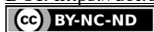


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Original scientific paper

COMPARISON OF EXTRACTION AGENTS FOR METAL DETERMINATION IN SEDIMENTS FROM ARTIFICIAL LAKES AND RIVERS IN SERBIA

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The purpose of this paper was to investigate the effectiveness of three different extraction agents for the extraction of 25 elements from sediment samples collected from 4 artificial lakes and 12 rivers in Serbia (33 samples in total). The extraction efficiency of the agents was evaluated by its ability to extract the highest quantity of the elements. For that purpose, three acids (1M HCl, 2M HNO₃ and 0.43M CH₃COOH) have been used. Inductively coupled plasma atomic emission spectrometry (ICP-OES) was used for quantitative determination of following elements: Al, As, B, Ba, Be, Bi, Ca, Cd, Cr, Co, Cu, Fe, Hg, K, Li, Mg, Mn, Ni, Na, Pb, Sb, Se, Sr, V, and Zn. The extraction with 1M HCl has shown the best results for the majority of investigated elements (especially Sr, Mn and Ca). Antimony (Sb) was detected only after extraction with 0.43M CH₃COOH, while selenium (Se) could not be detected when 2M HNO₃ was applied as extraction agents. The present study could be very useful for choosing a suitable method for specific elements and also can be helpful in the evaluation of the contaminants in freshwater sediments in Serbia. This might contribute to environmental risk assessment of the present elements.

Keywords: extraction agents, metals, river and lake sediments

INTRODUCTION

The consequences of growing population and fast industrialization have brought a lot of pollutants into the environment. Among the others, heavy metals represent one of the most abundant. Their presence indicates different natural or anthropogenic sources. Main natural sources in water systems are soil erosion, rocks dissolution, volcano eruptions, while the anthropogenic sources belong to different industries, untreated waste waters, implementation of non-sustainable agricultural practices, etc. (1, 2, 3, 4). Most of the present metal elements are microelements which are beneficiary for human health, but their increased concentration might have toxic effects on biota (5, 6). The metals released into the water are deposited in sediments, which represents complex dynamic, chemical and biological system that contains matter from erosion and sedimentation process (7). The metal accumulation process in soil and sediments represents a potential risk for human health because of metals easily transport to food chains (8, 9). The toxicity and the pathway of toxic elements in sediments depend on the chemical species of the metal compound as well as its surroundings. Thus it is very important to determine the available metal species rather than its total content. The metal fraction bonding with the sediment will influence metal availability and potential risk connected (10).

Trace elements that are intensively investigated in the last decades are Cu, Zn, Fe, Mn, Mo, B, Co, Ni, Pb, Cd, Cr, As, Hg and Se. Metals necessary for higher plants and

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mammals are Fe, Mn, Zn, Cu, Mo, Co, and Ni as well as non-metals B and Se. Regarding environment pollution of soil, water, and food contamination, elements that are important to monitoring are Cu, Cd, Zn and Pb (11). The concentrations of biologically available metals have great influence in many researches since it represents a toxic fraction that has the biggest influence on biota. Bioavailability of mentioned elements depends on stability and reactivity of element species present in different sediment fractions (12, 13). The availability of specified elements can be examined by extraction process which can give answers on bond type and its strength in various sediment phases and the rest of ions, carbonates, reduction and oxidation species (14). Extraction process can be one step or multi sequential extraction, and it can provide information about distribution and availability of elements in geochemical fractions. Different extraction agents have purpose to simulate natural processes, such as acidification or oxidation. Obtained metal concentrations represent bioavailable fraction which is strongly correlated with their leaching potential from soil and sediments (15).

For the purpose of investigation of the metal availability from sediments present in artificial lakes and rivers in Serbia, three different extraction agents were used: 1M HCl, 2M HNO₃ and 0.43M CH₃COOH (Carlo Erba).

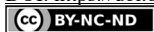
MATERIALS AND METHODS

Study region and the sampling procedure

Investigated samples were taken from different locations of the watercourse in Serbia (represented in Figure 1). The set of 34 samples of river and lake sediments were taken from: Danube (Black Sea watershed), Sava (Danube watershed), Tisa (Danube watershed), West Morava (Great Morava watershed), Nišava (South Morava watershed), Tamiš (Danube watershed), Topčiderska River (Sava watershed), Kolubara (Sava watershed), Pek (Danube watershed) and Toplica (South Morava watershed).



Figure 1. Network of surface water stations - hydrological stations and basins (<http://www.hidmet.gov.rs/>)



The sediment samples analyzed in this manuscript were obtained from the Republic Hydrometeorological Service of Serbia. The sediment sampling was performed in accordance with their standard methodology. A total of 34 samples was taken from 12 rivers and 4 lakes. In the case of larger rivers, sampling was conducted at several locations. For sampling representative sediment sample, Van Veen grab sampler was used. After sampling, sediments were transported to the laboratory. For the purpose of macro and microelements determination, the fraction with an average diameter less than 63 μm of the bottom sediment was used (“grab”– the sample).

Experimental procedure

The procedures applied for element extraction in this manuscript are described in the following manuscripts: Snape et al., 2004 (16), Šmejkalová et al. 2003 (17) and Quevauviller et al. 1996 (18). For each extraction procedure, a solid-liquid ratio is defined as well as the duration of extraction aiming for the best efficiency of element extraction. Fraction A was prepared mixing 4 g of sample with 40 ml 2M HNO_3 , for 1 hour on rotation mixer (Heidolph) at room temperature (17). Fraction B was prepared mixing 2 g of sample with 40 ml 1M HCl , for 4 hours on rotation mixer at room temperature (16). Fraction C was prepared mixing 1g of a sample with 40 ml of 0.43M CH_3COOH , for 16 hours on rotation mixer at room temperature (18). After required contact times, all the samples (A, B and C) were centrifuged for 15 minutes (Hettich Zentrifugen – Universal 320), supernatants were filtered, and filtrates filled with 1M HNO_3 , up to 100 ml.

Chemical analyses

The quantitative analysis of the studied elements (Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, Hg, K, Li, Mg, Mn, Na, Ni, Pb, Sb, Se, Sr, V, Zn) were done with atomic emission spectrometry with inductively coupled plasma (Thermo Scientific).

RESULTS AND DISCUSSION

After extraction with HCl , HNO_3 , and CH_3COOH , concentration of elements were recalculated in the mass of dry sample, by the following equation:

$$C = \frac{C^*V}{m} \quad [1]$$

where the marks represent:

- C – concentration of the element in dry sample ($\mu\text{g/g}$),
- C^* – concentration of element obtained with ICP-analytical method ($\mu\text{g/ml}$),
- V – sample volume (ml),
- m – dry sample mass (g)

Extractions were done for all stated elements, but in Table 1, 2 and 3 are presented elements which have prescribed maximal permissible concentration (MPC). The MPC of heavy metals is defined in the Ordinance on the permissible quantities of hazardous and harmful substances in soil and irrigation water and methods of their testing, prescribed by the Serbian law. Article 2 of this document defines MPC of cadmium, lead, mercury, arsenic, chromium, nickel, fluorine, copper, zinc, and boron.



The ratio of obtained element concentration after extraction with HCl and HNO₃

The bigger amount of following elements was extracted with HCl: Al, B, Ba, Bi, Cd, Co, Cu, Fe, Mg, Mn, Na, Ni and Pb in all samples (Table 1). After extraction with HCl and HNO₃, most of the samples were under limits of detection, which implied that some ratios couldn't be calculated. Extracted sample of Sb was under the limits of detection for both acids used.

Table 1. Ratio of obtained concentration elements after extraction with HCl and HNO₃, in sediments sample from Serbia

	As	B	Cd	Cr	Cu	Hg	Ni	Pb	Zn
Barje-1	1.10	2.47	1.13	1.06	1.08	0.31	1.37	1.14	1.24
Barje-2	0.94	/	1.28	1.05	1.10	/	1.13	1.15	1.18
Begej	0.94	1.29	0.99	0.63	1.00	0.49	1.07	1.04	1.07
Bojnik	1.12	/	1.18	0.83	1.09	0.49	1.19	1.21	1.15
Bovan-1	1.06	1.33	1.12	1.08	1.08	/	1.20	1.09	1.18
Bovan-2	1.37	3.99	0.48	0.15	0.86	/	0.23	0.61	0.50
Dunav-1	1.25	1.95	1.11	0.95	1.11	/	1.18	1.16	1.16
Dunav-2	1.13	2.93	1.06	0.93	1.07	0.18	1.11	1.12	1.11
Dunav-3	1.09	/	1.02	0.92	1.03	1.54	1.28	1.09	1.18
Dunav-4	1.08	1.98	1.04	0.72	1.01	2.18	1.09	1.10	1.11
Dunav-5	1.02	1.83	1.04	0.85	1.01	1.10	1.10	1.10	1.11
Kolubara	1.02	1.82	1.04	0.94	1.06	1.55	1.33	1.13	1.28
Nišava-1	1.11	1.35	1.07	0.69	1.13	/	1.11	1.13	1.11
Nišava-2	0.99	1.99	1.04	0.78	1.08	0.08	1.15	1.12	1.20
Pek	1.02	1.77	1.03	1.05	1.02	1.01	1.22	1.13	1.20
Sava-1	1.14	1.77	1.02	1.04	1.06	1.62	1.40	1.10	1.21
Sava-2	1.03	2.42	1.06	0.91	1.07	0.65	1.28	1.14	1.19
Sava-3	1.30	2.03	1.16	1.34	1.20	/	1.58	1.21	1.26
Sava-4	1.01	0.75	2.69	8.41	1.47	/	7.86	2.14	2.90
Tamiš	1.15	/	1.14	1.27	1.13	1.04	1.62	1.15	1.37
Tisa-1	0.99	2.27	1.02	0.90	1.02	0.44	1.26	1.10	1.19
Tisa-2	0.90	1.52	1.02	0.91	1.01	1.43	1.28	1.10	1.21
Tisa-3	1.08	1.79	1.05	1.08	1.12	1.81	1.50	1.13	1.30
Tisa-4	1.03	4.05	1.05	0.93	1.07	3.82	1.28	1.12	1.22
Tisa-5	0.98	5.39	1.01	1.01	1.09	2.09	1.40	1.10	1.24
Tisa-6	0.99	2.56	0.99	0.89	1.03	1.01	1.25	1.08	1.17
Tisa-7	0.99	4.41	1.00	0.92	1.04	0.86	1.32	1.09	1.19
Tisa-8	1.01	6.00	1.02	0.89	1.07	5.43	1.20	1.10	1.18
Topčiderska	1.26	1.74	1.03	1.06	1.08	8.59	1.36	1.10	1.30
Toplica	1.08	1.35	1.09	0.99	1.07	0.54	1.30	1.13	1.17
V. Han	1.04	1.28	1.04	0.99	1.05	0.28	1.28	1.11	1.14
Vrutci	0.84	1.98	1.11	0.88	1.03	0.36	1.21	1.08	1.21
Z. Morava	1.12	1.34	1.02	1.24	1.12	0.63	1.56	1.15	1.24
Average	1.06	2.32	1.10	1.16	1.08	1.52	1.45	1.13	1.23

/ - values were not obtained because they were below the limit of detection using one or both acids



The ratio of obtained elements concentration after extraction with HCl and CH₃COOH

The bigger content of elements was released by extraction with HCl than by extraction with CH₃COOH for the following elements: Al, As, B, Ba, Bi, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Ni, K, Na, Se, Sr, V, Zn, and Pb. The observed ratio HCl/HNO₃ much bigger than 1 for Al, As, Pb, V, Fe, Mn (Table 2). For Sb, Hg, Se, and Bi, the ratio could not be calculated because the contents of these elements were under limit detection.

Table 2. The ratio of obtained concentration elements after extraction with HCl and CH₃COOH, in sediment sample from Serbia

	As	B	Cd	Cr	Cu	Hg	Ni	Pb	Zn
Barje-1	7.96	/	2.79	13.30	3.18	/	3.21	13.06	3.08
Barje-2	29.35	/	/	17.61	9.43	/	4.02	24.72	8.73
Begej	57.31	/	3.18	7.25	/	/	1.72	12.05	2.18
Bojnik	8.54	/	2.77	10.22	3.87	/	2.64	12.56	3.40
Bovan-1	5.11	/	2.62	5.51	6.32	/	2.50	6.94	2.16
Bovan-2	5.08	/	2.45	5.83	6.88	/	2.39	7.41	1.96
Dunav-1	/	/	1.84	7.56	3.26	/	2.59	9.38	2.30
Dunav-2	8.28	/	1.81	7.92	3.44	/	3.69	7.77	1.85
Dunav-3	7.08	/	1.29	8.01	4.27	/	4.75	4.83	2.60
Dunav-4	26.14	/	1.68	8.65	5.12	/	2.94	14.58	2.43
Dunav-5	11.55	/	1.84	6.95	4.10	/	3.23	8.98	2.25
Kolubara	/	/	1.33	12.28	5.71	/	7.02	9.90	3.84
Nišava-1	3.08	/	1.93	14.66	7.81	/	2.30	12.76	1.47
Nišava-2	6.86	/	1.45	5.45	9.89	/	2.81	9.64	2.42
Pek	10.20	/	1.10	9.11	1.82	/	1.95	7.77	1.36
Sava-1	8.03	/	1.25	9.24	4.90	/	4.68	8.47	2.32
Sava-2	6.12	/	1.17	7.77	3.86	/	4.41	6.64	1.90
Sava-3	5.78	/	1.50	8.61	5.02	/	4.47	8.48	2.29
Sava-4	8.07	/	1.75	9.99	5.77	/	4.89	10.40	2.84
Tamiš	/	/	2.25	16.59	3.66	/	4.94	18.82	4.13
Tisa-1	16.82	/	1.33	6.53	2.74	/	3.20	6.98	2.15
Tisa-2	13.85	/	1.31	7.90	2.86	/	3.42	8.94	2.32
Tisa-3	/	/	1.44	8.86	3.81	/	3.80	10.47	2.71
Tisa-4	/	/	1.57	5.66	3.76	/	4.33	16.73	3.86
Tisa-5	7.12	/	1.13	6.22	2.49	/	3.53	5.36	1.77
Tisa-6	8.61	/	1.08	6.23	2.17	/	3.25	4.73	1.64
Tisa-7	5.38	/	1.11	5.86	2.73	/	3.36	5.73	1.77
Tisa-8	10.53	/	1.12	7.36	2.95	/	3.53	8.09	1.74
Topčiderska r.	7.08	/	1.42	11.36	7.31	/	5.94	9.14	4.63
Toplica	4.59	13.38	2.20	8.87	4.67	/	3.03	7.75	2.02
V. Han	10.20	/	1.38	9.22	3.78	/	3.18	7.57	1.82
Vrutci	/	/	3.97	15.95	8.44	/	3.23	8.03	5.69
Z. Morava	8.51	/	1.16	8.07	3.86	/	3.82	4.45	1.62
Srednja vrednost	11.37	/	1.76	9.11	4.67	/	3.60	12.10	2.70

/ - values were not obtained because they were below the limit of detection using one or both acids



The ratio of obtained element concentration after extraction with HNO₃ and CH₃COOH

The extraction with HNO₃ gave better results for the following elements: Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Ni, K, Na, Se, Sr, V, Zn, and Pb. Better extraction with HCl is remarked for Li in some samples (Kolubara, Begej, Dunav-1). As can be seen from Table 3, extracted ratio for Al, As, B, Bi, Cr, Pb, V, Fe, Mn, and Ni is much higher with HNO₃ than for CH₃COOH. In the case of Sb extraction with both acids, the majority of the samples were below the limit detection except for three samples where the acetic acid gave better results (Kolubara, Begej, Dunav-1). At extraction Ba in Barje sample (Barje-1 and Barje-2) the obtained value was bigger with CH₃COOH extraction; this was also case with Ca (Kolubara river samples, Dunav-1, Tisa-2, 3, 4, 5, 7, 8, Sava-1, 2, Barje-1, 2, Bojnik), and Li (Kolubara, Begej, Dunav-1). In samples Bojnik and Barje-2 the concentration of B were below limit detection after applied all extraction agents in this study.

Table 3. The ratio of obtained concentration elements after extraction with HNO₃ and CH₃COOH, in sediment samples from Serbia.

	As	B	Cd	Cr	Cu	Hg	Ni	Pb	Zn
Barje-1	7.24	/	2.47	12.50	2.96	/	2.34	11.46	2.49
Barje-2	31.23	/		16.74	8.55	/	3.57	21.57	7.42
Begej	60.95	/	3.20	11.56	/	/	1.61	11.54	2.04
Bojnik	7.63	/	2.34	12.38	3.55	/	2.22	10.42	2.97
Bovan-1	4.81	/	2.34	5.08	5.83	/	2.09	6.34	1.83
Bovan-2	3.72	/	5.15	37.92	7.88	/	10.64	12.11	3.91
Dunav-1	/	/	1.66	7.96	2.93	/	2.20	8.07	2.98
Dunav-2	7.34	/	1.71	8.55	3.22	/	3.32	6.92	1.67
Dunav-3	6.49	/	1.26	8.74	4.13	/	3.71	4.41	2.20
Dunav-4	24.28	/	1.61	10.51	5.04	/	2.70	13.30	2.19
Dunav-5	11.28	/	1.76	8.16	4.08	/	2.95	8.20	2.03
Kolubara	/	/	1.28	13.05	5.37	/	5.27	8.78	3.01
Nišava-1	2.77	/	1.81	21.20	6.88	/	2.08	11.33	1.33
Nišava-2	6.90	/	1.39	6.98	9.19	/	2.45	8.58	2.02
Pek	9.98	/	1.06	8.64	1.77	/	1.59	6.86	1.14
Sava-1	7.07	/	1.22	8.89	4.61	/	3.34	7.67	1.91
Sava-2	5.92	/	1.10	8.50	3.62	/	3.45	5.83	1.60
Sava-3	4.43	/	1.29	6.43	4.17	/	2.83	7.01	1.82
Sava-4	7.98	/	0.65	1.19	3.91	/	0.62	4.86	0.98
Tamiš	/	/	1.97	3.08	3.24	/	3.05	16.32	3.02
Tisa-1	17.03	/	1.30	7.28	2.67	/	2.53	6.34	1.81
Tisa-2	15.26	/	1.29	8.69	2.83	/	2.68	8.13	1.91
Tisa-3	/	/	1.38	8.22	3.23	/	2.54	9.48	2.08
Tisa-4	/	/	1.50	6.07	3.51	/	3.38	14.48	3.16
Tisa-5	7.25	/	1.11	6.18	2.29	/	2.51	4.89	1.42
Tisa-6	9.34	/	1.09	6.97	2.12	/	2.60	4.40	1.41
Tisa-7	5.43	/	1.11	6.35	2.62	/	2.55	5.24	1.48
Tisa-8	10.43	/	1.10	8.26	2.76		2.94	7.35	1.48



Table 3. Continuation

	As	B	Cd	Cr	Cu	Hg	Ni	Pb	Zn
Topčiderska r.	5.62	/	1.39	10.74	6.75	/	4.37	8.33	3.57
Toplica	4.26	9.88	2.03	8.81	4.36	/	2.32	6.85	1.73
V. Han	9.79	/	1.33	9.30	3.59	/	2.49	6.82	1.59
Vrutci	/	/	3.56	18.12	8.19	/	2.66	81.22	4.69
Z. Morava	7.60	/	1.14	6.50	3.45	/	2.44	3.88	1.30
Srednja vrednost	11.19	/	1.71	10.29	4.35	/	2.97	10.39	2.28

/ - values were not obtained because they were below the limit of detection using one or both acids

CONCLUSIONS

Investigations presented in this paper were focused on the determination of specific elements in river and lake sediments after their extraction with three different acids: HCl, HNO₃ and CH₃COOH. The element concentrations were analyzed in a total of 34 sediment samples, taken from rivers in Serbia. Comparing the extraction efficiency, it was concluded that the most efficient extraction agent is 1M HCl. The extraction efficiency decreased in following order: 1M HCl > 2M HNO₃ > 0.43M CH₃COOH. The results of extracted metals which were compared with the maximum allowed concentrations have indicated that there are significant anthropogenic sources of sediment pollutions. They are especially significant for the following metals: Zn (Zapadna Morava and Pek); Pb (Zapadna Morava); Cu (Pek); Ni (Vrutci and Zapadna Morava) and Cd (Zapadna Morava). The sources of these pollutants are mainly consequence of ore mining, improper agricultural activities as well as release of untreated waste waters from different industries. It is important to emphasize that most of the extracted metals represent mobile fractions, which could further contaminate environment and have negative impact on human health.

The results of this study might be important not only in local but in regional water management and can be used also, for future comparisons of sediment quality. In addition, this data can be used in scientific and preventative approaches for better understanding of sources, fates, and effects of microelements in aquatic systems.

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