

# The content and composition of organic matter in bottom sediments of the Rybnik reservoir – preliminary studies

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**Abstract:** Organic matter has important influences on the fate of environmental pollution in water dam reservoirs. The aim of the studies was to assess content of organic matter fractions in Rybnik reservoir bottom sediments, and to determine their influence on the content of heavy metals and PAHs. In three sediment samples, the content of C organic (Corg), C extracted (Cex), C humic acid (Cha), C fulvic acid (Cfa) and C non-hydrolyzed (Cnh), buffer capacity, and content of heavy metals and PAHs was analyzed. We found the highest content of Corg, Cex, Cha, Cfa and Cnh in sample 3 (the outlet, near the dam), while the lowest content was found in sample 2 (middle). The fraction of Cnh was dominant in sediment sample 3 (outlet, near dam) and 1 (inlet), whereas the fraction of Cha dominated in sample 2 (middle). Rybnik bottom sediments are characterized by their high buffer capacities (samples 3, 1) shaped among others by the high content of organic matter. The high organic matter content in samples 3 (outlet) and 1 (inlet) and also high total contents of heavy metals and PAH<sub>s</sub> in these sediments demonstrate that these pollutants have a strong affinity for organic matter in the sediments. Moreover, the highest mobility of metals, from sample 2 (middle), is connected with the low content of organic matter substances and the low buffer capacities of sediments.

**Keyword:** bottom sediments, organic matter, buffer capacity, pollutions

## INTRODUCTION

Bottom sediment in reservoirs originates from the sedimentation of particulate matter delivered by a river, slope runoff and the precipitation of detritus. A wide variety of contaminants, including heavy metals, PAHs, PCB, dioxins and chloroorganic pesticides, are discharged into the aquatic environment and accumulate in sediments (Boháček et al. 2003, Szarek-Gwiazda 2014,

Urbaniak et al. 2015, Baran et al. 2016, 2017, Marmontova et al. 2016). The contents of the above substances in sediments may be three times higher than those found in water, and although certain chemicals are strongly sorbed to sediment, they may still be available for biota, thus causing a risk to benthic organisms (Tuikka et al. 2011, Bojakowska & Krasuska 2014). Organic matter is one of the most important components of bottom sediments, determining their physical, chemical

and biological properties, and is mainly responsible for the sorption properties of bottom sediments. Moreover, since organic matter in bottom sediment is an accumulating medium, it can provide important information about the pollution level of a water body, including pollution by heavy metals and organic pollutants (PAHs, PCB, dioxins) (Boháček et al. 2003). In the sediments, humic substances account for about 60–70% of the total organic matter and are recognized as the most persistent storage of sedimentary organic carbon (Hou et al. 2014). Humic substances have an important influence on the sorption, distributions, transport and fate of environmental pollution (Oleszczuk 2007, Mengchang et al. 2008, Giovanela et al. 2010, Morales et. al. 2012, Ukalska-Jurga et al. 2015).

The aims of the studies were as follows: to assess the content of organic matter fractions in bottom sediment collected from the Rybnik reservoir, and to determine their impact on the content of heavy metals and PAHs.

## METHODS

The Rybnik dam reservoir is one of the most important reservoirs in the Silesian area and it is the only anthropogenic-type reservoir in Poland (Fig. 1). The Rybnik reservoir is situated on the Ruda River, impounding a catchment with an area of 308 km<sup>2</sup>, the long side of the reservoir is 7 km long and its initial capacity was 25.8 mln m<sup>3</sup> in the impoundment area, together with lateral impoundments of 555 ha (Baran et al. 2016). The functions of the reservoir include a source of cooling water for the Rybnik Power Plant JSC, flood control, recreation and waterfowl habitat.

The bottom sediment samples were collected using an Ekman sampler from three characteristic set locations: 1 – inlet (backwater station), 2 – middle and 3 – outlet (close to the dam) (Fig. 1). In order to average the collected material, the samples were collected from five points within a radius of 5–8 meters from the appointed location. The top layer of the sediment was collected from 0–15 cm. All sediment samples were refrigerated until analyzed.

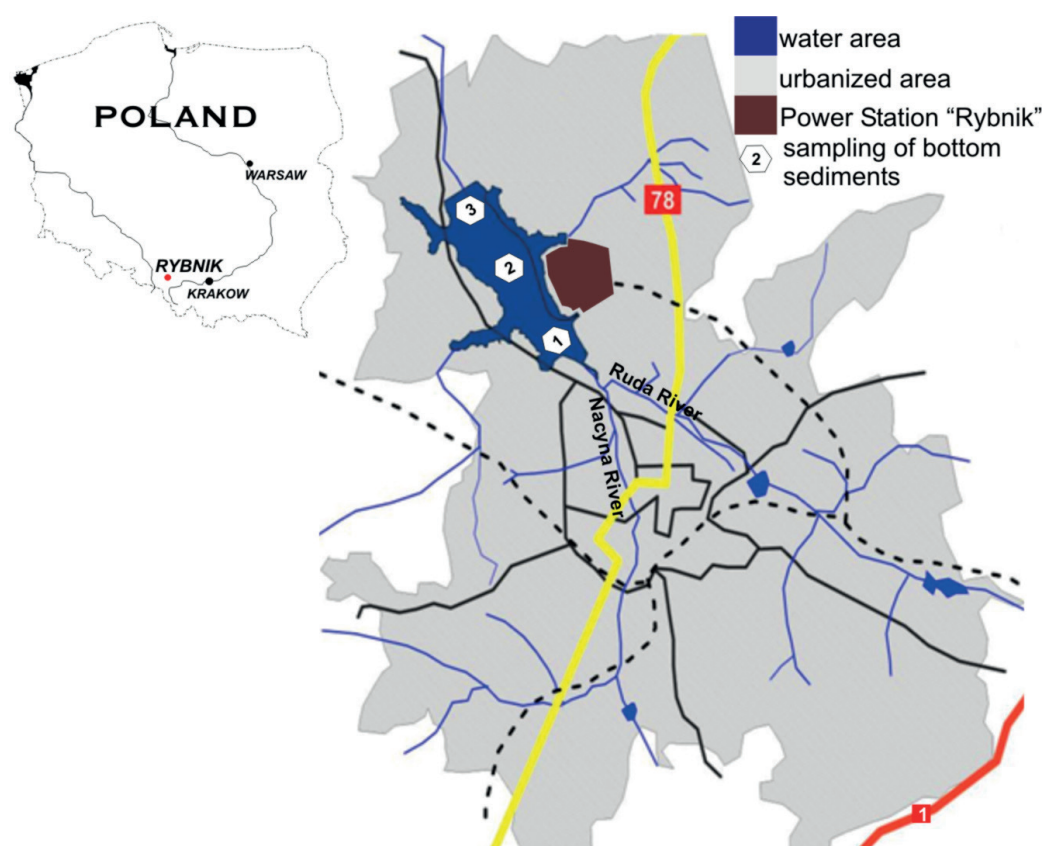


Fig. 1. Localization of Rybnik reservoir

The organic carbon content, after mineralization in potassium dichromate (VI), was assessed by means of Tiurin's method. The content of humus compounds was extracted from sediments by a solution mixture of  $0.1 \text{ mol}\cdot\text{dm}^{-3} \text{ Na}_4\text{P}_2\text{O}_7$  +  $0.1 \text{ mol}\cdot\text{dm}^{-3} \text{ NaOH}$  (Kononowa 1968). The carbon of humic acids (Cha) was isolated in an extract of sodium pyrophosphate and sodium base, whereas the carbon of fulvic acids (Cfa) was calculated from the difference between the amount of carbon in the extract (Cex) and the amount of humic acid carbon (Cha) contained in the extract. The extraction residue a non-hydrolyzing carbon (Cnh) was computed from the difference between the organic carbon content (C org) and the amount of carbon in the extract (Cex) (Gondek & Kopec 2006, Gondek & Mierzwa-Hersztek 2014). The buffer capacity of sediments, with an increase in acidity, was determined by the Arrhenius method (Baran et al. 2011a). In the sediment, the buffer was assessed after adding to the sediment an appropriate amount of  $0.1 \text{ mol}\cdot\text{dm}^{-3} \text{ HCl}$  solution (2, 4, 6, 8 and  $10 \text{ cm}^3$ ) and a subsequent measurement of changes in the pH value. This method consists in plotting the curves of the buffer on the basis of measurements of the pH series of the weighed amounts of bottom sediment suspended in an acid solution of  $0.1 \text{ mol}\cdot\text{dm}^{-3} \text{ HCl}$  ( $0\text{--}10 \text{ cm}^3$ ) at a solid phase/solution ratio of 1:10. After 24 h, the pH was measured. The total contents of heavy metals (Zn, Cu, Ni, Cr, Pb and Cd) and their forms soluble in  $1 \text{ mol}\cdot\text{dm}^{-3} \text{ HCl}$  were assessed in air-dried samples of the sediments. Pseudo total metal contents in the sediments were assessed after dry and hot digestion in a mixture of  $\text{HNO}_3$  and  $\text{HClO}_3$  acids (3:2). The extraction of metal from the sediments was conducted using the static method through a single shaking of sediment samples with  $1 \text{ mol}\cdot\text{dm}^{-3} \text{ HCl}$  at the sediment to a solution ratio of 1:10 and an extraction time of 1 h. (Baran et al. 2011a, 2011b). The metal concentrations in the obtained solutions and extracts were assessed using the ICP-OES method with the use of Optima 7300 DV PerkinElmer. The reference material CRM 16-05 (trace elements) was used for the accuracy of the analyses. The percentage of recovery was between: 95–112% for Zn; 81–98% for Cu; 98–114% for Pb; 88–99% for Cr; 78–98% for Cd and 96–107% for Ni.

Concentrations of the 9 PAHs (naphthalene (NAP), fluorene (FLN), phenanthrene (PHE), anthracene (ANT), fluoranthene (FLT), pyrene (PYR), benzo(a)anthracene BAA, chrysene CHR, benzo(a)pyrene (BAP)) were determined by gas chromatography-mass spectrometry on a Varian GC/MS/MS 4000 equipped with an ion trap (Baran et al. 2017). The recoveries for PAHs were: 98.5% (NAP); 86.4% FLN; 89% (PHE); 88% (ANT); 90% (FLT); 85% (PYR); 84% (BAA); 80% (CHR); 83% (BAP). Moreover, the limit of detection (LOD) was  $2 \mu\text{g}\cdot\text{kg}^{-1} \text{ DM}$  and the limit of quantification (LOQ) was  $6 \mu\text{g}\cdot\text{kg}^{-1} \text{ DM}$ .

Statistical computations comprised the following basic parameters: arithmetic mean, standard deviation and correlation coefficient. The statistical analysis was conducted using the Statistica 12.0 software.

## RESULT

The content and quality of organic matter is an important characteristic of bottom sediments from the perspective of the physicochemical conditions at the reservoir bottom. The carbon content in the fractions of organic matter in surface sediments was presented in Table 1. Generally, the highest content of C organic, C extracted, Cfa and Cnh was found in sample 3 (outlet), while the lowest content was found in sample 2 (middle). However, the highest content of Cha was observed in bottom sediments from sample 3 (outlet) and the lowest content in sample 1 (inlet). The content of C extracted accounted for 32 (sample outlet – 3) – 79% (sample middle – 2), whereas the C non-hydrolyzing (Cnh) accounted for about 21 (sample middle – 2) – 68% (samples 3) of the total organic C in the sediment samples. The contents of Cha, Cfa and Cnh in the sediment samples were detected in the range of  $0.96\text{--}5.50 \text{ g}\cdot\text{kg}^{-1}$  (Cha),  $1.21\text{--}5.85 \text{ g}\cdot\text{kg}^{-1}$  (Cfa) and  $0.72\text{--}24.04 \text{ g}\cdot\text{kg}^{-1}$  (Cnh). The fraction of Cnh dominated in samples 3 and 1, whereas the fraction of Cha dominated in sample 2 (Tab. 1). In samples 3 and 1, the fraction of Cnh was about 68% and 60%, the fraction of Cfa was about 17% and 29%, and the fraction of Cha was about 16% and 11% of the total organic carbon. In the middle sample, the fraction of humic substances accounted for about 43% (Cha), 36% (Cfa) and 21% (Cnh) of the total organic carbon (Tab. 1).

**Table 1**  
Carbon content in the fractions of organic matter in the bottom sediments

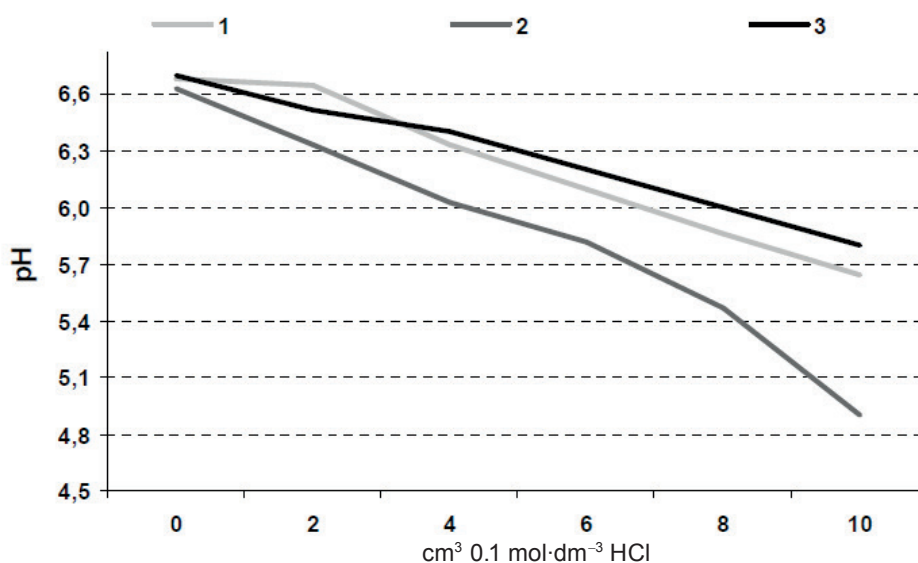
Bottom sediment	C organic	C extracted	C humic acid (Cha)	C fulvic acid (Cfa)	Cha/Cfa	C non-hydrolyzing (Cnh)
	[g·kg d.m.]					[g·kg d.m.]
Inlet – 1	8.98 ±0.32	3.57 ±0.26	0.96 ±0.08	2.61 ±0.34	0.37 ±0.08	5.42 ±0.58
Middle – 2	3.36 ±0.02	2.65 ±0.17	1.43 ±0.17	1.21 ±0.004	1.18 ±0.14	0.72 ±0.17
Outlet – 3	35.38 ±2.04	11.35 ±0.02	5.50 ±0.51	5.85 ±0.507	0.95 ±0.17	24.04 ±2.04

The ratio of Cha/Cfa is used as an indicator of humus quality (Hou et al. 2014). The ratio of Cha/Cfa ranged between 0.37 (sample 1) – 1.18 (sample 2). Generally, the higher value of Cha/Cfa indicated a relatively good humus quality (Hu et al. 2014).

Buffer is the sediment's ability to counteract changes of its pH under the influence of acids or bases (Baran et. al 2011a, Szarek-Gwiazda 2014). An influence of a decreasing pH on the release of heavy metals from the sediment was found in dam reservoirs (López et al. 2010). The data presented in Figure 2 show that the Rybnik bottom sediment has high buffering properties, especially in samples 3 and 1. A decrease in the pH value in the sediment samples was between 7–13% (sample 3),

0.5–16% (sample 1) and 5–26% (sample 2). In the studies of Szarek-Gwiazda (2014) and Baran et al. (2011a), high buffer capacities in the sediments of some Carpathian dam reservoirs were also found. However, the buffer capacity of these reservoir sediments was mainly associated with clay, carbonate and calcium content.

Our previous studies have shown that bottom sediments from the Rybnik reservoir are characterized by a greater sand fraction than clay and silt (Baran et al. 2016). Therefore, we suppose that organic matter is likely to be an important factor influencing the buffer capacity of sediments. It should be mentioned that the sorption capabilities of humus compounds are several times higher than the sorption capacity of mineral compounds.



**Fig. 2.** Buffer capacity of the bottom sediments: 1 – inlet, 2 – middle, 3 – outlet

This property makes it possible for humus substances to react with different pollutants, creating permanent bonds (Oleszczuk 2007a, 2007b, Klimowicz-Pawlas 2009, Maliszewska-Kordybach et al. 2010). Moreover, we found that a higher organic matter content tends to increase the buffering capacity of sediments (Fig. 2).

The total content of heavy metals in the bottom sediment is presented in Table 2. The highest total content of Zn and Ni was found in sample 1 (inlet); Cu, Cd in sample 2 (middle); and Cr, Pb in sample 3 (outlet). The content of heavy metals in the bottom sediment formed the following order Zn > Cu > Cr > Pb > Ni > Cd (inlet); Zn > Cu > Pb ≈ Cr > Ni > Cd (middle); Zn > Cr > Pb > Cu > Ni > Cd (outlet), and concerning the coefficient of variation (CV%), respectively, Cu (90%) > Cr (40%) > Pb (28%) > Cd (18%) > Ni (10%) > Zn (6%).

**Table 2**  
Content of heavy metals in the bottom sediment [ $\text{mg}\cdot\text{kg}^{-1}$  d.m.]

Bottom sediment	Zn	Cu	Ni	Cr	Pb	Cd
	[1 mol·dm <sup>-3</sup> HCl]					
Inlet – 1	705 ±75	79 ±1.5	27 ±0.1	40 ±0.4	52 ±3	5.7 ±0.1
Middle – 2	680 ±31	409 ±30	25 ±1	35 ±1	55 ±1	8.7 ±0.5
Outlet – 3	569 ±21	51 ±25	14 ±3	83 ±5	90 ±10	2.0 ±0.3
Sample sediment	Pseudo-total metal content					
Inlet – 1	950 ±30	130 ±3	42 ±1	120 ±3	64 ±2	9.6 ±0.2
Middle – 2	821 ±65	527 ±58	37 ±2	61 ±5	62 ±2	12.2 ±0.5
Outlet – 3	907 ±14	84 ±3	34 ±2	162 ±9	108 ±4	3.3 ±0.7
TEC	121	32	22.7	43.3	34	0.9
PEC	459	149	48.6	111	128	4.9

Besides knowledge about the total heavy metal content in the sediment, also knowing their soluble forms is incredibly useful because of their possible mobilization from the solid phase and penetration into the environment. The content of the solubility forms of metals depended primarily on their total content in the bottom sediment. This was true for Zn, Ni, Cu, Cd, Cr and Pb of which the highest contents of solubility forms were registered in the sediments collected from sampling

point 1 (Zn, Ni), point 2 (Cu, Cd) and point 3 (Cr, Pb) (Table 2). The solubility in 1 mol·dm<sup>-3</sup> HCl, depending on the sampling place, was very high and ranged: 63–83% (Zn), 61–72% (Cu), 44–69% (Ni), 34–57% (Cr), 80–87% (Pb) and from 37% to almost 72% (Cd) in comparison to their total content (Fig. 3). The lowest average solubility in 1 mol·dm<sup>-3</sup> HCl was characterized by Cr, followed by Cd < Ni < Cu < Zn < Pb. The highest solubility of metals was found in sample 2 (middle), and the lowest in sample 1 (inlet) (Cr) and 3 (outlet) (Zn, Cu, Ni, Pb, Cd) (Fig. 3).

According to the authors, the low organic matter content and low buffer capacities of bottom sediments from sample 2 (middle) may affect the highest mobility of metals. Heavy metals in sediments may exist in different chemical forms or in terms of their ways of binding. In unpolluted sediments, metals are mainly bound to silicates and primary minerals, forming relatively immobile forms, whereas in polluted ones, metals are generally more mobile and bound to other sediment phases. In environmental studies, the determination of the different ways of binding gives more information on trace metal mobility, as well as on their availability or toxicity, in comparison with the total element content. The presented studies used acid extraction to assess the mobility of metals. The obtained results allow the determination of potential metal solubility. Metals soluble in 1 mol·dm<sup>-3</sup> HCl allow the estimation of their mobilization as a result of the acidification of the environment in which they occur. Generally, metals extracted with the use of hydrochloric acid were bound to exchangeable carbonate and Fe/Mn oxide fractions as well as to organic matter, but this does not reflect their actual hazard to organisms (Baran et al. 2011b).

The contents of PAHs in the sediment samples are given in Table 3. Generally, the highest content of individual PAHs (without CHR) and  $\Sigma_9$ PAHs was found in sample 3 near the dam; the lowest in sample middle – 2 (without FLN, BAP). From the PAHs, in all samples, FLT was represented most strongly – its percentage share in the  $\Sigma_9$ PAHs amounted to 32% (middle – 2), 29% (inlet – 1) and 27% (outlet – 3). The second in line was PYR, with the percentage share in the  $\Sigma_9$ PAHs between 20% (inlet – 1) and 24% (middle – 2) (Fig. 4).

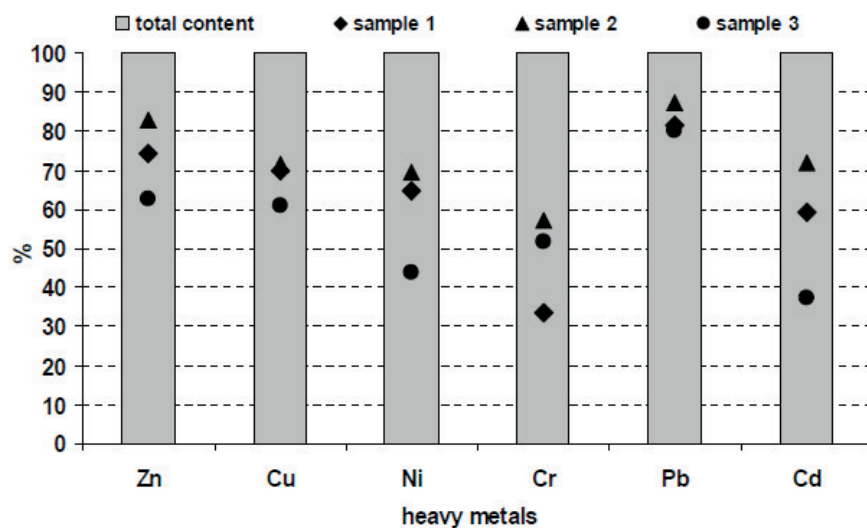


Fig. 3. Percentage share of available forms [1 mol·dm<sup>-3</sup>HCl] of heavy metals in their total contents in bottom sediment

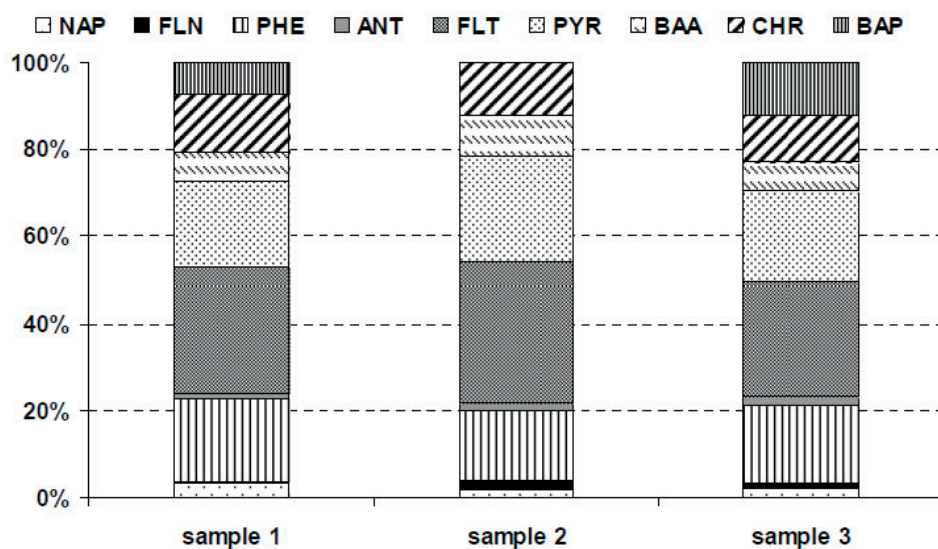


Fig. 4. Percent share of individual PAHs in their Σ<sub>9</sub>PAHs in the bottom sediment

Table 3

Content of PAHs in the bottom sediment

Bottom sediment	NAP	FLN	PHE	ANT	FLT	PYR	BAA	CHR	BAP	Σ <sub>9</sub> PAHs
	[μg·kg <sup>-1</sup> d.m.]									
Inlet - 1	880 ±39	170 ±40	5310 ±200	320 ±46	8120 ±700	5500 ±88	1820 ±173	3730 ±122	2080 ±265	27,930
Middle - 2	250 ±43	280 ±32	2090 ±102	280 ±61	4300 ±155	3230 ±88	1210 ±89	1610 ±67	n.d.	13,258
Outlet - 3	770 ±28	320 ±34	6180 ±153	640 ±83	9000 ±22	6980 ±76	2210 ±74	3710 ±243	4080 ±169	33,900

n.d. – not detected.

The lowest content of PAHs was found for ANT, FLN and NAP. The sources of PAHs can be divided into petrogenic and pyrogenic (Wang et al. 2013). Generally, petrogenic PAHs show the predominance of 2- and 3-ring PAHs, whereas pyrogenic PAHs are characterized by a high proportion of the above 4-ring PAHs. In the bottom sediment from the Rybnik reservoir, 4-ring PAHs were predominant. These compounds (FLT, PYR, BAA, CHR) constituted from 65% (inlet – 1) to 78% (middle – 2) of the  $\Sigma_9$ PAHs. Pyrogenic PAHs are characteristic for incomplete combustion of organic compounds, such as fossil fuels (heating oil, coal, grass, and wood combustion, vehicle emissions, waste tires) (Tavakoly Sany et al. 2014, Wang et al. 2016). A detailed description of PAHs content in the sediments from the Rybnik reservoir has been presented in a previous study (Baran et al. 2017).

It should be noted that the reservoir is located in one of the most industrialized areas of Poland (the Upper Silesian District), which affects the contamination of the sediments. The sources of sediment contamination in the reservoir include: treated industrial sewage emitted by the Rybnik power plant, municipal sewage, rain wastewaters, sewage from the water treatment plant, cooling tower blowdowns, and dry precipitation (Baran et al. 2016). The high content of copper in the middle part of sediments (middle – 2) is probably connected with the eluting copper from the Rybnik power plant cooling system. Bojakowska & Krasuska (2014) also observed that the copper pollution of bottom sediments of another water reservoir (Gosławskie, Licheńskie, Pątnowskie, Ślesieńskie) near Konin is caused by passing the water through the Konin-Pątnów power plant cooling system. The inflow of heated water from the power plant to the middle part of the reservoir influences the increased solubility of heavy metals in sediments and affects the transformation of PAHs and organic matter. The highest solubility of metals in 1 mol·dm<sup>-3</sup> HCl and the lowest content of C organic and PAHs was demonstrated in sample 2 (middle – 2) (Tabs. 1, 3, Fig. 2). Moreover, as demonstrated by the other research, the content of individual metals in sediments increases from the coarsest to the finest fraction (Aleksander-Kwarczak 2007). The results obtained for the Rybnik reservoir bottom sediments did not confirm

the above relationship. The typical phenomenon of granular segregation which is encountered in other reservoirs does not occur in the Rybnik reservoir. Due to disturbances in the hydrodynamic processes in the reservoir, it may be presumed that geochemical relationships between the metals and PAHs, as well as the granulometric composition of the sediments from this reservoir, are disturbed.

The content of clay fraction and the content of organic matter are the key factors of the capacity of bottom sediments to accumulate metals (Farkas et al. 2007, Shaheen & Rinklebe 2014). Organic matter has a high capacity to complex and adsorb cations due to the presence of numerous negatively charged groups. Other factors that influence the mobility of heavy metals in sediment are: salinity, presence of sulfur, carbonates, pH (Du Laing et al. 2009). Organic matter is also a very important factor in the sorption process of hydrophobic organic pollutants, such as PAHs (Simson et al. 2005, Yang et al. 2016). Klimkiewicz-Pawlas (2009) states that not only the content of organic matter, but also its qualitative composition, are important in the sorption process of PAHs. The highest sorption capability in relation to PAHs is exhibited by humins, a significantly lower capability is exhibited by humic acids, and the lowest one by fulvic acids (Klimkiewicz-Pawlas 2009). Moreover, an important role in the sorption process of PAHs is played by organic matter of organic origin, the so-called black carbon. Black carbon exhibits 10–1000 times stronger sorption properties in relation to PAHs than humic or fulvic acids (Cornelissen et al. 2006, Klimkiewicz-Pawlas 2009, Ukalska-Jurga et al. 2015). Black carbon includes substances created in the process of pyrolysis and/or incomplete combustion of biomass or fossil fuels, and these include: kerogen, charcoal, charred plant matter, soot, graphite and ash (Ukalska-Jurga et al. 2015). We assume that this fraction of organic matter may be of significant importance in the sorption of organic pollutants in the bottom sediments of the Rybnik reservoir.

To sum up, organic matter is an important parameter determining the transport and fate of inorganic and organic pollutants. Many authors have found that organic matter has a great complexing ability of heavy metals and PAHs in bottom sediments (Dahle et al. 2003, Raposo et al. 2016,

Wu et al. 2016, Baran et al. 2017). Moreover, humic substances have a variety of functional groups and can form strong complexes with metal ions, affecting their mobilization and toxicity (Hou et al. 2014). In our studies, a high organic matter content in the samples of outlet – 3 and inlet – 1 as well as high total contents of heavy metals and PAH<sub>s</sub> in those sediments demonstrate that these pollutants have a strong affinity for organic matter in the sediments collected from the Rybnik reservoir. Moreover, we think that the higher solubility of metals extracted in 1 mol·dm<sup>-3</sup> HCl from samples 2 (middle) is connected with a low content of organic matter, the low buffer capacities of sediments as well as the inflow of heated water from the power plant to the middle part of the reservoir.

## CONCLUSION

1. The highest content of C organic, C extracted, Cha, Cfa and Cnh was found in sample 3 (outlet, near the dam), while the lowest content was found in sample 2 (middle).
2. The fraction of Cnh was dominated in the sediment sample 3 (outlet, near the dam) and 1 (inlet), whereas the fraction of Cha was dominated in sample 2 (middle).
3. Rybnik bottom sediments are characterized by high buffer capacities (samples 3, 1), among others, shaped by a high content of organic matter.
4. The contents of heavy metals and PAHs in sediments demonstrate that these pollutants have a good affinity for organic matter in the Rybnik reservoir sediments.
5. However, it is necessary to perform other studies, such as the determinations of black carbon and the content of metals and PAHs in the tissues of benthic organisms and fish, because the Rybnik reservoir is used for fishing and recreational purposes on a large scale. It is also important to increase the number of bottom sediment samples for the analysis.

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