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Adsorption Properties of Ni, Cu, and Zn in Young Alkaline Lake Sediments in South Hungary (Lake Fehér, Szeged)

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Abstract

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Adsorption properties of Cu, Ni, and Zn in alkaline sediments of Lake Fehér at Szeged (Hungary) were investigated. The effects of pollution of these three chosen phytotoxic elements in sodic sediments were primarily examined. These elements are strongly adsorbed in the soils and sediments with relatively high pH values for a long time without any influence on the geochemical processes. However, the salinization (indicated by the global climate change) of soils and sediments can strongly change the original geochemical status. For the adsorption experiments, the horizons with the highest organic matter contents were selected from two profiles. The pH, electrical conductivity (EC), particle size distribution, carbonate content, quality and quantity of organic matter, and clay mineral content of the selected samples were also determined. Efficiency of the adsorption is reduced in Cu >> Zn > Ni and Cu > Ni >> Zn order based on the calculated maximum and specific adsorption values. The adsorption properties of heavy metals are dependent on the content of soil constituents. The results showed that Cu has the highest but not the same affinity to each of the sorbent materials. Ni is strongly while Zn is less bounded to the organic matter. In most cases the results showed that the most effective fixative soil constituent is carbonate, followed by clay minerals and, last, organic matter regarding to the investigated elements.

Keywords: heavy metal; organic matter; salinization

In Hungary, salinization is one of the most recent terraforming effects because of the decreasing rainfall and the basinal location. The soils and sediments of the alkaline areas exhibit extraordinary dynamically altering geochemical and hydrological properties (Pál-Molnár & Bozsó 2007; Bozsó & Pál-Molnár 2010). Basically, the cause of the salinization is the accumulation of mobile alkaline and alkaline earth metal salts in the fluctuation zone of the groundwater table (and even in the root zone) (Bohn *et al.* 2001).

The primary aim of this paper was to learn about the effects of the heavy metal pollution of Cu, Ni, and Zn. The investigated heavy metals were chosen because of their phytotoxicity (Toribio & Romanya 2006; Walter *et al.* 2006). Sodic areas have very sensitive and specialized ecosystems. These ecosystems are based on halophyte plants so a possible contamination could ruin the whole biota. The chosen elements are the most commonly investigated regarding some kind of anthropogenic pollution (Chen *et al.* 2015; Hamdoun *et al.* 2015; Li *et al.* 2015; Rodríguez Martín *et al.* 2015). With respect to the main target of the research, the study area was chosen in a typical alkaline lake system called Lake Fehér, near the city of Szeged, in south Hungary. Four profiles were studied in the area of the lake system and based on the organic matter-, clay-, and carbonate content and hydrological differences, two of them were designated for further analyses: profile A is on the area (hereafter area A) which is affected by fish-breeding activity, and profile B is on the area (hereafter area B) which is under natural conditions (Figure 1). Adsorption properties were investigated in two representative samples, derived from these two different profiles A and B, selected on the basis of the organic matter, carbonate, and clay mineral contents, pH, and electrical conductivity (EC) (Figure 1). In the selection of the two samples the primary concern was that the sample should contain the most organic matter in its profile.

Lake Fehér at Szeged is located in the southern part of the Great Hungarian Plain in the Carpathian Basin. The natural lake is one of the largest alkaline lake systems in Hungary, with a total area of 14 km^2 (Figure 1) (Pál-Molnár & Bozsó 2007; Bozsó *et al.* 2008; Rakonczai *et al.* 2008). The extent of the drainage basin is 200 km^2. Molnár (1996) described the sediments of the Lake Fehér area consisting of fluvial and aeolian drift, infusion loess, fluvial sediments, and typical loess.

MATERIAL AND METHODS

Four sampling points were designated in the area of Lake Fehér at Szeged. The exact location of the sampling points was determined based on the preliminary geological, pedological, botanical, and hydrological knowledge (Molnár 1996). After examining each subsample of the four profiles, two sections were found to be the most representative: area A (Figure 1) and area B (Figure 1). Area A is in the southern part of the eastern lake system, located 4 km from the city of Szeged. There is intensive fishbreeding in the lakes; fertilizer and fish feed are dosed in the northeastern corner of the lake. The lakes are refilled to the operating water level (1–1.5 m) in October and this level evaporates away until the end of the following spring. The lake bed dries up in summer, the ground cracks to a depth of 20 cm, and salt efflorescence usually appears. During this period, 10–20-cm high vegetation grows sparsely on the surface of the lake bed.

The area B is a saline tussock meadow formed on the alkaline sediments; in this case the aim was to find a profile which is far from anthropogenic impacts and shows the natural conditions (Figure 1). The selected meadow is a typical alkaline area. The groundwater level rises in spring, and after the snowmelt the area is for 1–2 months under water. The soil dries out gradually by mid-summer, but vegetation survives through this period. The sampling was done during the spring of 2007. The 4-m long drill cores with diameters of 10 cm were lifted by gimlet; 80–80 samples were separated from the two drill cores, and the thickness of each sample was 5 cm. The sediment from area A has the nature of solonchak soil on the basis of the macroscopic properties (IUSS Working Group WRB 2007). The drill core from area B clearly has the nature of meadow soil (IUSS Working Group WRB 2007). The main geochemical processes that are important from the adsorption viewpoint take place in the water table fluctuation zone, i.e. the upper 50 cm.

The soil particle sizes were examined using a SediGraph 5000ET (Micromeritics, Norcross, USA). The measurement was performed on a suspension of soil particles with diameters under 63 μm. The fraction with a soil particle size larger than 63 μm was separated by wet sieving.

A Delsi Oil Show Analyzer (Vinci Technologies, Nanterre, France) was used for the measurements of organic matter content. Rock-Eval pyrolysis is basically used to determine the quality, quantity, and thermal maturity of geological organic matter (Hetényi & Nyilas 2007), but with appropriate

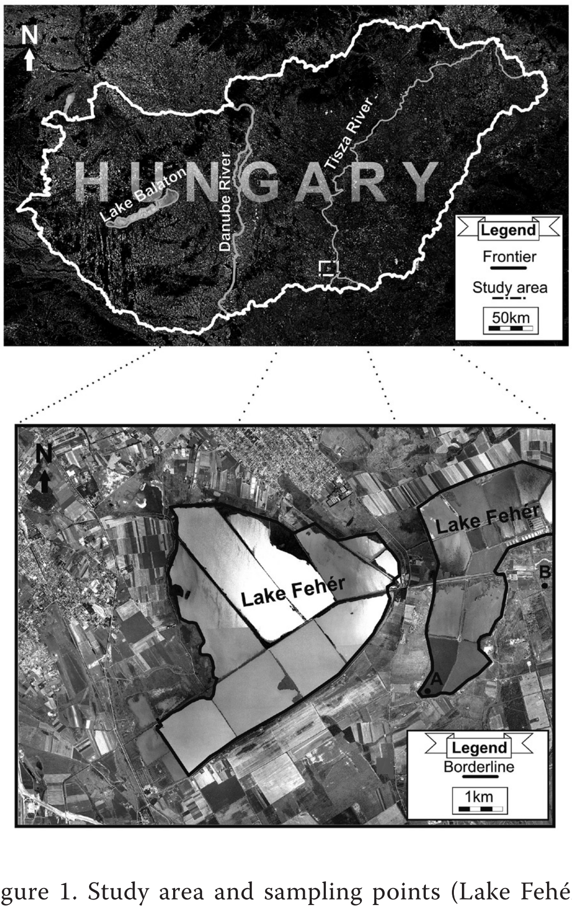

Figure 1. Study area and sampling points (Lake Fehér, Hungary)

modification it was used on the organic matter of recent soils and sediments. For the later adsorption experiments, the levels with the highest organic matter contents were designated based on the total organic carbon (TOC) values (Tables 1 and 2).

The active acidity of the soil and sediment samples was measured in 1:2.5 suspension (soil/sediment:pH 7 distilled water) in a potentiometric way according to the current Hungarian standard (MSZ 21470/2-81, 1982). A Consort C561 type pH meter (Consort, Turnhout, Belgium) and WTW SenTix 52 type combined glass electrode (WTW, Weilheim, Germany) were used for the measurements. To specify the electrical conductivity values, the Consort C561 (Consort, Turnhout, Belgium) was used in the same suspension as in the case of the active acidity.

The mineralogical investigations were done with a Rigaku Ultima IV type X-ray diffractometer (Rigaku, Tokyo, Japan). The X-ray source was a Cu-tube with 50 kV excitation voltage, 40 mA anode current, and LiF monochromator, and a scintillation counter with a 1°/min stepping speed was used. The clay mineral composition of 18 samples derived from profiles A and B was determined from the fraction under 2 μm at a 2θ angle of 1–16°. To define the swelling clay minerals, the samples were treated in ethylene glycol at 60°C for an hour, and then the diffraction peaks were measured again at a 2θ angle of 1–16°.

The carbonate content was determined by the Dean method (Dean 1974). The samples with the highest organic matter content were designated for the adsorption experiments on the basis of the Rock-Eval investigations of both drill cores (Bozsó & Pál-Molnár 2010).

Sodium content was measured with Perkin-Elmer 3110 flame atomic absorption spectrometer (Perkin-Elmer, Akron, USA) based on 7 points calibration in the range of 0.2–2 mg/l.

The upper 5 cm layers of both drill cores contained the most organic matter, so they were taken out for further investigations. After the sample preparation (mentioned above), 200 mg of the samples were suspended in a 20 ml solution of zinc-, nickel-, and copper-nitrate. The initial concentrations of the solutions were 50, 100, 200, 500, 1000, and 2000 mg/l for each sample and element and two parallel measurements were conducted in all cases. The adsorption experiments were conducted over 48 h. The pH of the suspensions was measured continuously during the experiments until it became stable. After 48 h the suspensions were centrifuged at 4000 1/min and the element content of the supernatant was measured with X-ray fluorescence spectrometry. A Rigaku Supermini wavelength dispersive X-ray fluorescence spectrometer (Rigaku, Tokyo, Japan) was used for the measurements (with a Pd X-Ray source, 50 kV

Table 1. Parameters of sediments of sampling point A, Lake Fehér (Szeged, Hungary)

Depth (cm)	pH (H_2O)	EC (mS/cm)	$CaCO_3$ (%)	Rates of particle fractions (%)			TOC (%)	Na	Cu	Ni	Zn
				sand	silt	clay		(mg/kg)			
0–5	8.48	1.89	6.4	47.8	37.3	14.9	1.24	460	19	25	118
5–10	8.44	1.89	nd	nd	nd	nd	nd	nd	17	47	140
10–15	8.47	2.04	5.6	19.8	44.5	35.7	0.93	nd	11	30	123
15–20	8.39	2.32	nd	nd	nd	nd	nd	nd	58	48	127
20–25	8.53	1.93	nd	nd	nd	nd	nd	nd	46	24	124
25–30	8.75	1.69	5.9	29.9	47.0	23.1	nd	533	18	43	134
30–35	8.82	1.82	nd	nd	nd	nd	nd	nd	23	44	134
35–40	8.76	2.01	17.3	nd	nd	nd	nd	nd	35	28	124
40–45	8.75	2.33	13.0	18.7	55.3	26.0	0.60	nd	28	28	140
45–50	8.83	2.17	20.4	12.6	58.1	29.3	nd	816	24	41	112
50–55	8.93	2.11	26.1	20.1	53.5	26.4	nd	nd	16	37	111
Mean	8.65	2.02	13.5	24.8	49.3	25.9	0.92	603	27	36	126
SD	0.19	0.20	8.1	12.6	7.8	6.9	0.32	188	14	9	10
Minimum	8.39	1.69	5.6	12.6	37.3	14.9	0.60	460	11	24	111
Maximum	8.93	2.33	26.1	47.8	58.1	35.7	1.24	816	58	48	140

SD – standard deviation; EC – electrical conductivity; TOC – total organic carbon; nd – no data excitation voltage, 4 mA anode current, and 40 s of measuring time for each element). The same instrument was used to determine the background element concentration of the sediment samples.

RESULTS AND DISCUSSION

Evaluation of selected physicochemical parameters of Lake Fehér sediments (Szeged, Hungary)

Nearly 50% of the upper 5 cm of the sediment section of area A was sand, which is a direct consequence of the flooding of the area every October (Table 1). During the flooding (3330 m^3/h) enough energy is produced to wash the sand into the part of the lake that is the closest to the Tisza River. The sediment organic matter content in sample A (TOC = 1.24%) is lower than that in sample B (TOC = 2.12%) (Table 1). In spring the sediment becomes dry and cracks; then the organic matter in the well-vented sediment is degraded more easily under oxidative conditions (Sarah 2006).

In area B, silt was the prevailing sediment: the clay and the silt fractions together represented 85.19% of the sediment (Table 2). Based on the previous data it can be stated that area B is mostly characterized by calm sedimentation and low grade energy. High-grade energy processes similar to those in area A during the flooding are missing here. The area has permanent vegetation cover during the growing season, so despite the salt nature of the meadow the organic matter content is higher than in the area of the lake (Table 2).

The pH of both areas is in the alkaline range typical of solonetz type alkaline areas (i.e. pH above 8.5) (IUSS Working Group WRB 2007). Area A has a higher pH value of the surface sediment than area B (Tables 1 and 2). The difference could be because of the denser vegetation cover in area B and the existence of root acids from the root zone of plants (Kim *et al*. 2010a, b; McGahan *et al*. 2014). The salts of the alkaline hydrolysis accumulate at the 50 cm depth of the profile, which can also be explained by the denser vegetation cover of area B compared to area A. Area A has much lower average EC (mean of the EC values: 2.02 mS/cm) in the upper 50 cm of the section than area B (mean of the EC values: 10.12 mS/cm) (Table 1). The lower value of the EC profile in area A is a consequence of salts leaching due to flooding of the area in autumn and the different land management. The electrical conductivity was 1.89 mS/cm in the surface sediment of area A and 2.85 mS/cm in area B (Tables 1 and 2). The EC shows almost a 1 mS/cm difference between the suspensions of the A 0–5 cm and B 0–5 cm samples (Tables 1 and 2).

Table 2. Parameters of sediments of sampling point B, Lake Fehér (Szeged, Hungary)

Depth (cm)	pH (H_2O)	EC (mS/cm)	$CaCO_3$ (%)	Rates of particle fractions (%)			TOC (%)	Na	Cu	Ni	Zn
				sand	silt	clay		(mg/kg)			
0–5	7.82	2.85	3.2	14.8	52.8	32.4	2.12	595	28	33	136
5–10	7.74	2.43	nd	nd	nd	nd	nd	nd	37	29	122
10–15	8.17	3.49	3.2	9.7	65.9	24.4	1.52	1099	43	31	124
15–20	8.68	3.87	nd	nd	nd	nd	nd	nd	25	47	126
20–25	8.74	6.73	nd	nd	nd	nd	nd	1610	43	39	129
25–30	8.77	9.10	nd	9.1	41.4	49.5	nd	nd	33	32	130
30–35	8.77	11.90	4.4	nd	nd	nd	nd	2572	40	41	143
35–40	8.60	17.20	nd	nd	nd	nd	nd	nd	34	29	127
40–45	8.65	15.20	5.6	8.2	45.0	46.8	0.70	3292	18	19	130
45–50	8.62	18.30	5.8	8.2	45.9	45.9	nd	nd	31	32	128
50–55	8.58	20.30	6.4	11.8	50.3	37.9	nd	3999	33	45	133
Mean	8.47	10.12	4.8	10.3	50.2	39.5	1.45	2195	33	34	130
SD	0.38	6.76	1.4	2.6	8.7	9.8	0.71	1204	8	8	6
Minimum	7.74	2.43	3.2	8.2	41.4	24.4	0.70	595	18	19	122
Maximum	8.77	20.30	6.4	14.8	65.9	49.5	2.12	3999	43	47	143

SD – standard deviation; EC – electrical conductivity; TOC – total organic carbon; nd – no data

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Table 3. Relative rates of clay minerals (%) in sediments of sampling points A and B, Lake Fehér (Szeged, Hungary)

Depth (cm)	A			B		
	smectite	kaolinite-chlorite	illite	smectite	kaolinite-chlorite	illite
0–5	11.8	15.6	11.2	12.4	13.5	13.0
40–45	11.2	11.8	11.2	19.2	21.9	10.1
55–60	10.9	9.4	9.3	15.4	17.5	16.6
65–70	10.9	11.3	10.6	2.5	6.6	5.0
95–100	6.3	8.0	8.1	3.8	4.0	5.0
Mean	10.2	11.2	10.1	10.7	12.7	9.9
SD	2.2	2.9	1.4	7.3	7.4	5.1
Minimum	6.3	8.0	8.1	2.5	4.0	5.0
Maximum	11.8	15.6	11.2	19.2	21.9	16.6

SD – standard deviation

The relative ratio of the clay minerals is equally low in the surface sediment of both areas but is lower in area A (Table 3). The reason for this is that the colloidal clay minerals change their dispersion in the section and can leach into deeper levels, adjusting to the water supply (Table 3). The clay minerals fall under the colloidal size range is verified by the fact that the distribution of the clay minerals is slightly different from the distribution of the clay fraction (Bozsó & Pál-Molnár 2010). Twice as much $CaCO_3$ is found in the surface sediment of area A than of area B (Tables 1 and 2). This phenomenon is due to the dense vegetation cover of area B, as the carbonate is decomposed by the organic acids of the roots and leached to the deeper levels of the soil (Bohn *et al.* 2001).

The background concentration of the investigated elements was relatively low according to MAC in sediment samples (MAC 2012).

Adsorption of heavy metals on the samples of relatively high organic matter content

Adsorption of copper. In the case of sample A, Cu adsorption isotherm is L-shaped, which indicates a strong affinity (Table 4 and Figure 2) (Hinz 2000). At a higher concentration range the adsorption isotherm continues to rise, which may be related to the surface precipitation, because the high concentration of heavy metals may cause this process (Sastre *et al.* 2006). Cu shows the highest affinities to the soils and sediments among the investigated elements, and it is very immobile because of the large electronegativity. Cu is strongly fixed by organic matter and clay minerals (Wahla & Kirkham 2008). The maximum adsorption is 25 685 mg/kg, which is the largest value among the measured elements (Table 4). The specific adsorption is 25%, which is also larger than that of Ni and Zn (Table 4).

The adsorption isotherm is of L type (Table 4 and Figure 2) (Hinz 2001; Sastre *et al.* 2006). Despite the greater organic matter content of sample B 0–5 cm, the maximum adsorption (14 409 mg/kg) and the specific adsorption (13%) are both low compared to sample A 0–5 cm (Table 4). The reason for the weaker adsorption is the lower pH and the higher EC value of sample B 0–5 cm.

Adsorption of nickel. The amount of adsorbed Ni on the individual samples, the concentration of the equilibrium solution, and the values of the maximum

Table 4. Amount of adsorbed elements on individual samples (*q*), values of maximum (*Q*) and specific adsorption (depth 0–5 cm)

Sample	*q* (mmol/kg)	*Q* (mg/kg)	Specific adsorption (%)	Type of isoterm
Cu				
A	403	25 684	25	L
B	226	14 409	13	L
Ni				
A	233	13 702	12	L
B	233	13 693	11	L
Zn				
A	236	15 486	15	S
B	49	3 263	7	L

L – strong adsorption affinity; S – weak adsorption affinity

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and the specific adsorption are shown in Table 4. The adsorption isotherms are presented in Figure 2.

The TOC of sample A 0–5 cm is a typical value for salt-affected sediments (Peinemann *et al.* 2005) but is high among the local conditions (Table 1). The initial pH measured in the surface sediment was 8.48. Ni is adsorbed primarily on the organic matter of the soil at pH = 8 (Gupta & Bhattacharyya 2006). The isotherm is of L type (Hinz 2001). The maximum adsorption (13703 mg/kg) and the specific adsorption (12%) are both low (Table 4).

Sample B 0–5 cm contains slightly more organic matter (2.12% TOC) than sample A 0–5 cm (Tables 1 and 2). The isotherm is L-shaped (Hinz 2000) and in the case of sample B, the maximum adsorption of Ni is 13 693 mg/kg, which is the same value as for sample A 0–5 cm. The specific adsorption of sample B 0–5 cm is 11%, which is slightly lower than in the case of sample A 0–5 cm, even though the latter contains less organic matter.

Adsorption of zinc. In the case of sample A, the adsorption isotherm of Zn is of L type (Table 4 and Figure 2) (Hinz 2001). Based on the method and results from Hinz (2001), Sipos (2004) identified in forest soils that the reduction in the specific adsorption is only determined by the pH. In the case of Zn, the reduction of the specific adsorption starts at pH = 6–7 (Sipos 2004). In the sample A, the specific adsorption of Zn is 15% and the maximum adsorption is 15 486 mg/kg (Table 4).

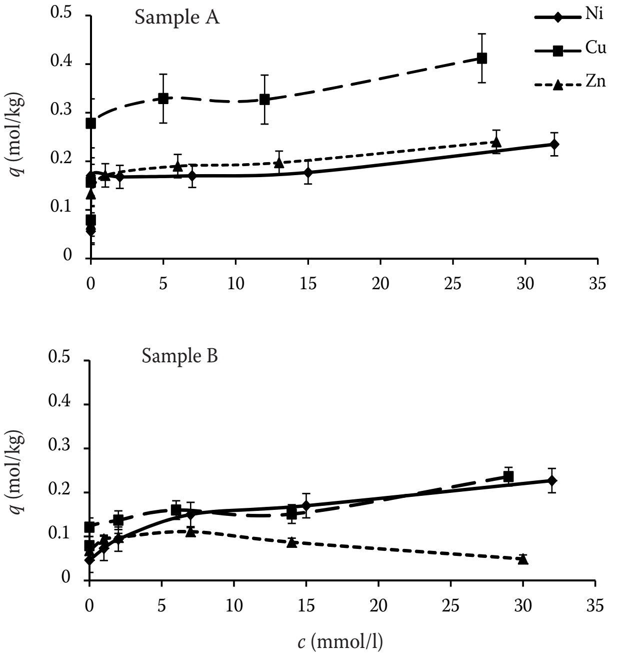

Figure 2. Adsorption isotherms of samples A and B; c – concentration of equilibrum solution, q – amount of adsorbed elements on samples

The Zn adsorption isotherm of sample B is also L-shaped (Figure 2). At high concentrations (1000 and 2000 mg/l) the adsorption curve turns to lower concentration while the pH is constant during the experiment (pH=~5). This phenomenon is probably caused by the increasing sodium concentration (which was originally adsorbed on the solid surface) in the solution and because of that the mono-element Zn adsorption turns to competitive with the sodium. Further reason of the reduced adsorption may be the low affinity and the high concentration of Zn because of its weak electronegativity. The reduced adsorption and low pH are the reasons why Zn has the lowest specific adsorption (7%) and the lowest maximum adsorption (3263 mg/kg) of the analyzed elements (Table 4).

The element sequences

Based on the results, it can be stated that the most important factor in the adsorption is the hydrolysis capacity of the heavy metals. The sediments of the two distinct areas have different geochemical properties (Tables 1 and 2). The secondary important factors are the features and amounts of adsorbents, as well as the pH values of the initial suspensions. Sipos (2004) identified that the reduction of the specific adsorption starts for different elements at different pH values: 5–6.5 for Cu, 6–7 for Zn, and 6.5–7.5 for Ni. These pH values are the reason why Cu and Zn are sorbed better under these alkaline conditions than Ni.

The element sequence of sample A 0–5 cm is Cu >> Zn > Ni. Sample B 0–5 cm has a slightly different element sequence: Cu > Ni >> Zn. Based on these element sequences, Cu has the highest adsorption affinity to the sediment samples. It was determined that Cu is adsorbed well in less alkaline conditions, but the adsorption affinity of Ni and Zn is reduced in such conditions. Sample A 0–5 cm has by 7.8% higher pH, 66.7% lower EC, and 58.5% lower TOC content (Table 1). The clay mineral content is almost the same in both samples (Table 3). In these conditions, Cu shows the highest affinity and Ni the lowest in the case of sample A 0–5 cm (Table 4). Despite the higher organic matter content, sample B 0–5 cm has lower specific and maximum adsorption values and this sample immobilized the fewest Zn.

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CONCLUSION

Based on the maximum and specific adsorption values, two different element sequences (Cu >> Zn > Ni and Cu > Ni >> Zn) were specified. From the results of the experiments it can be stated that the adsorption of heavy metals on organic matter is less effective at a lower pH. Cu shows the strongest adsorption on the basis of the experiments, primarily because of its high electronegativity. The two samples adsorbed the same amount of Ni, which means that this element has stronger affinity to the organic matter. This is supported by the fact that the amount of adsorbed Ni is similar to the quantity of Cu in sample B 0–5 cm. Based on the examined samples, in most of the cases lower adsorption affinity is shown in samples which have higher organic matter content and the adsorption affinity is moderately strong to the carbonate content. The most effective sorbent is the carbonate, followed by the clay minerals, and the least effective is the organic matter.

The adsorption of investigated elements usually has lower efficiency in the sediment if the pH is less alkaline but the proportion variance of the sorbents can change it under the same pH conditions. This paper pointed out that out of the investigated samples, one (compared to the others) with lower organic matter content, similar clay mineral content, higher carbonate content, and higher pH can immobilize amounts of Cu and Zn many times higher and a similar amount of Ni.

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