCFM/S	AM	5.56	2.87	10.72	14.03	5115.0	39.55	51.47	87.04
	SD	1.44	1.02	4.27	2.28	1858.7	28.19	13.42	92.30

d. w.–dry weight; AM–arithmetical mean; SD–standard deviation; Min–minimum value; Max–maximum value; BCFN/S–bioaccumulation factor between *Pinus sylvestris* needles and soil; BCFM/S–bioaccumulation factor between *Pleurozium schreberi* and soil

Świercz [2003] presented a study of 11-year-old needles collected from cement factory influence area in Świętokrzyskie voivodeship, Poland. Comparing the results, the differences in Fe, Mn, Pb, Na, K and Ca bioaccumulation were observed. The concentrations of Fe (120.7 mg•kg⁻¹ d. w.) and Mn (504.6 mg•kg⁻¹ d. w.) in this research were higher than those by Świercz [2003] (94.0 and 46.0 mg•kg⁻¹ d. w., respectively). However, the Pb and Hg contents were lower. The results of other elements were comparable. The received findings of 11-year-old *Pinus sylvestris* needles correspond to the research by Parzych and Jonczak [2013] at Słowiński National Park in Poland, but there is a significant difference in Mn accumulation. Namely, the range of Mn in conducted study is 105.9–900.2 mg•kg⁻¹ d. w., while in Słowiński NP the range was not as wide (211.1–449 mg•kg⁻¹ d. w.) [Parzych and Jonczak, 2013]. Borowiec and Zabłocki [1984] and Borowiec et al. [2005] conducted the *Pinus sylvestris* needles sulphur accumulation tests at the same area of Chemical Works “Police” in 1984–2000. The average content of S in *Pinus sylvestris* needles in 1984 had been 1.9–2.1 g•kg⁻¹ d. w. and it progressively decreased to 1.2–1.5 g•kg⁻¹ d. w. in 1980–1989 and 0.9–1.1 g•kg⁻¹ d. w. in 2000 [Borowiec and Zabłocki, 1984; Borowiec et al., 2005]. The average concentration of S in *Pleurozium schreberi* collected from the fertilisers factory influence area in 1984 was in the range of 2.6–3.6 g•kg⁻¹ d. w. [Borowiec and Zabłocki, 1984]. Based on the results of other authors mentioned above, it can be concluded that there has been a significant improvement in the level of sulphur accumulation in *Pinus sylvestris* needles from the Chemical Works “Police” influence area.

The investigation of sulphur content in pine needles within 0.5–7.7 km from Lucchini smelter in Warsaw [Dmuchowski and Czwa-kiel, 2005] also showed that the negative influence of Chemical

Works “Police” is less. Furthermore, according to Huttunen et al. [1985], the harmful level of sulphur for plants (1.2 g•kg⁻¹) has not been exceeded.

Gałuszka [2006] presented the findings of Fe (304–1289 mg•kg⁻¹ d. w.), Hg (0.05–0.123 mg•kg⁻¹ d. w.), Mn (124.0–837.0 mg•kg⁻¹ d. w.), Pb (<5.0–11 mg•kg⁻¹ d. w.) and Zn (36–52 mg•kg⁻¹ d. w.) accumulation in *Pleurozium schreberi* from Wigierski National Park in Poland, comparable with the results obtained in this study. In turn, Dołęgowska et al. [2013] performed research from the Kielce area, which included Cd, Co, Hg, Mn, Ni, Pb and Zn concentrations in *Pleurozium schreberi*. A substantial part of the results of Cu (6.75–15.46 mg•kg⁻¹ d. w.), Fe (406.0–753.0 mg•kg⁻¹ d. w.), Mn (35.0–697.0 mg•kg⁻¹ d. w.), Hg (0.028–0.078 mg•kg⁻¹ d. w.) and Zn (45.0–81.0 mg•kg⁻¹) was comparable with findings of our study. However, the contents of Cd (0.38–1.06 mg•kg⁻¹ d. w.), Co (0.20–0.65 mg•kg⁻¹ d. w.) and Pb (8.3–21.5 mg•kg⁻¹ d. w.) in *Pleurozium schreberi* from Kielce area were higher than those observed from Police area. Merely one element concentration (Ni–7.62 mg•kg⁻¹ d. w.) was clearly higher in moss samples from this study than in samples from Dołęgowska et al. [2013] (1.0–2.2 mg•kg⁻¹ d. w.).

4. CONCLUSIONS

A significant improvement of sulphur accumulation in *Pinus sylvestris* needles and *Pleurozium schreberi* over many years was observed. The situation can be related to reduction of pollutants emission and implementation of new technologies by Chemical Works “Police”. *Pleurozium schreberi* accumulated metals in a higher level than 11-year-old needles. The soil contamination with heavy metals was not observed within the studied area.

Table 4. Metal-to-metal Spearman correlation coefficients in *Pinus sylvestris* needles

	Co	Cr	Cu	Fe	Mn	Ni	Pb
Cr	-0.250						
Cu	0.367	0.517					
Fe	-0.100	0.800*	0.550				
Mn	0.667*	-0.650	-0.167	-0.567			
Ni	0.133	0.617	0.700*	0.617	-0.117		
Pb	0.383	-0.483	-0.567	-0.283	0.567	-0.200	
Zn	-0.250	-0.150	-0.250	0.033	-0.383	-0.417	0.067
Hg	0.317	0.617	0.317	0.400	-0.283	0.233	0.017

*Significant with $p \leq 0.05$

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