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Scientific Paper

CHEMICAL PROFILE OF PLOMIN BAY SEDIMENTS

Višnja OREŠČANIN[1], Karlo NAĐ[2], Anamarija BARTOLINČIĆ[3], and Vladivoj VALKOVIĆ[2]

USKNI, Laboratory for Applied Nuclear Analytics[1], Institute Ruđer Bošković[2], Belupo[3], Zagreb, Croatia

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Granulometric, chemical, and leaching properties of sediments dredged in the Plomin Bay (Northern Adriatic Sea, Croatia) were investigated in order to asses the risk of remobilisation of heavy metals into the water column. In total 65 samples from 65 sampling sites were taken from different sediment depths within the bay. Analysis of variance confirmed the homogeneity of granulometric and elemental composition of the investigated sediment throughout its volume. Granulometric analysis showed that all samples corresponded to a pelitic fraction (<0.063 mm). Bulk elemental mass fractions in the sediments were similar to literature data on relatively unpolluted areas of the Adriatic Sea. High sedimentation rate caused by constant inflow of material from the Boljunčica River drainage may be responsible for low levels of heavy metals and negligible influence of fly and bottom ash from a nearby disposal site on the chemical composition of the sediments. In contact with sea water only 0.29 mg kg^{-1} of V, 0.04 mg kg^{-1} of Cr, 0.07 mg kg^{-1} of Ni, 0.33 mg kg^{-1} of Cu, 0.67 mg kg^{-1} of Zn and 0.06 mg kg^{-1} of Pb could be remobilised from sediment material into the water column. However, these values increased three to ten times in case of leaching with organic acids.

KEY WORDS: *dredging, heavy metals, leaching, marine sediments, organic complexes, remobilisation, sea water*

The Plomin Valley, approximately 6 km long, is situated on the east coast of Istria, Croatia (North Adriatic), 1 km to the south from the town of Plomin, and 6 km to the northeast from a larger town of Labin. The first three km of the valley is dry land and the rest is below the sea level and forms the Plomin Bay. It extends in the NE-SE direction. Its outer part is surrounded by a steep slope (reaching 60° to 65° at places), while the inner part is marked by a gentle slope (6° to 7°) because of the constant fluvial deposit of the Boljunčica River from its drainage area Čepić polje. For the first 600 m the sea is only about 1 m deep. The following 800 m sink to a depth of 20 m to gradually reach the deepest point of 52 m at the mouth of the bay. Sediment gets wider from 15 m to 50 m across this longitudinal profile.

A small settlement, Plomin Luka, started to develop at the beginning of this century and by 1932 became the most important trade harbour for central Istria. In 1932, a 4550 m long channel was built to drain swamps of the Čepić polje into the Plomin Bay. This channel transported not only water, but also sediments from Lake Čepić. Constant inflow of mud into the bay required constant removal to maintain this area suitable for port activity. Due to a high cost of dredging, cleaning of the bay was stopped in 1938. Transport of the deposit was partially stopped after building the Letaj dam in 1970, that redirected the Boljunčica to flow into the Raša River, but it was too late for the Plomin Bay which was almost completely filled with mud. After the dam was built, transport of suspended matter through the channel continued, and today the estimated sedimentation rate in the bay is greater than 10 cm a year (1).

In 1970, Plomin 1 thermal power plant (TPP) was put in operation using local coal as fuel (2), which contained 10 % to 15 % of non-combustible matter. This non-combustible, mineral part is rich in heavy metals and radioactive materials and consists of two phases. The first is bottom ash, which settles to the bottom of the boiler together with the non-combustible part, and the second is fly ash, which is vented together with hot gases through electrostatic filters. A part of it passes through filters and is released into the atmosphere. Plomin 1 power plant used 200,000 t of coal a year, and produced 30,000 t of ash, depending of the type of coal. With power plant Plomin II in operation, this figure will eventually reach 70,000 t a year. Ash disposal site is situated in the vicinity of the power plant on the left bank of the Bišac, a stream which flows into the Plomin Bay. The composition of the bottom and fly ash depends on the type of coal used in combustion (3). Previously published data (4) for two months worth of daily measurements in coal and waste material (fly and bottom ash) from Plomin 1 power plant showed that Ti, V, Cr, Fe, Ni, Cu, Zn, As, Se, Pb, Sr, Y and U were several times higher in the ash than in coal. In 1999, dredging of 610,000 m^3 of mud to the depth of 6 m was authorised to restore activity to the Plomin Port. The mouth of the bay was designated for the dumping site.

Preliminary radiometric investigation of the Plomin Bay sediments (1) showed that the top three meters of the material were mostly flysch (95 %) eroded from the Boljunčica catchment area and deposited over the last 30 years. In other words, these three meters of sediments were deposited while the power plant was in operation.

Disposal of dredged material to the sea bottom is a common route for the transport of contaminants into the open sea (5, 6). Our study of the granulometric, chemical, and leaching properties of the Plomin Bay sediment was undertaken in order to assess a possible influence of the power plant on the quality of sediments as well as the risk of heavy metal remobilisation from dredged material into the water column and further penetration into the food chain.

MATERIALS AND METHODS

We took 65 samples of mud from different depths (1 m to 6 m) at different sites of the bay while dredging was in progress. Each step included removal of 1 m of sediment by a dredger and sampling of the sediment from the created surface at -1 m, -2 m, -3 m, -4 m, -5 m, and -6 m. To collect the samples we used a grab sampler and placed them in acid-washed plastic containers. Samples for chemical analysis were oven-dried at 60 °C for two days, ground, sieved, and homogenised. For granulometric analysis, approximately 300 g of wet sample was taken and composition determined by wet sieving (fraction >0.045 mm) and pipetting (fraction <0.045 mm). Elemental mass fractions were measured using energy dispersive X-ray fluorescence (EDXRF) (7). Measurements were carried out with a Mo anode and Mo secondary target in order to reduce background effect and to improve monochromaticity. A Siemens X-ray apparatus, Kristallofleks 710/710H model, was used at 35 kV and 35 mA. Irradiation time was 2000 s. X-ray spectra were collected with a Si(Li) detector (FWHM = 190 at 6.4 keV) and analysed using analysis of X-ray spectra by the iterative least squares fitting (AXIL) (8). IAEA standard reference material (SRM) Soil-7 and SL-1 (Lake Sediment) provided quality control for the measurements.

Following EDXRF analysis, ten grams of each dried sample was taken in order to prepare a composite sample of the dredged material. The material was mixed and homogenised and 1 g of the mixture was extracted by 2x10^{-2} mol L^{-1} of citric acid, ascorbic acid, ethylenediaminetetraacetic acid (EDTA), or oxalic acid for eight hours (9, 10). Sediment was also extracted with sea water (solid/liquid ratio=1:10) for 24 hours. At the end of the contact time, the solid part was separated from the leachate by filtration through a white ribbon-filter paper. The extracts were diluted to 100 mL with double distilled water divided in two sub-samples, and adjusted to pH 3 and pH 11 by the addition of concentrated HNO_3 or NH_4OH. All pH measurements were made with a Mettler Toledo digital pH meter. Following pH adjustment, 2 mL of 1 % APDC was added to each flask. After 20 minutes of complexation, the suspension was filtered through a Millipore HAWP filter (pore size 0.45 μm; diameter 25 mm) using a Millipore micro-filtration system (11). Thus prepared thin targets were air dried, protected by thin Mylar foil (2 μm), and analysed with an X-ray spectrometer. All experiments were done in triplicate.

Samples were analysed using a MiniPal4 (12) EDXFR spectrometer (PANalytical, Almelo, Nederland). X-rays were generated from an Rh tube (maximum power: 9 W; window: 75 μm Be; maximum high voltage: 30 kV; maximum current: 300 μA; cooling medium: air). In order to reduce background, filters were used between the source and the sample. Measurement time was 300 s per sample, and measurements were carried out in the air. Characteristic X-ray radiation from the sample was detected with a Si drift detector [surface: 5 mm^2; FWHM for 5.9 keV ^{55}Fe: 145 eV; window: 13 µm Be; cooling: thermo-electrical (peltier)]. The incident and emerging angles were 45°. Spectral data were analysed using MiniPal/MiniMate software, version 3.0. (PANalytical, Almelo, the Netherlands). In order to reduce errors due to non-homogeneous samples, a spinner system constantly rotated samples during measurement. Instrument settings were automatically checked every hour using Al-Cu alloy reference material. AlKα and CuKα-X-ray lines from the plate were used for corrections to the energy scale, where CuKα X-ray line was used for automatic gain control.

A calibration model for qualitative and quantitative analysis was created on the basis of measurements of standard solutions (Merck) in the concentration range from 10 µg L^{-1} to 200 µg L^{-1}, prepared and measured in the same way as unknown samples.

Statistical analysis was done using Statistica 6.0 for Windows package. The difference between sampling depths was assessed using one-way ANOVA.

RESULTS AND DISCUSSION

Granulometric and chemical composition

Table 1 shows the basic statistical parameters computed for granulometric fractions determined in sediment samples taken from six layers during the dredging of the Plomin Bay sediments. Basic statistical parameters computed for the elemental mass fractions measured in the same samples are shown in Table 2.

According to the granulometric analysis, fine-grained fraction dominated in the sediment with Silt and clay constituting more than 70 % of the total sediment composition. There was no significant difference in the granulometric composition between the six layers.

Chromium levels in the dredged sediments ranged from 25 mg kg^{-1} to 62 mg kg^{-1}, of copper from 20 mg kg^{-1} to 44 mg kg^{-1}, nickel from 20 mg kg^{-1} to 40 mg kg^{-1}, zinc from 69 mg kg^{-1} to 103 mg kg^{-1}, and lead from 7 mg kg^{-1} to 27 mg kg^{-1} (Table 2). In 1994, Giani et al. (5) reported chromium levels from 92 mg kg^{-1} to 171 mg kg^{-1} and lead levels from 15 mg kg^{-1} to 58 mg kg^{-1} for dredged Northern

Table 1 *Granulometric analysis of 24 samples taken from the Plomin Bay sediment by depth*

Granulometric parameter	Statistical parameter / %	Sampling depth / m					
		1	2	3	4	5	6
Coarse-grained sand	Mean	0.9	0.8	0.4	0.4	0.6	0.9
	SD	5.9	0.7	0.3	0.3	0.7	1.6
	Minimum	0.1	0.1	0.1	0.1	0.1	0.1
	Maximum	1.4	1.5	0.6	0.9	1.1	5.0
	Median	0.9	0.9	0.5	0.3	0.6	0.1
Fine-grained sand	Mean	27.1	29.2	25.1	23.8	29.3	21.2
	SD	7.8	10.8	4.2	9.0	9.2	7.4
	Minimum	16.4	18.1	20.3	11.6	22.8	10.9
	Maximum	37.1	39.3	27.7	36.1	35.8	34.1
	Median	27.9	29.8	27.4	23.9	29.3	21.6
Silt	Mean	44.1	42.5	43.5	44.4	42.6	44.8
	SD	7.7	6.4	2.5	4.0	5.2	3.4
	Minimum	39.2	36.0	41.6	38.1	39.0	37.7
	Maximum	48.9	48.1	46.3	48.6	46.3	48.6
	Median	44.9	43.0	42.7	44.8	42.6	45.6
Clay	Mean	27.8	27.4	30.9	31.4	27.5	33.1
	SD	3.2	5.2	2.2	5.4	4.7	4.8
	Minimum	20.3	22.9	29.1	25.0	24.1	27.1
	Maximum	32.4	33.8	33.3	40.2	30.8	43.4
	Median	28.4	26.5	30.4	31.3	27.5	32.7

Table 2 *Basic statistical parameters of elemental mass fractions measured in the Plomin Bay sediment samples taken from different depths*

Element	Statistical parameter / mg kg^{-1}dry mass	Sampling depth / m					
		1	2	3	4	5	6
Ti	Mean	3481	3664	3377	3421	3140	3557
	SD	221	577	463	421	264	519
	Minimum	3248	3010	2970	2849	2970	2926
	Maximum	3779	4451	4543	4388	3721	4992
	Median	3448	3705	3188	3329	3090	3478
V	Mean	106	88	96	97	98	100
	SD	11	6	14	15	9	18
	Minimum	100	82	79	81	87	71
	Maximum	123	97	120	140	110	151
	Median	101	89	96	96	96	99
Cr	Mean	41	36	42	42	36	40
	SD	5	7	8	9	11	10
	Minimum	37	27	25	30	26	25
	Maximum	47	43	52	56	50	62
	Median	39	39	45	41	30	38
Mn	Mean	741	799	750	790	755	736
	SD	46	84	49	67	42	86
	Minimum	693	697	674	650	683	520
	Maximum	785	895	831	880	819	882
	Median	742	784	761	799	748	738
Ni	Mean	32	37	34	35	35	34
	SD	3	2	4	4	1	5
	Minimum	28	34	24	27	34	20
	Maximum	33	38	40	39	36	38
	Median	33	38	35	36	35	35
Cu	Mean	30	33	33	35	34	32
	SD	1	5	3	5	5	6
	Minimum	28	29	27	28	26	20
	Maximum	31	40	37	43	41	44
	Median	31	32	33	35	34	31
Zn	Mean	79	87	84	86	85	83
	SD	2	6	6	7	4	8
	Minimum	77	79	75	73	79	69
	Maximum	82	96	94	96	90	103
	Median	79	87	85	86	87	82
Pb	Mean	13	10	11	11	14	10
	SD	2	2	2	2	8	3
	Minimum	11	8	9	8	8	7
	Maximum	15	13	17	15	27	16
	Median	13	9	11	10	10	9
U	Mean	4	3	4	4	4	5
	SD	2	3	3	3	3	3
	Minimum	3	0	0	0	0	0
	Maximum	6	5	6	7	7	8
	Median	3	5	5	5	5	5

Adriatic sediments. The following values were reported by Valković and Bogdanović for the Punat Bay sediments in 1996: ND to 229 mg kg^{-1} for Cr, 26 mg kg^{-1} to 4018 mg kg^{-1} for Cu, ND to 89 mg kg^{-1} for Ni, 35 mg kg^{-1} to 269 mg kg^{-1} for Pb, and 38 mg kg^{-1} to 781 mg kg^{-1} for Zn. Chromium levels reported by Paul and Meischner in 1976 (13) ranged from 18 mg kg^{-1} to 78 mg kg^{-1} and for Cu from 20 mg kg^{-1} to 25 mg kg^{-1}. In 1978, Frignani et al. (14) reported Cr to range from 24 mg kg^{-1} to 66 mg kg^{-1} and from 15 mg kg^{-1} to 35 mg kg^{-1}. In 1988, Bernardi et al. (15) found 31 mg kg^{-1} to 41 mg kg^{-1} of Cr, 35 mg kg^{-1} to 46 mg kg^{-1} of Pb, and 131 mg kg^{-1} to 194 mg kg^{-1} of Zn. In 1989, Valković and Moschini (16) reported 25 mg kg^{-1} to 93 mg kg^{-1} for both Cr and Cu, 16 to 129 mg kg^{-1} for Pb, and 31 mg kg^{-1} to 163 mg kg^{-1} for Zn in the Punat Bay sediments. For the same locality in 1990, Legović et al. (17, 18) reported Cr levels to range from 26 mg kg^{-1} to 186 mg kg^{-1} and Zn from 25 mg kg^{-1} to 186 mg kg^{-1}. Zonta et al. (19) reported the following ranges in Venice lagoon sediments in 1994: 16 mg kg^{-1} to 45 mg kg^{-1} for Cr, 18 mg kg^{-1} to 76 mg kg^{-1} for Cu, 22 mg kg^{-1} to 36 mg kg^{-1} for Ni, 37 mg kg^{-1} to 81 mg kg^{-1} for Pb, and 111 mg kg^{-1} to 343 mg kg^{-1} for Zn.

All these data suggest that heavy metal mass fractions measured in the Plomin Bay sediments kept within in the ranges reported for other relatively unpolluted localities of the Northern Adriatic Sea (5, 7, 13-19).

One-way ANOVA for either original or log-transformed variables (elemental mass fractions) showed no significant difference in the composition between the six layers for any of the variable tested. The difference in chemical composition between sediment samples from the first three layers (deposited at the time of power plant activity) and those deposited before this period was statistically insignificant. This means that the dredged material is homogenous in elemental composition throughout its volume.

Leaching tests

The dredged material was deposited at the sea bottom of the bay. The influence of sea water can mobilise certain elements to a significant extent (9). It is known that various naturally occurring organic complexes can highly increase the leaching potential of sea/rain water (9, 10). Therefore, we applied leaching procedures with the most common, naturally occurring organic acids such as EDTA, oxalic acid, and citric acid on a composite sample of dredged material to assess whether there was a risk of remobilisation of heavy metals from the mud into the water column.

Table 3 shows that sea water extraction capability ranged from 0.1 % for Cr to 1 % for Cu, which is low. The opposite was found for oxalic acid which was the most effective among five extraction agents. As Cu, Zn, and Pb have high affinity to organic complexes, these were the most susceptible to leaching by organic acids.

Under the influence of sea water, only 0.29 mg kg^{-1} of V, 0.04 mg kg^{-1} of Cr, 0.07 mg kg^{-1} of Ni, 0.33 mg kg^{-1} of Cu, 0.67 mg kg^{-1} of Zn, and 0.06 mg kg^{-1} of Pb could be remobilised from the sediment material into the water column. Under the worst possible leaching conditions (high load of organic matter, low pH value) leading to the maximum release of elements from sediment particles into the sea water, 0.78 mg kg^{-1}, 0.40 mg kg^{-1}, 0.44 mg kg^{-1}, 1.68 mg kg^{-1}, 4.45 mg kg^{-1} and 0.46 mg kg^{-1} of the V, Cr, Ni, Cu, Zn and Pb, respectively could be extracted from dredged material.

Table 3 *Bulk elemental mass fractions measured in composite sample of Plomin Bay sediment and the mass fractions and percentages of elements extracted from Plomin Bay sediments by various extraction agents*

Element	Mass fraction / mg kg^{-1} dry mass						Percentage of elements extracted from sedimens with				
	Total	Extracted with					Sea water	Ascorbic acid	EDTA	Citric acid	Oxalic acid
		Sea water	Ascorbic acid	EDTA	Citric acid	Oxalic acid					
V	98	0.29	0.39	0.10	0.49	0.78	0.3	0.4	0.1	0.5	0.8
Cr	40	0.04	0.12	0.24	0.28	0.40	0.1	0.3	0.6	0.7	1.0
Ni	34	0.07	0.14	0.17	0.37	0.44	0.2	0.4	0.5	1.1	1.3
Cu	33	0.33	0.79	1.68	1.42	1.29	1.0	2.4	5.1	4.3	3.9
Zn	84	0.67	1.93	2.86	3.44	4.45	0.8	2.3	3.4	4.1	5.3
Pb	11	0.06	0.15	0.30	0.46	0.36	0.5	1.4	2.7	4.2	3.3

EDTA – ethylenediaminetetraacetic acid

CONCLUSION

Heavy metal mass fractions in sediment samples from the investigated area exhibited values lower or equal to those reported for relatively unpolluted regions of the Adriatic Sea (5, 13-19). Granulometric and chemical composition of the dredged material was highly homogeneous, exhibiting no significant difference between layers. Sediments deposited in the Plomin Bay before and during power plant activity had almost identical chemical composition.

Uniform elemental composition of samples from different layers suggests that was one prevailing source of material during the whole sedimentation period.

Contaminants introduced into the Bay by man-made sources like the power plant were highly diluted by the constant inflow of large volumes of sediment material from the Boljunčica River drainage area, which resulted in relatively low mass fractions of heavy metals.

Sea water showed low extraction capability, ranging from 0.04 mg g^{-1} to 0.67 mg g^{-1} of heavy metals. These values changed significantly in the presence of organic complexes.

Our results suggest that deposited material in the Plomin Bay poses low environmental health risk, which is in agreement with earlier findings of neither cytotoxic nor mutagenic effects of this material on bacterial test systems (20).

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Sažetak

KEMIJSKI PROFIL SEDIMENATA PLOMINSKOG ZALJEVA

Granulometrijska i kemijska svojstva te mogućnost otpuštanja teških metala ispitivani su u sedimentima Plominskog zaljeva (Sjeverni Jadran, Hrvatska) u svrhu utvrđivanja rizika od remobilizacije teških metala iz sedimenta u stupac vode. Uzeto je 65 uzoraka s različitih točaka i dubina unutar zaljeva. Analizom varijance potvrđena je granulometrijska i kemijska homogenost cijelog volumena sedimenta, što upućuje na jedan prevladavajući izvor tijekom cijeloga sedimentacijskog razdoblja. Granulometrijskom analizom je utvrđeno da u svim uzorcima prevladava sitnozrnata frakcija (<0,063 mm). Koncentracije elemenata u ukupnim uzorcima sedimenata slične su literaturnim vrijednostima objavljenim za relativno onečišćena područja Jadranskog mora. Velika brzina sedimentacije uzrokovana konstantnim donosom materijala iz slijevnog područja Boljunčice vjerojatan je uzrok niskih koncentracija teških metala i slabo vidljivog utjecaja odlagališta šljake i pepela na sastav sedimenata. U kontaktu s morskom vodom moguća je remobilizacija samo 0,29 mg kg^{-1} V, 0,04 mg kg^{-1} Cr, 0,07 mg kg^{-1} Ni, 0,33 mg kg^{-1} Cu, 0,67 mg kg^{-1} Zn i 0,06 mg kg^{-1} Pb iz sedimenta u stupac morske vode. Ipak ove vrijednosti su tri do deset puta povećane u slučaju izluživanja s pomoću organskih kiselina.

KLJUČNE RIJEČI: *izluživanje, morska voda, morski sedimenti, organski kompleksi, remobilizacija, teški metali, uklanjanje*

CORRESPONDING AUTHOR:

Višnja Oreščanin
USKNI (Udruga servisera i korisnika nuklearne instrumentacije)
Laboratory for Applied Nuclear Analytics
A. Jakšića 30, 10000 Zagreb, Croatia
E-mail: *vorescan@gmail.com*