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Speciation of Heavy Metals in Bottom Sediments of Lakes

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Abstract

The work reported in this paper deals with the content of trace metals (Cd, Cr, Cu, Fe, Mn, Ni, Pb, and Zn) in bottom sediments of Goreckie Lake, situated in Wielkopolski National Park, western Poland. Apart from total concentration, the distribution of the above metals into five fractions: exchangeable, bound to carbonates, bound to Fe-Mn oxides, bound to organic matter, and residual, was studied by means of an analytical procedure involving sequential chemical extraction.

Results were discussed on the basis of concentrations found and the resulting trace metal speciation.

Keywords: heavy metals, bottom sediments, lakes, sequential extraction, speciation.

Introduction

The term speciation of heavy metals in bottom sediments of natural and man-made water reservoirs refers to quantitative and qualitative differentiation of the forms of their occurrence. The criteria of division into various forms can be different, e.g.(l) functional - forms available for organisms living in water, mobile forms, exchangeable cations etc., (2) operational - according to procedures, reagents and extractant used for their isolation, identification and determination, and (3) according to the element oxidation state or specific compounds [1-3].

Speciation of metals in bottom sediments has been the subject of interest for a long time; however, the first work in which a complete scheme of operational differentiation of metal forms by sequential extraction was proposed was a paper by Tessier et al. [4]. They have identified and defined five fractions.

Fraction I. Exchangeable metals. This fraction comprises metals adsorbed on the surface of the sediment. It is the most accessible, and metal migration from the solid phase to water can occur, e.g., after a change of ionic composition of water or as a result of a shift of adsorption equilibrium in the system.

Fraction II. Metals bound to carbonates. They can be released as a result of pH decrease. *Fraction HI. Metals bound to iron and manganese oxides.* This fraction is sensitive to the redox potential changes and in anaerobic conditions it is thermodynamically unstable.

Fraction IV. Metals bound to organic matter. These metals are temporarily inaccessible. With time passing the processes of aerobic or anaerobic decomposition of organic matrix induce their release to water or other fractions.

Fraction V. Metals in other forms. This fraction includes mainly metals built in the crystal lattice of minerals. In natural conditions they are practically inaccessible for living organisms and can be treated as permanently immobile.

To separate particular fractions Tessier et al. [4] applied various extraction systems. Later other authors introduced certain modifications both in the division into fractions and in the method of sequential extraction [1-3, 5-7].

The aim of this work was speciation of heavy metals on the level of the geochemical background, in bottom sediments of a lake localized in the area under special protection so as not to be subjected to anthropopression. The idea of division into fractions was the same as that proposed by Tessier et al. [4] but the scheme of sequential extraction at the first two stages of the procedure was a bit modified.

Materials and Methods

The studies were carried out for Goreckie Lake localized in the central part of Wielkopolski National park (Western Poland). The geographical landscape of this Park was

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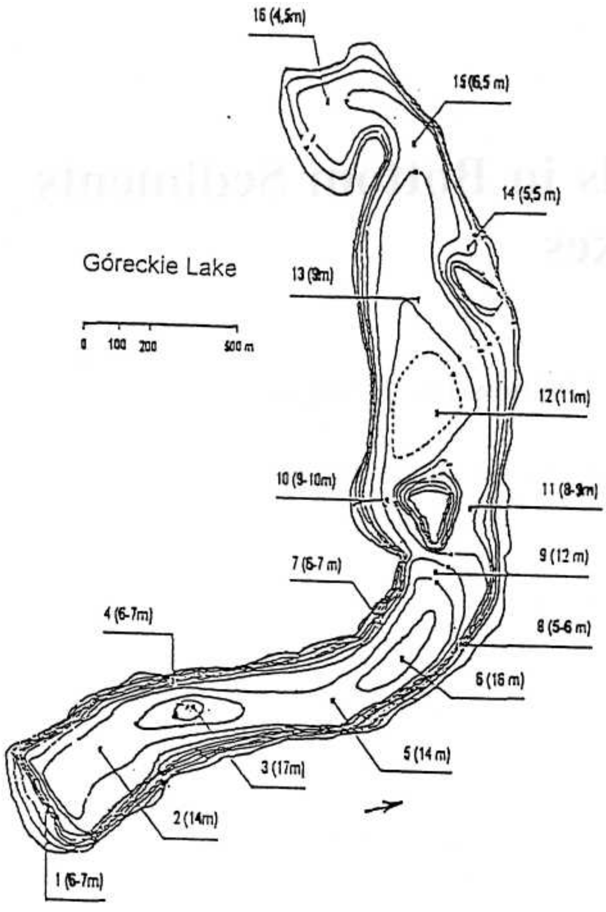
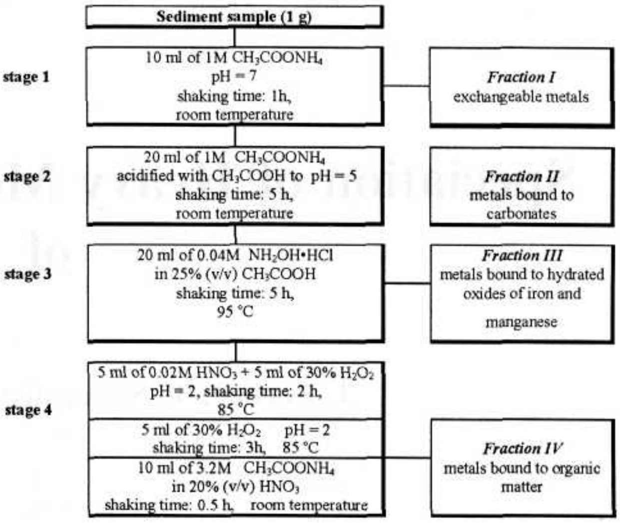


Fig. 1. Bathymetric map showing the sampling sites (the first number indicates the number of the site, while the number in parentheses is the depth).

moulded by the last glacial period, which also formed the Baltic. It is a typical moraine lake surrounded mostly by forest, only over a short section it borders with an agriculturally used area. Its length is about 3000 m and its mean width 347 m, so it covers an area of 103 ha. The length of its mostly undeveloped shoreline is 7300 m. The mean depth of this lake is 9.5 m and the maximum depth 17 m. A transversal underwater hump naturally divides the lake basin into two parts: a deeper southern basin and a shallower northwestern basin. Earlier investigations did not indicate the presence of anthropogenic contamination of the lake sediments by heavy metals [8].

The surface sediment samples (0-10 cm) were collected at 16 sites, 9 of which were in the south basin and 7 in the north-western one in the summer of 1995. The location of the sites and the depth from which the samples were taken are shown in Figure 1. The samples were taken from a boat by a stainless steel/glass pipe probe. In laboratory the samples were dried at 105°C, then ground in agate mortar and screened through a nylon sieve. The fraction of particle size < 0.2 mm was assigned for further studies. Prior to sequential extraction the samples were stored at -20°C.

The distribution of metal among particular forms was established by sequential extraction conducted according to the following diagram:



As mentioned above, the diagram of sequential extraction applied was slightly different from that proposed by Tessier et al. [4]. At the first stage, instead of $MgCl_2$ and CH_3COONa solutions, we used a solution of CH_3COONH_4. This way, due to the decrease of the salinity of the solutions, it was easier to determine metals by atomic absorption spectrometry (AAS). At the second stage we used a solution of CH3COONH4 acidified with CH_3COOH, increasing its volume in order to avoid possible changes of pH. The content of metals in fraction V (residual metals) was calculated by subtracting the sum of metal contents in fractions I-IV from the total concentration of metals determined by using a separate sample. The total content of metals was determined after sample digestion with concentrated HNO3 and 30% H_2O_2 [9].

All stages of extraction were carried out in glass centrifuge tubes closed with glass funnels to minimize evaporation. The samples were shaken by a horizontal shaker equipped with a thermostat. After each stage of extraction the sample was centrifuged for 20 min at 1400 g. The supernatant was removed with a pipette. After acidifying it with a drop of concentrated HNO_3 the content of heavy metals in the obtained solution was determined by the flame absorption atomic spectrometry (AAS) on a Varian spectrometer Spectr AA 20 plus. Cadmium was determined by using the graphite furnace technique. The concentration of particular heavy metals was expressed per 1 kg of air dry sample.

Between subsequent extractions, the precipitate remaining in a centrifuge tube was washed up with 10 ml of distilled water and centrifuged, and supernatant was discarded.

Preliminary analyses of sediment samples carried out in 10 replications have shown that relative standard deviaton (RSD) for heavy metals determination in fractions I, II, III and IV varied from 12% to 10%, 10% to 8%, 10% to 8% and 8% to 6%, respectively.

Results and Discussion

Tables 1-8 show the results of determination of the total content of particular metals in the samples studied and the percentage of the five fractions distinguished. In each case the presented value is a mean observed in three determinations. For the sake of better illustration, the mean values are shown as bar diagrams in Figure 2.

Analysis of the data obtained shows that iron occurred mainly in the residual fraction (46%) and in the form bound to organic matter (40%), and in much smaller amounts in the form of oxides (14%). Only minor amounts of iron were detected in the exchangeable and bound to carbonate fractions. These results are similar to the distribution of iron among particular fractions reported for bottom sediments of man-made lake Goczalkowice on the Vistula river. According to the studies by Kwapulinski and Wiechula [10], in the sediments of the above lake the greatest amount of iron was present in the residual fraction, but in the fraction bound to the oxides they found twice as much iron as in the fraction bound to organic matter. The same were the observations of Tessier et al. [4] for fluvial sediments in the rivers Yamaska and Saint-Francois in Canada. Moreover, the total content of iron, as well as other metals, was much greater in sediments from Canadian rivers and Goczalkowice man-made reservoir than in bottom sediments from Goreckie Lake.

Manganese was found in the sediments from Goreckie Lake mainly in the fraction bound to carbonates (41%) and hydrated oxides (31%). Less was determined in the exchangeable fraction (11%) and residual fraction (9.9%). Only 6.1% of the metal was in the fraction bound to organic matter. In the studies of the sediments in Goczalkowice reservoir, Kwapulinski and Wichula [10] observed a more homogeneous distribution of manganese among particular fractions, and the greatest amount of this metal in the fraction bound to oxides, while the lowest in the fraction bound to carbonates. In typical fluvial sediments Tessier et al. [4] determined the greatest amount of manganese in the residual fraction. In one of the rivers studied by them also a considerable amount of this metal was in the fraction bound to carbonates. In almost all cases the least amount of Mn was bound to organic fraction.

In the sediments from Goreckie Lake, zinc was found mostly in the fraction bound to hydrated oxides of iron and manganese (58%). Considerably smaller amounts of this metal were detected in the fraction bound both to carbonates (16%) and organic matter (16%), while the amounts found in the exchangeable and residual fractions were 8.6 and 1.9%, respectively. In the sediments from Goczalkowice reservoir, also the greatest concentration of zinc was in the fraction bound to hydrated oxides of iron and manganese. A significant amount of zinc was found in the residual fraction and in the fraction bound to organic matter, while a relatively small concentration of this metal was in the fraction bound to carbonates [10]. In sediments from the Canadian rivers the highest concentration of zinc was observed in the residual fraction, then in the fraction bound to hydrated oxides of iron and manganese, and a relatively small amount of this metal was found in the fraction bound to organic matter [4].

Nickel in sediments from Goreckie Lake was mainly found in the residual fraction (66%). Its contribution to the fractions bound to hydrated oxides of iron and manganese, organic matter and carbonates were 20%, 12% and 3.8%, respectively. A similar distribution of this metal was reported in fluvial sediments [4]. In sediments from Goczalkowice reservoir nickel was also found mainly in the residual fraction, although its significant amounts were bound to organic matter [10]. Different results were reported by Ure et al. [2] for sediments from Vegoritis Lake in Greece, where the greatest amount of nickel was present in the phase bound to organic matter and a smaller one in the residual fraction. The other forms of nickel made an insignificant contribution to total nickel contents.

Copper, likewise nickel, was mainly found in the residual fraction (83%). Much fewer amounts of this metal were bound to organic matter (16%) and hydrated oxides of iron and manganese (2.9%). Distribution of copper forms among particular fractions were similar both in sediments from Goczalkowice reservoir and in the Canadian rivers Yamaska and Saint Fran§ois [4, 10], although in the latter the contribution of copper in the residual fraction was relatively lower.

Lead in the sediments from the Goreckie Lake, was found mainly in the form bound to hydrated oxides of iron and manganese (34%), then in the residual fraction (34%), and bound to carbonates (21%). In a smaller amount it was bound to organic matter (10%) and exchangeable forms (0.8%). A similar distribution of lead forms among the five fractions was reported for the fluvial deposits by Tessier et al. [4]. In sediments from Goczalkowice reservoir, lead was found mainly in the residual fraction, although its significant amount was in the fraction bound to hydrated iron and manganese oxides and organic matter [10].

Cadmium was present mainly in the forms bound to organic matter (43%), in lesser amount in the fraction bound to hydrated iron and manganese oxides (28%) and in that bound to carbonates (18%) and the residual one (5.9%). In sediments from Goczalkowice reservoir, the cadmium form bound to organic matter made the greatest contribution and its amount in the residual fraction was rather significant, while its contribution to the first three fractions was very small [10]. The situation was different for fluvial sediments, Tessier et al. found only insignificant amounts of cadmium in the fraction bound to hydrated iron and manganese oxides, whereas in the other fractions cadmium concentrations were below the limit of detection [4].

Chromium, in the sediments from Goreckie Lake, was mainly found in the residual fraction (56%) and in that bound to organic matter (38%), while in smaller amounts in the fractions bound to hydrated iron and manganese oxides (7.2%). It was determined neither in the Goczalkowice sediments nor in the fluvial sediments from the Yamaska and Saint-Francois rivers. In the Vegoritis Lake sediments in Greece chromium was mainly found in the fraction bound to organic matter, then in smaller amounts in the residual fraction [2].

Conclusions

In bottom deposits from Goreckie Lake, the total amounts of metals determined were in the range: iron from 1700 mg/kg to 15000 mg/kg, mean value 9150 mg/kg; manganese from 78 to 690 mg/kg, mean value 350 mg/kg; zinc from 13 to 110 mg/kg, mean value 64 mg/kg; nickel from 4.5 to 21 mg/kg, mean 13 mg/kg; copper from 2.9 to 19 mg/kg, mean value 12 mg/kg; lead from 11 to 74 mg/kg,

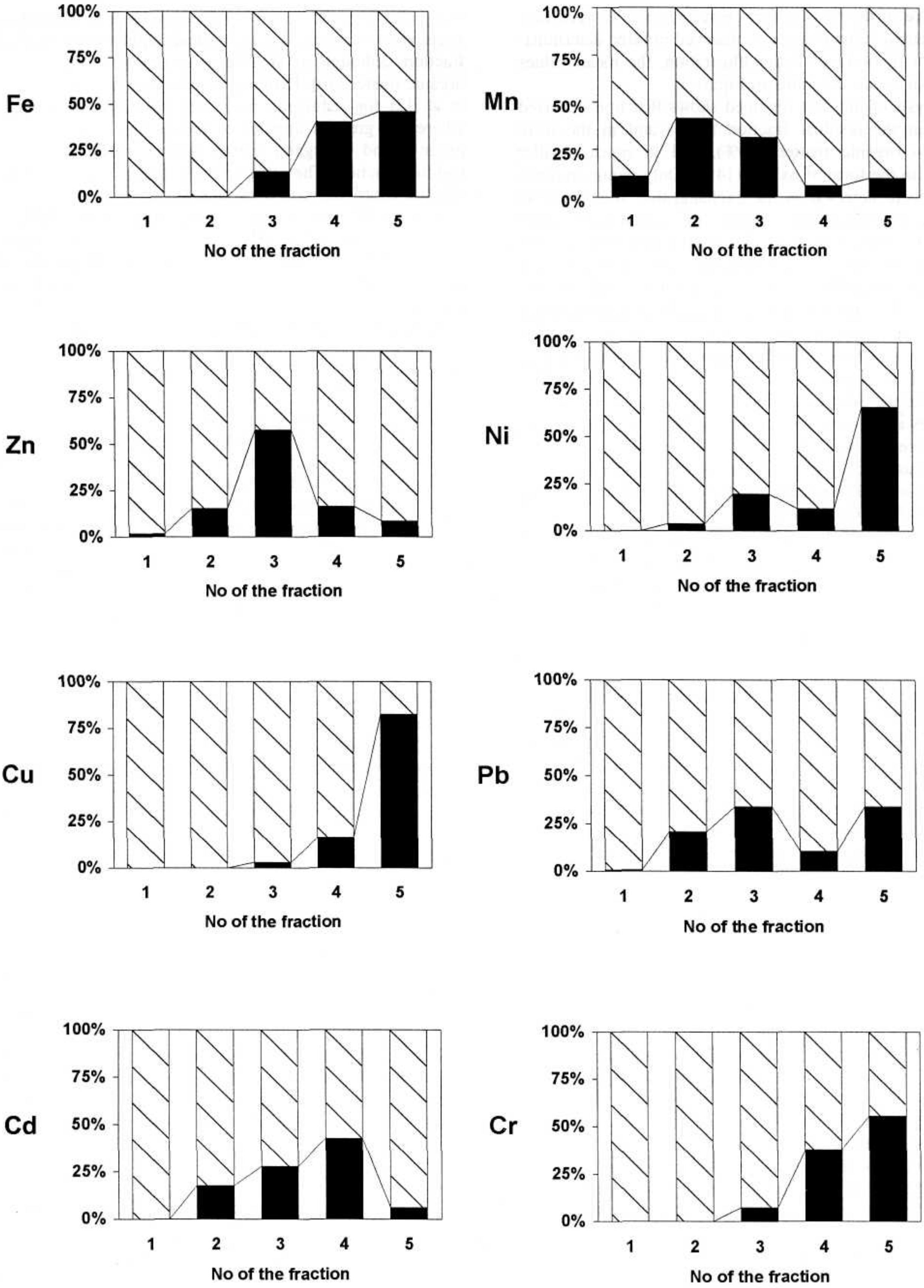


Fig. 2. Metal distribution among particular fractions.

Table 1. Total content of iron in Goreckie Lake bottom sediment samples, and its distribution among particular fractions.

No. of the site	Iron					
	Total content	Fraction I	Fraction II	Fraction III	Fraction IV	Fraction V
	mg/kg dry matter					
1	7100	5.8	8.1	930	3100	3100
2	14000	8.4	17	1700	4500	7800
3	13000	11	150	3200	4600	5000
4	1700	8.4	12	380	600	700
5	13000	n.d.	65	3000	4100	5800
6	14000	3.7	83	2600	4700	6600
7	4400	6.7	25	940	1700	1700
8	1900	7.2	18	35	320	1500
9	15000	n.d.	3.4	1200	5500	8300
10	4100	4.5	51	380	2700	960
11	7200	n.d.	1.6	1200	3100	2900
12	14000	3.7	17	1100	4600	8300
13	12000	n.d.	2.4	980	4600	6400
14	8300	2.7	25	940	4500	2800
15	8100	n.d.	n.d.	820	4800	2500
16	8900	2.0	n.d.	500	6000	2400
average value	9150	4.0	30	1250	3700	4200

n.d. = not detected

Table 2. Total content of manganese in Goreckie Lake bottom sediment samples, and its distribution among particular fractions.

No. of the site	Manganese					
	Total content	Fraction I	Fraction II	Fraction III	Fraction IV	Fraction V
	mg/kg dry matter					
1	310	23	130	86	12	59
2	470	26	170	150	33	91
3	690	76	260	250	70	34
4	78	15	39	19	4	1
5	490	48	240	170	21	11
6	470	52	230	120	37	31
7	110	11	52	27	7	13
8	210	39	100	37	n.d.	34
9	360	59	120	130	41	10
10	260	24	130	100	n.d.	6
11	270	36	130	50	n.d.	54
12	410	66	170	93	25	56
13	400	47	170	80	24	79
14	340	34	120	120	21	45
15	340	24	140	150	11	15
16	360	28	130	150	41	11
average value	350	38	145	110	21.5	34.5

n.d. = not detected

Table 3. Total content of zinc in Goreckie Lake bottom sediment samples, and its distribution among particular fractions.

No. of the site	Zinc					
	Total content	Fraction I	Fraction II	Fraction III	Fraction IV	Fraction V
	mg/kg dry matter					
1	36	n.d.	8.1	18	3	6.9
2	60	n.d.	10	31	12	7.0
3	94	n.d.	16	41	15	22
4	13	n.d.	2.3	9.6	0.5	0.6
5	60	n.d.	11	36	7.5	5.5
6	71	0.9	18	50	1.0	1.1
7	25	0.9	7.8	13	n.d.	3.3
8	13	0.7	1.3	9.4	0.8	0.8
9	83	n.d.	7.8	58	6.3	11
10	44	1.1	10	28	3.9	1.0
11	68	n.d.	14	39	9.4	5.6
12	99	n.d.	11	56	30	2.0
13	98	3.0	8.8	53	19	14
14	70	1.7	8.9	46	11	2.4
15	80	0.6	9.4	50	16	4.0
16	110	10	15	53	31	1.0
average value	64	1.2	9.95	37	10.5	5.5

n.d. = not detected

Table 4. Total content of nickel in Goreckie Lake bottom sediment samples, and its distribution among particular fractions.

No. of the site	Nickel					
	Total content	Fraction I	Fraction II	Fraction III	Fraction IV	Fraction V
	mg/kg dry matter					
1	12	n.d.	n.d.	2.4	1.5	8.1
2	19	n.d.	n.d.	4.3	2.4	12
3	19	n.d.	n.d.	2.3	5.8	11
4	6.7	n.d.	n.d.	n.d.	n.d.	6.7
5	21	n.d.	2.0	3.2	2.5	13
6	17	n.d.	1.6	4.2	1.7	9.5
7	5.1	n.d.	n.d.	n.d.	n.d.	5.1
8	13	n.d	n.d.	2.1	1.2	9.7
9	16	n.d.	1.3	4.2	1.1	9.4
10	4.5	n.d.	n.d.	n.d.	n.d.	4.5
11	12	n.d.	n.d.	3.0	1.0	8.0
12	13	n.d.	n.d.	3.0	1.9	8.1
13	11	n.d.	n.d.	3.2	1.4	6.4
14	15	n.d.	1.2	2.8	1.7	9.3
15	12	n.d.	n.d.	2.1	1.0	8.9
16	14	n.d.	1.3	3.9	1.5	7.3
average value	13	-	0.5	2.55	1.55	8.55

n.d. = not detected

Table 5. Total content of copper in Góreckie Lake bottom sediment samples, and its distribution among particular fractions.

No. of the site	Copper					
	Total content	Fraction I	Fraction II	Fraction III	Fraction IV	Fraction V
	mg/kg dry matter					
1	6.3	n.d.	n.d.	n.d.	2.8	3.5
2	15	n.d.	n.d.	n.d.	1.2	14
3	16	n.d.	n.d.	n.d.	5.0	11
4	2.9	n.d.	n.d.	n.d.	1.2	1.7
5	13	n.d.	n.d.	n.d.	n.d.	13
6	14	n.d.	n.d.	1.0	2.1	11
7	4.8	n.d.	n.d.	n.d.	n.d.	4.8
8	4.8	n.d.	n.d.	n.d.	1.8	3.0
9	18	n.d.	n.d.	1.2	2.1	15
10	7.8	n.d.	n.d.	n.d.	1.2	6.6
11	9.2	n.d.	n.d.	n.d.	1.7	7.5
12	19	n.d.	n.d.	1.0	3.0	15
13	18	n.d.	n.d.	1.3	2.9	14
14	13	n.d.	n.d.	n.d.	1.8	11
15	14	n.d.	n.d.	1.1	2.6	10
16	19	n.d.	n.d.	n.d.	1.7	17
average value	12	-	-	0.35	1.95	9.9

n.d. = not detected

Table 6. Total content of lead in Goreckie Lake bottom sediment samples, and its distribution among particular fractions.

No. of the site	Lead					
	Total content	Fraction I	Fraction II	Fraction III	Fraction IV	Fraction V
	mg/kg dry matter					
1	22	n.d.	8.7	8.4	2.0	2.9
2	59	n.d.	15	19	9.3	16
3	67	n.d.	18	26	4.3	19
4	11	n.d.	2.1	4.2	2.3	2.4
5	55	2.1	8.4	18	10	17
6	52	2.0	5.0	17	9.3	19
7	20	n.d.	5.2	6.0	4.7	4.1
8	24	n.d.	4.4	6.2	2.1	11
9	74	2.5	21	26	5.4	19
10	30	n.d.	5.0	8.0	2.0	15
11	50	n.d.	8.1	17	4.7	20
12	66	n.d.	12	22	5.0	27
13	66	n.d.	15	22	4.9	24
14	47	n.d.	8.4	19	3.8	16
15	50	n.d.	8.0	19	4.3	19
16	66	n.d.	13	20	5.1	28
average value	47.5	0.4	9.85	16	4.95	16

n.d. = not detected

Table 7. Total content of cadmium in Goreckie Lake bottom sediment samples, and its distribution among particular fractions.

No. of the site	Cadmium					
	Total content	Fraction I	Fraction II	Fraction III	Fraction IV	Fraction V
	mg/kg dry matter					
1	1.9	n.d.	n.d.	n.d.	1.6	n.d.
2	4.8	n.d.	1.3	1.3	1.7	0.5
3	5.2	n.d.	1.4	1.2	1.6	1.0
4	1.2	n.d.	n.d.	n.d.	1.0	n.d.
5	3.3	n.d.	n.d.	1.2	1.6	0.5
6	3.2	n.d.	n.d.	1.3	1.8	n.d.
7	1.8	n.d.	n.d.	n.d.	1.2	0.6
8	2.6	n.d.	n.d.	1.3	1.0	n.d.
9	4.7	n.d.	1.5	1.3	1.6	n.d.
10	1.9	n.d.	n.d.	n.d.	1.6	n.d.
11	2.6	n.d.	n.d.	1.2	1.0	n.d.
12	4.6	n.d.	1.4	1.2	1.8	n.d.
13	4.1	n.d.	1.4	1.2	1.4	n.d.
14	4.1	n.d.	1.3	1.3	1.2	n.d.
15	3.7	n.d.	n.d.	1.3	1.6	0.8
16	4.8	n.d.	1.4	1.5	1.6	n.d.
average value	3.4	-	0.6	0.95	1.45	0.2

n.d. = not detected

Table 8. Total content of chromium in Goreckie Lake bottom sediment samples, and its distribution among particular fractions.

No. of the site	Chromium					
	Total content	Fraction I	Fraction II	Fraction III	Fraction IV	Fraction V
	mg/kg dry matter					
1	6.5	n.d.	n.d.	n.d.	3.3	3.2
2	13	n.d.	n.d.	1.2	5.1	6.7
3	22	n.d.	n.d.	2.0	8.4	12
4	1.5	n.d.	n.d.	n.d.	n.d.	1.5
5	13	n.d.	n.d.	1.0	5.0	7.0
6	14	n.d.	n.d.	1.5	3.9	9.0
7	2.9	n.d.	n.d.	n.d.	1.3	1.6
8	3.3	n.d.	n.d.	n.d.	1.5	1.8
9	12	n.d.	n.d.	1.0	4.7	6.3
10	3.6	n.d.	n.d.	n.d.	1.2	2.4
11	8.5	n.d.	n.d.	n.d.	3.1	5.4
12	14	n.d.	n.d.	1.6	5.2	7.2
13	13	n.d.	n.d.	1.2	5.0	6.8
14	8.7	n.d.	n.d.	n.d.	2.8	5.9
15	8.3	n.d.	n.d.	n.d.	3.0	5.3
16	11	n.d.	n.d.	2.0	5.0	4.0
average value	9.7	-	-	0.7	3.65	5.4

n.d. = not detected

mean value 47.5 mg/kg; cadmium from 1.2 to 5.2 mg/kg, mean value 3.4 mg/kg; chromium from 1.5 to 22 mg/kg, mean value 9.7 mg/kg.

From among these metals only a few were detected in fraction I comprising exchangeable metal cations, and these were first of all manganese, in less amount iron and in very small amounts lead and zinc. The other metals were either sporadically found in this fraction or not found at all. In fraction II, bound to carbonates, manganese was also the most abundant, followed in the order of decreasing amounts by iron, zinc, lead, cadmium and nickel. Copper and chromium were not found in this fraction. Most of the metals studied were detected in fractions III, IV and V. In the fraction bound to hydrated oxides of iron and manganese, apart from these two metals, we found mainly zinc and lead, and in smaller amounts nickel, cadmium, chromium and copper. In the fraction bound to organic matter, the metals found in the greatest amounts were iron and manganese, and in less amounts zinc, lead, chromium, copper, nickel and cadmium. The residual fraction contained mainly iron, manganese, lead, copper, nickel, zinc, chromium and in insignificant amount cadmium.

Taking into account the fact that an earlier survey of local surroundings of Goreckie Lake did not show the presence of point sources of metal contamination [8], and that legal protection of the area considerably limits anthropogenic pollution, it can be assumed that the heavy metals found in the lake sediments are to a predominant extent of natural origin and occur at concentrations typical of the geochemical background. Therefore, the results of this speciation analysis can be used as a reference for investigating heavy metal content and speciation in other lake sediments. Moreover, they provide a reference for monitoring the effect of anthropopression on the natural water basins and for assessment of the actual hazard coming from heavy metals penetrating the environment as a result of human activities.

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