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ASSESSMENT OF THE CONTENT AND SOLUBILITY OF HEAVY METALS IN BOTTOM SEDIMENTS OF THE CHANCZA RESERVOIR

OCENA ZAWARTOŚCI I ROZPUSZCZALNOŚCI METALI CIĘŻKICH W OSADACH DENNYCH ZBIORNIKA CHAŃCZA

Abstract: The investigations aimed at an assessment of the content and solubility of heavy metals in bottom sediments of the Chancza water reservoir. The bottom sediments were sampled from previously identified cross-sections characteristic for individual reservoir zones: inlet – section 8; middle – section 5; the dam-side – section 1. Bottom sediment was sampled along each section, in three points in the vicinity of the right and left bank and in the middle of the cross-section. In order to average the collected material, the samples were collected from 6 points within the radius of 5–8 meters from the appointed location. The sediment was sampled using Ekman's sampler. Total contents of heavy metals (Zn, Cu, Ni, Cr, Pb and Cd) and their forms soluble in 1 mol HCl, in 0.01 mol $CaCl_2 \cdot dm^{-3}$ and in distilled water were assessed in air-dried samples of the sediments. Metal concentrations in the obtained solutions were assessed using ICP-AES method. The highest concentrations of heavy metals were assessed in the sediment samples collected on the reservoir inlet, then at the dam-side, whereas the lowest amounts were found in the samples from the middle part of the reservoir. Solubility of heavy metals depended on the extracting solution used and their total concentrations in the analyzed sediment. The best average solubility in 1 mol $HCl \cdot dm^{-3}$ characterized Pb, followed by Cd > Zn > Cu > Ni > Cr, whereas in 0.01 mol $CaCl_2 \cdot dm^{-3}$: Cu > Pb > Ni > Cr > Zn. Strong linear correlations were demonstrated between individual pairs of heavy metals in bottom deposits, which indicates their identical origin, most frequently connected with the natural content.

Keywords: bottom sediment, heavy metals, total content, soluble forms

Bottom sediments accumulated in water reservoirs constitute a very important part of ecosystems, play an important role in their functioning and element cycling between individual components of soil and groundwater system [1, 2]. Bottom sediments play

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the role of a natural filter and are indicators of the degree of environmental degradation. Heavy metals constitute a significant part of these pollutants, which at some concentrations may be toxic, they also reveal durability of forms enabling their migration to considerable distances. Moreover, elevated concentrations of heavy metals inhibit the processes of water self-cleaning, disturbing biological balance of water ecosystems. At this point it should be mentioned that the forms of trace heavy metals (total and soluble) occurring in the sediments are very important from the ecological point of view because some are toxic (Pb, Cd), whereas in excess all are harmful for living organisms [3–5].

Total element contents are very often taken into consideration because they are used as indicators of the degree of the environmental pollution [2, 4–8]. Still, total contents are not the best indicator of heavy metal bioavailability and mobility. The investigations aimed at an assessment of the content and solubility of heavy metals in bottom sediments of the Chancza water reservoir.

Materials and methods

Chancza retention reservoir is localized on the Czarna Staszowska River close to the Staszow town in the south-eastern part of the Swietokrzyskie province. The area is a part of The Kielce Uppland macroregion and particularly the Nida Basin macroregion and mezoregions of the Szydlowskie Upland and Orlowskie Range. The valley bottom is lined with alluvia built of coherent grounds (silts, loams and clays) and fine or medium grained quartz sands. The Czarna Staszowska River is a left bank tributary to the Vistula River, whereas its waters and Chancza reservoir waters are classified to III water purity class [9]. The main tasks of the reservoir which was commissioned for operation in 1984 comprise: meeting the current water requirements of industrial and municipals users, flood waves reduction and compensation of water flows, conditions for tourism and recreation. Earth dam with a concrete spillway and outlet block
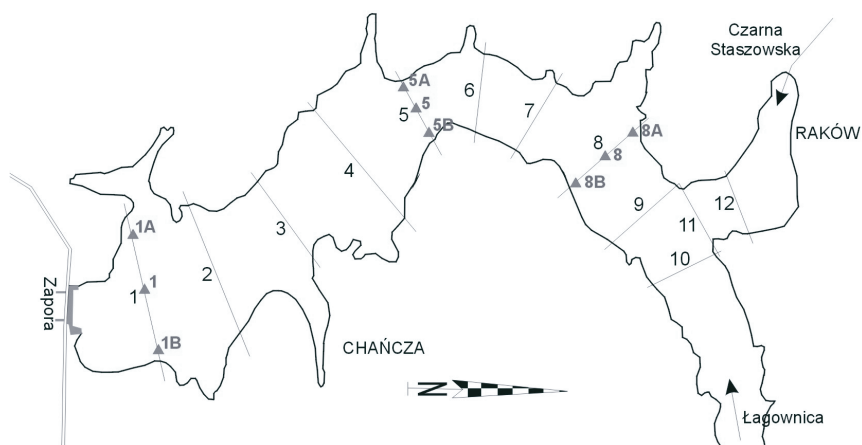

Fig. 1. Chancza water reservoir with marked measuring cross sections and the places of collected bottom sediment

closes the 475 km^2 river basin enabling formation of a reservoir with 24 Mm^3 capacity and 11 m deep. Field works were conducted in September 2009. The bottom sediments were sampled from previously identified cross-sections characteristic for individual reservoir zones: inlet – section 8; middle – section 5; the dam-side – section 1 (Fig. 1).

Bottom sediment was sampled along each section, in three points in the vicinity of the right and left bank and in the middle of the cross-section. In order to average the collected material, the samples were collected from 6 points within the radius of 5–8 meters from the appointed location. The sediment was sampled using Ekman's sampler. The bottom sediments were classified to the deposits with granulometric composition of loamy sand (cross-section 1), loam (cross-section 5) and silt loam (cross-section 8) with neutral pH (Table 1).

Table 1

Selected properties of bottom sediments

Cross sections	pH		CaCO$_3$	Organic matter	C-organic	N-total	Fe
	H$_2$O	KCl		[%]		[g · kg^{-1} d.m.]	
1	7.16	7.01	3.20	9.97	38.66	2.87	14.54
5	7.31	7.22	2.10	6.08	36.65	2.23	11.30
8	7.04	6.86	2.30	11.22	31.37	3.31	18.92

Total contents of heavy metals (Zn, Cu, Ni, Cr, Pb and Cd) and their forms soluble in 1 mol HCl, in 0.01 mol CaCl$_2$ · dm^{-3} and in distilled water were assessed in air-dried samples of the sediments. Total contents of heavy metals in the sediments were assessed after hot mineralization in a mixture of HNO$_3$ and HClO$_3$ acids (3:2). Extraction of the soluble metal forms from the deposits was conducted using static method through a single shaking of sediment samples with the solution at the sediment to solution ratio 1:10 and extraction time 1 h (1 mol HCl · dm^{-3}) and 2 hrs (0.01mol CaCl$_2$ and H$_2$O) [8, 10, 11]. Metal concentrations in the obtained solutions were assessed using ICP-AES method on JY 238 ULTRACE apparatus, (Jobin Von Emission). Metals soluble 1 mol HCl · dm^{-3} allow to estimate their mobilization in result of acidification of the environment in which they are deposited. Application of calcium chloride makes possible isolation from the sediments of a readily available heavy metal fraction which under natural conditions may release from the sediments posing a real hazard to ground and water environment [4]. On the other hand, water soluble components will mobilize and translocate from the sediments under the influence of precipitation water, immediately after their supply to the environment. Statistical computations comprised the basic parameters: arithmetic mean, standard deviation, median, minimum, maximum, variability coefficient and Person correlation coefficient. Statistical analysis was conducted using Statistica 8.0 programme.

Results

Total contents of heavy metals in the samples of analyzed bottom deposit ranged from 61.50–212.00 mg Zn; 6.50–89.60 mg Cu; 5.25–30.20 mg Cr; 5.05–29.90 mg Ni;

13.90–43.15 mg Pb; trace – 0.86 mg Cd · kg^{-1} d.m. (Table 2). In view of their quantity, heavy metals in the deposits, irrespective of the analyzed cross-section, formed the following order: Zn > Cu > Pb > Cr > Ni > Cd, whereas concerning their diversification, the order was as follows Cu > Ni > Zn, Cd > Cr > Pb (Table 2). The highest metal concentrations were assessed in cross-section 8 on the inlet to the reservoir, the same samples contained also the highest amount of colloidal fraction, then in dam-side cross-section 1, where the highest proportion (ca 92 %) of sand fraction was determined. The lowest concentrations of heavy metals were noted in cross-section 5 in the middle part of the reservoir, where silt fraction dominated. Heavy metal contents in the bottom sediments collected from the middle of the reservoir (cross-section 5) were by 52 % Zn, 45 % Cu, 62 % Ni, 56 % Cr, 48 % Pb and 31 % Cd lower in comparison with the sediments sampled on the reservoir inlet (cross-section 8).

Table 2

Total content of heavy metals in bottom sediment

Cross sections	Zn	Cu	Ni	Cr	Pb	Cd
	[mg · kg^{-1} d.m.]					
1	111.83	23.10	13.87	17.90	28.45	0.50
1A	93.75	51.10	10.45	17.57	19.13	0.44
1B	104.00	55.65	11.50	20.48	20.88	0.47
Average	104.43	45.46	12.21	18.54	23.62	0.47
5	62.33	7.14	5.42	6.67	14.50	0.29
5A	72.50	41.55	6.65	11.73	14.75	0.47
5B	99.00	41.00	10.40	17.35	20.73	0.43
Average	75.71	27.14	7.19	11.16	16.35	0.36
8	203.17	15.90	26.15	28.60	41.10	0.36
8A	134.25	89.20	13.65	24.90	24.63	0.85
8B	107.75	60.20	13.22	21.28	23.52	0.62
Average	156.21	49.41	18.89	25.45	31.37	0.52
Statistical parameters						
Average	112.12	40.67	12.76	18.39	23.78	0.50
Standard deviation	43.51	24.4	6.37	6.90	8.62	0.20
Median	105.00	44.65	11.90	18.30	21.50	0.51
Minimum	61.50	6.50	5.05	5.25	13.90	trace
Maximum	212.00	89.60	26.90	30.20	43.15	0.86
V%	39	61	50	38	36	39
Norm[a]	< 1000	< 150	< 75	< 200	< 200	< 7.5
Geochemical background[b]	25–50	10–20	< 10	< 10	< 25	1–3

[a] [18], [b] [19].

Numerous investigations have shown that the content of individual metals grows from the most coarse to the finest fraction [1, 12–14]. Obtained results did not corroborate this dependence because no typical phenomenon of grain segregation, encountered on the other hydraulic structures, occurs in the Chancza reservoir. It may

be caused by a considerable shallowing of the reservoir inlet part, overgrowing with aquatic vegetation or unprepared bowl, ie not removed vegetation including trunks and roots of trees and bushes. Persistent low-level water states caused that the supplied bedload was stopped before the reservoir inlet. The situation changes during high water states when the flood wave carries coarser bottom material into further reservoir zones at the same time floating fine mineral fractions. Diminishing the reservoir width in the middle zone causing a local increase in velocity may contribute to increased floating force of the particles secondarily included in the sediment transport. Sand deposits in the dam-side zone may also constitute of abrasion products (banks are sandy beaches) and indigenous deposits lining the Czarna Staszowska River valley.

Beside knowledge about the total heavy metal content in the sediment, also knowing their readily soluble forms is most useful because of their possible mobilization from the solid phase and penetrating into aquatic environment. The content or chemical element forms readily soluble in distilled water and 0.01 mol $CaCl_2$ and in 1 mol $HCl \cdot dm^{-3}$ in the analyzed sediment may inform about bottom deposits effect on the environment (Table 3).

Table 3

Content of soluble forms in H_2O; 0.01 mol $CaCl_2$ and 1 mol $HCl \cdot dm^{-3}$ heavy metals in botom sediment

Cross-sections		Zn	Cu	Ni	Cr	Pb	Cd
		$[mg \cdot kg^{-1}$ d.m.]					
1		0.06	0.63	n.o.[a]	n.o.	0.06	n.o.
5	H_2O	0.02	0.42	n.o.	n.o.	0.04	n.o.
8		0.07	1.18	n.o.	n.o.	0.08	n.o.
Average ±SD		0.05 ± 0.03	0.74 ± 0.49	—	—	0.06 ± 0.02	—
% total		0.04	1.82	—	—	0.25	—
V %		53	66	—	—	31	—
1		0.51	1.99	0.07	0.08	0.25	n.o.
5	$CaCl_2$	0.06	1.30	0.04	0.04	0.14	n.o.
8		0.71	3.32	0.09	0.11	0.12	n.o.
Average ±SD		0.42 ± 0.30	2.22 ± 0.97	0.06 ± 0.02	0.07 ± 0.02	0.17 ± 0.7	—
% total		0.37	5.46	0.47	0.38	0.71	—
V %		71	44	40	33	41	—
1		39.19	8.26	3.58	1.31	13.04	0.13
5	HCl	32.94	6.20	2.07	0.87	9.57	0.06
8		81.44	14.08	2.72	2.15	16.69	0.52
Average ±SD		51.18 ± 25.3	9.51 ± 4.8	2.79 ± 1.7	1.44 ± 0.66	13.81 ± 7.1	0.23 ± 0.2
% total		45.65	23.38	21.87	7.83	58.07	48.00
V %		49	51	61	46	51	99

[a] n.o. – undetectable value.

Heavy metal solubility assessed in the conducted analyses depended primarily on their total content in bottom deposits. This was true for zinc, copper, chromium, lead

and cadmium, whose highest total content and solubility were registered in the sediments collected in cross section 8 on the reservoir inlet, whereas lower in dam-side cross-section 1 and the lowest in cross-section 5 (Table 3, Fig. 2).
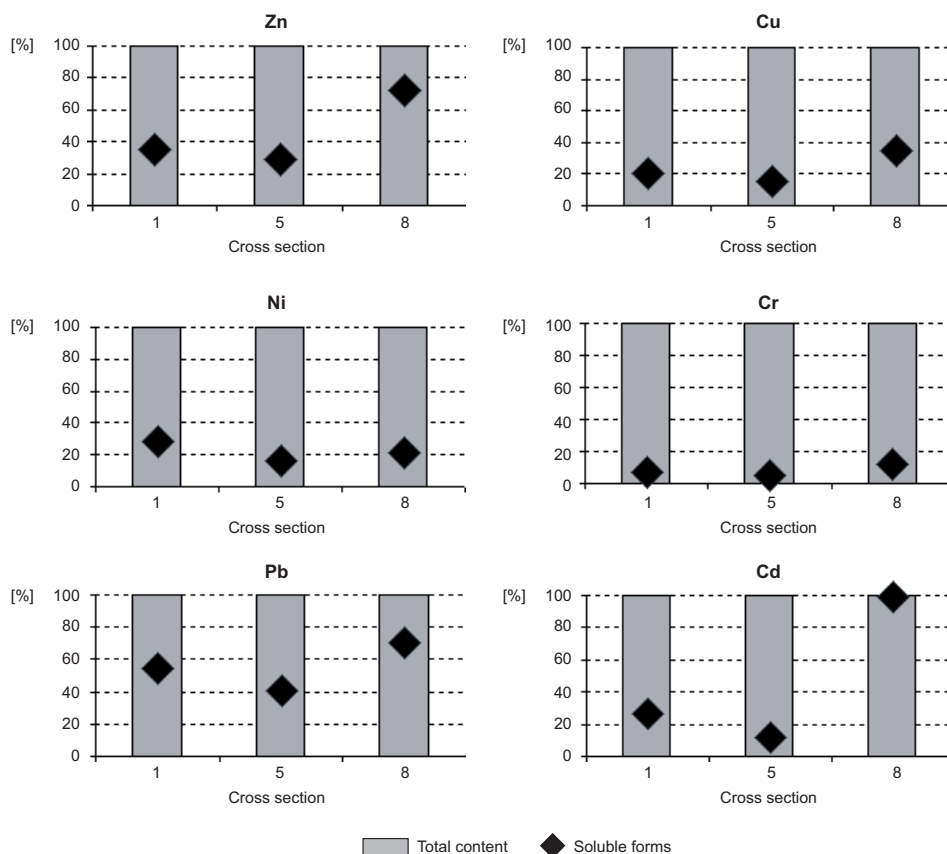

Fig. 2. Percent share of available forms (1 mol HCl · dm^{-3}) of trace elements in their total contents in bottom sediment

Heavy metal solubility in distilled water and calcium chloride was very poor, which is connected with low concentration of hydrogen ions in the analyzed sediments (Table 1). Heavy metal solubility in distilled water and calcium chloride with reference to their total content constituted respectively: 0.04 and 0.37 % for Zn; 1.82 and 5.46 % for Cu and 0.25 and 0.71 % for Pb; 0.47 % for Ni and Cr (0.01 mol CaCl$_2$) (Table 3). The solubility in 1 mol HCl · dm^{-3} was better and depending on the sampling place ranged from 34.95 to 72.64 % (zinc), from 15.24 to 34.62 % (copper) from 16.22 to 28.06 % (nickel), from 4.73 to 11.69 (chromium), from 40.24 to 70.19 (lead) and from 12 to almost 100 % (cadmium) in comparison with their total content (Fig. 2).

In the opinion of many authors the 0.01 mol CaCl$_2$ · dm^{-3} solution, unlike 1 mol HCl · dm^{-3} solution, is counted among solutions with low extraction power and ability

for assessing so called bioavailable (active) fraction of heavy metals [7, 4]. In the presented research as may have been expected, lesser amounts of the analyzed elements were assessed in the 0.01 mol $CaCl_2 \cdot dm^{-3}$ extract (Table 3). Generally, hydrochloric acid leaches metals bound to exchangeable, carbonate, Fe/Mn oxides and organic matter fractions. Copper, lead, chromium and nickel are commonly regarded as the least mobile elements in the environment, zinc and cadmium are counted among the most mobile ones, whereas manganese and iron place in the middle [15]. Considering the elements analyzed in the presented investigations, the best average solubility in 1 mol $HCl \cdot dm^{-3}$ characterized Pb, followed by Cd > Zn > Cu > Ni > Cr , whereas in 0.01 mol $CaCl_2 \cdot dm^{-3}$: Cu > Pb > Ni > Cr > Zn. Numerous investigations have shown that sediment reaction may affect heavy metal mobility in the environment [1, 14]. An opinion has been prevailing that metals may pass into less soluble forms at higher pH values [14, 16]. In the presented research a significant influence of the sediment reaction was observed on diminishing solubility of most of the investigated elements. This phenomenon has been corroborated by negative and significant values of correlation coefficients between pH value and element content in the sediments (Table 4). Linear dependence between individual heavy metals in the sediment may result from their geochemical bonds but may also indicate their mobility and sources of origin [1]. The existence of such dependencies has been checked in the analyzed sediments. For this purpose linear correlation coefficients were computed between individual pairs of metals (Table 4). In the studied bottom sediments a strong dependence was found between the respective pairs of metals: Zn and Cr, Ni, Pb; Ni and Cr, Pb and Cr and Pb with linear correlation coefficient exceeding 0.8 and $p \leq 0.001$ and Cu and Cd (Table 4). Strong linear correlations between individual pairs of heavy metals confirm their identical origin, frequently associated with natural content. In the analyses of the interrelations between the content of various metals, iron was regarded individually. The correlation between iron content and the concentration of other element may allow to differentiate the sediments with natural content of element from those enriched in result of human activity [2, 17]. A lack of significant correlation between iron and another heavy metal may point to their anthropogenic origin [1]. In samples analyzed in the presented research, in most cases Fe was significantly positively correlated with Zn, Cr, Pb and Ni, non-significantly with Cu and Cd; yet, at the same time the amount of these metals in the samples has been relatively small. A comparison of heavy metal values in the samples with values of geochemical background from reservoir localization area, shows that only Cd concentration within the whole reservoir did not exceed the background values. Ni and Pb concentrations in the reservoir middle zone were lower than the values of geochemical background, whereas the values for the other metals (Zn, Cu and Cr) exceeded these value considerably (Table 2). It has been also confirmed by the assessment of the degree of the studied sediments pollution as stated in the Decree of the Minister of the Natural Environment dated 16 April 2002 on the kinds and concentrations of substances which cause that the yield is polluted [18]. According to the above-mentioned criterion, bottom sediments from the Chancza Reservoir were not polluted with heavy metals (Table 2).

Table 4

The correlation coefficients between content of heavy metals, organic matter
and pH in bottom sediment

Parameters		Total content						pH	
		Zn	Cu	Ni	Cr	Pb	Cd	H$_2$O	KCl
Total content	Cu	0.07							
	Ni	0.98***	−0.08						
	Cr	0.87***	0.42	0.84***					
	Pb	0.95***	−0.14	0.98***	0.80***				
	Cd	0.17	0.69**	0.14	0.45	0.19			
Soluble forms	Zn	0.94***	0.08	0.90***	0.81***	0.87***	0.25	−0.56*	—
	Cu	0.82***	−0.07	0.84***	0.71***	0.82***	0.04	0.55*	—
	Ni	−0.32	0.68**	−0.35	0.02	−0.29	0.78***	−0.14	−0.15
	Cr	0.94***	−0.04	0.91***	0.77***	0.91***	0.25	−0.50*	—
	Pb	−0.01	0.97***	−0.08	0.42	−0.16	0.55*	−0.55*	−0.35
	Cd	0.82***	−0.45	0.82***	0.49*	0.78***	−0.33	−0.24	—
pH	H$_2$O	−0.62**	−0.55*	—	—	−0.56*	−0.48*		
	KCl	—	−0.37	—	—	−0.67**	−0.35		
Org. matter		0.89***	−0.13	0.92***	0.77***	0.95***	0.28		
Fe		0.91*	0.34	0.89*	0.87*	0.86*	0.17		

Significant ***p $\leq$ 0.001, **p $\leq$ 0.01, *p $\leq$ 0.05.

Summing up, heavy metals form a protective barrier against metal penetration to waters but at the same time may be a potential source of pollution in result of physicochemical changes in the soil and groundwater environment. Moreover, determining the level of heavy metal solubility in bottom sediments is very important because of a potential of their mobilization from the solid phase and penetration to aquatic environment where they become bioavailable [1]. The issue has been addressed in the presented paper which investigated total heavy metal content and their soluble forms.

Conclusions

1. The analyzed sediments do not reveal heavy metal contamination level which would pose a hazard to the aquatic or terrestrial environment. However, their Zn, Cu and Cr concentrations have been higher than geochemical background for water sediments from the area where the reservoir is situated.

2. Total heavy metal contents in the investigated bottom sediment were as follows: 61.50–212.00 mg Zn; 6.50–89.60 mg Cu; 5.25–30.20 mg Cr; 5.05–29.90 mg Ni; 13.90–43.15 mg Pb; trace – 0.86 mg Cd $\cdot$ kg^{-1} d.m.

3. The highest concentrations of heavy metals were assessed in the sediment samples collected on the reservoir inlet, then at the dam-side, whereas the lowest amounts were found in the samples from the middle part of the reservoir.

4. Solubility of heavy metals depended on the extracting solution used and their total concentrations in the analyzed sediment.

5. A considerable leachability of heavy metals with 1 mol HCl · dm^{-3} was established, which may indicate a potential threat for the environment from the lecheates from the stored dredged sediment.

6. The best average solubility in 1 mol HCl · dm^{-3} characterized Pb, followed by Cd > Zn > Cu > Ni > Cr , whereas in 0.01 mol CaCl$_2$ · dm^{-3}: Cu > Pb > Ni > Cr > Zn.

7. Strong linear correlations were demonstrated between individual pairs of heavy metals in bottom deposits, which indicates their identical origin, most frequently connected with the natural content.

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OCENA ZAWARTOŚCI I ROZPUSZCZALNOŚCI METALI CIĘŻKICH W OSADACH DENNYCH ZBIORNIKA CHAŃCZA

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Abstrakt: Celem badań była ocena zawartości i rozpuszczalności metali ciężkich w osadach dennych zbiornika Chańcza, zlokalizowanego na rzece Czarnej Staszowskiej (woj. świętokrzyskie). Próbki osadu dennego pobrano z wyznaczonych przekroi będących charakterystycznymi dla danej strefy zbiornika: wlotowej – przekrój 8, środkowej – przekrój 5, przy zaporowej – przekrój 1. Wzdłuż każdego z przekrojów pobrano osad denny w trzech punktach w okolicach brzegów (prawego, lewego) oraz w środku przekroju.

W celu uśrednienia pobieranego materiału próbki pobrane były z 6 miejsc w promieniu 5–8 metrów od wyznaczonej lokalizacji. Pobranie osadu wykonano za pomocą próbnika Ekmana. W powietrznie suchych próbkach osadów oznaczono ogólną zawartości metali ciężkich (Zn, Cu, Ni, Cr, Pb, Cd) oraz ich formy rozpuszczalne w 1 mol HCl, w 0,01 mol $CaCl_2 \cdot dm^{-3}$ i w wodzie destylowanej metodą ICP-AES. Największą zawartość metali ciężkich wykazano w próbkach osadów pobranych na wlocie zbiornika, następnie przy zaporze, a najmniejszą w środkowej części zbiornika. Rozpuszczalność metali ciężkich była uzależniona od użytego roztworu ekstrahującego oraz całkowitej ich zawartości w badanym osadzie. Spośród badanych metali największą średnią rozpuszczalnością w 1 mol $HCl \cdot dm^{-3}$ charakteryzował się Pb, następnie Cd > Zn > Cu > Ni > Cr, natomiast w 0,01 mol $CaCl_2 \cdot dm^{-3}$: Cu > Pb > Ni > Cr > Zn. Wykazano silne korelacje liniowe pomiędzy poszczególnymi parami metali ciężkich w osadach dennych, co wskazuje na jednakowe ich pochodzenie, najczęściej związane z zawartością naturalną.

Słowa kluczowe: osad denny, metale ciężkie, zawartość całkowita, formy rozpuszczalne