Heavy Metals in the Sediment of the Islands of Lastovo and Mljet and the City of Dubrovnik

Teški metali u sedimentu otoka Lastova i Mljeta i grada Dubrovnika

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Abstract

The content of heavy metals (Fe, Mn, Zn, Cu, Cr, Hg, As, Pb and Cd) in the sediment of the marine aquatoriumof the islands of Lastovo and Mljet, and the city of Dubrovnik was examined in order to determine their impact on the environment. Divers collected the samples in a layer of 10 cm and 5 cm at the planned locations. The sample preparation was performed in the laboratory in an autoclave with acids in teflon vessels at elevated temperature. Determination of the concentration of elements was made on an atomic absorption spectrophotometer (AAS), by the method of flame, cold and hot vapors, and nickel vessels. The marine aquatorium of the islands of Lastovo and Mljet are not polluted with ecotoxic elements, while an increased concentration of mercury, arsenic and lead has been found in the marine aquatoriumof the city of Dubrovnik at several locations.

KEY WORDS

heavy metals
sediment
Dubrovnik
atomic absorption spectroscopy (AAS)
ecotoxicity
pollution
sampling

Sažetak

Ispitan je sadržaj teških metala (Fe, Mn, Zn, Cu, Cr, Hg, As, Pb i Cd) u sedimentu morskog akvatorija otoka Lastova i Mljeta te grada Dubrovnika kako bi se utvrdio njihov utjecaj na okoliš. Ronioci su uzorke prikupljali u sloju od 10 cm i 5 cm na planiranim lokacijama. Priprema uzorka obavljena je u laboratoriju u autoklavu s kiselinama u teflonskim posudama na povišenoj temperaturi. Određivanje koncentracije elemenata izvršeno je na atomskom apsorpcijskom spektrofotometru (AAS), metodom plamena, hladnih i vrućih para i posuda od nikla. Morski akvatorij otoka Lastova i Mljeta nije onečišćen ekotoksičnim elementima, dok je u morskom akvatoriju grada Dubrovnika na nekoliko lokacija utvrđena povećana koncentracija žive, arsena i olova.

KLJUČNE RIJEČI

teški metali
sediment
Dubrovnik
atomska apsorpcijska spektroskopija (AAS)
ekotoksičnost
onečišćenje
uzorkovanje

1. INTRODUCTION / *Uvod*

Marine pollution implies man's direct or indirect input of materials or energy into the marine environment that causes harmful consequences such as reduction and destruction of certain species of marine flora and fauna, endangering human health, disrupting maritime activities including fishing, deteriorating seawater quality, and reducing interest in housing and recreation.

The distribution and impact of heavy metals on the environment has been investigated since 1970, and has intensified in particular after a significant incident of the impact of mercury on marine organisms, and indirectly on people in Minamata Bay in 1995 [1]. It is generally know that changes in the redox regime, the action of free sulfides and organic substances affect the behavior of metals and alloy [2]. These changes occur most in dead zones of rapid (proliferative) reproduction when the seabed is poor in oxygen [3].

Sediment consists of inorganic and organic substances with complex physical, chemical, and biological characteristics [4]. These accumulations at the bottom of the sea can collect a large number of elements and thus become a reservoir with much higher concentrations of metals than those contained in the seawater column. The sediment thus becomes a favorable matrix for monitoring metal pollution [5]. However, the sediment is not only the reservoir but also possible source of emission of these contaminants into the seawater column due to desorption, remobilization processes, redox reactions, and decomposition of sorbed substances [4, 5]. In this way, the sediment contamination by metals can directly affect the quality of seawater and presents a long-term source of contamination of marine organisms used in human nutrition [5, 6]. Unlike most organic pollutants, heavy metals are not biodegradable, and once introduced into the environment they become part of it forever.

The term "ecotoxic" has been also used for the metals that are toxic to living organisms. Previously, the term heavy metals was used more often, which is not entirely true, because some metals with lower atomic mass are toxic (e.g. beryllium), while others (as iron) are only toxic at very high concentrations [7].

Research has often shown that the Mediterranean Sea possesses a high toxicological risk as the result of its hydrographic characteristics and the significant effects of human activity. The TheAdriatic Sea is a relatively shallow and semi-enclosed sea, with large Italian rivers, Po, Adige and Brenta, flowing into it. The rivers from the Croatian area have much smaller impact on the

sea pollution. This impact of rivers, together with pollutants from industrial and urban sources, which are located along the coast, make this environment particularly interesting from the point of view of metal contamination.

The aim of this paper is to investigate the content of heavy metals, most of which are ecotoxic, in the sediment of the coastal belt of the islands of Mljet and Lastovo and the city of Dubrovnik. Based on the obtained results, we believe that it is possible to determine the extent to which anthropological sources of pollution (traffic, construction and maintenance of ships, industrial waste, etc.) polluted the sediment and the extentof their impact on the environment.

2. MATERIALS AND METHODS / *Materijali i metode*

2.1. Sample preparation / *Priprema uzorka*

The sediment sampling was performed by divers by taking 10 cm samples at 11 locations in the seawaters of the island of Lastovo (June 2015), 11 in the aquatorium of the island of Mljet (November 2015) and five in the aquatorium of the city of Dubrovnik (April 2017). In the aquatoriumof Dubrovnik, the samples were taken at a depth of 5 cm too. All locations and results are shown in Figures 1, 2 and 3, respectively, in Tables 1., 2. and 3. In the area of Mljet, two locations within the islands of Blatina-Sobra and Blatina-Prožura were also examined, because they mainly contain seawater.

At each collection site, three sediment samples were taken in a range of three meters and the samples were stored in plastic boxes and transferred to the laboratory. They were dried for several days at room temperature and grounded in a mortar. Then the purified and homogenized samples were heated at 60 ° C for 60-90 minutes. The dried samples were sieved through a 250 μm sieve, homogenized again and deposited in a desiccator. A measured amount of 0.2 g was then poured into a 35 mL teflon flask into which 6 mL of nitric acid and 2 mL of

Figure 1 Lastovo
Slika 1. Lastovo

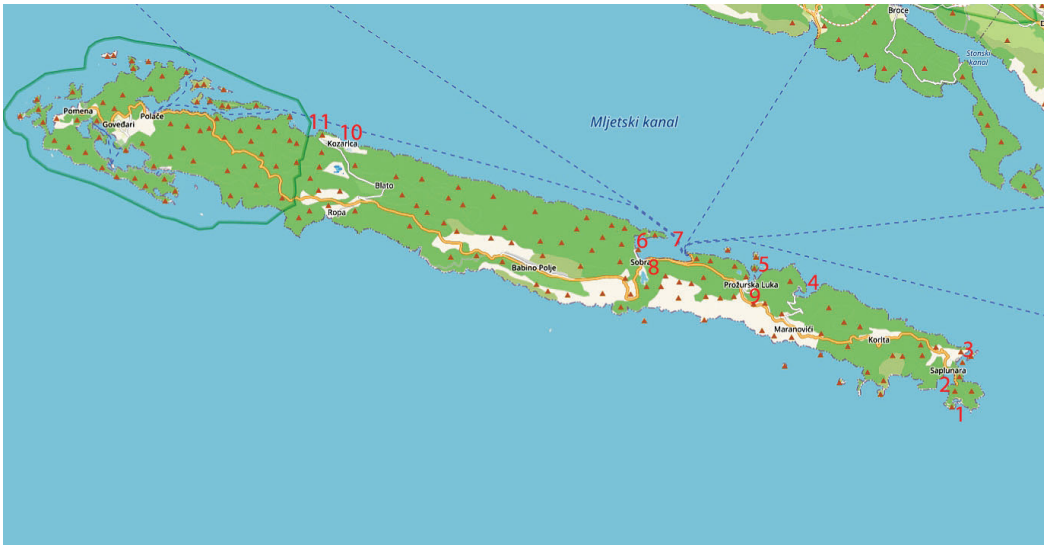

Figure 2 Mljet
Slika 2. Mljet

Figure 3 Dubrovnik
Slika 3. Dubrovnik

hydrofluoric acid and 2 mL of hydrogen peroxide were added [8, 9, 10]. When the vessel is placed in a chrome-plated steel autoclave, it is kept at a temperature of 180°C for 20 minutes. After cooling, the content was transferred to a volumetric flask and refilled up to 50 mL with water.

The preparation of samples for mercury analysis is done separately as follows according to the study of M. Sadique [11]: 0.3-0.4 g ofthe sediment is heated (digestion) with sulfuric and nitric acid in a ratio (2: 1) for three hours at 100-110°C and then 30 minutes at 100°C with 5% potassium permanganate. The excess potassium permanganate is then reduced with 5% hydroxylamine hydrochloride. The sample is then supplemented to 50 mL and transferred to a plastic container.

2.2. Measurement of the samples / *Mjerenje uzoraka*

Measurements of Fe, Mn, Zn, Cu and Cr contents were performed by the standard flame method on an atomic absorption spectrophotometer (AAS), Perkin Elmer, model 2380.

The concentrations of mercury and arsenic were measured by the method of cold and hot vapors, respectively, on AAS with a special device (MHS 10), where sodium borohydride is used as a reducing agent [9,10].

The content of Pb and Cd in the samples was determined on AAS by the nickel-plated micromethod, so that the solvent (water and acids) was evaporated before placing the sample in the flame.

3. RESULTS AND DISCUSSION / *Rezultati i rasprava*

The results of all measurements of heavy metal content in the sediment are shown in Table 1. for the island of Lastovo (L), in Table 2. for the island of Mljet (M) and in Table 3. for the aquatorium of the city of Dubrovnik (GD).

Due to the insight into the basic geological structure of the sediment and possible changes in it caused by human factor, in addition to ecotoxic elements, some non-toxic elements (toxic only in very high concentrations) were examined (Fe and Mn).

Table 1 The content of heavy metals in the sediment of island Lastovo (L)
Tablica 1. Sadržaj teških metala u sedimentu otoka Lastova (L)

No	Location	Layer cm	Fe %	Mn mg kg^{-1}	Zn mg kg^{-1}	Cu mg kg^{-1}	Cr mg kg^{-1}	Hg mg kg^{-1}	As mg kg^{-1}	Pb mg kg^{-1}	Cd mg kg^{-1}
1	Sv.Jura	10	0.495	66.3	41.9	10.0	84	<0.1	4.0	<4.0	<0.5
2	Kremina	10	0.157	62.0	27.8	13.2	94	<0.1	0.5	<4.0	<0.5
3	Ubli	10	0.389	112.0	36.7	12.1	63	<0.1	4.7	<4.0	<0.5
4	Zaglav	10	0.391	53.6	35.4	9.0	129	<0.1	2.5	<4.0	<0.5
5	Skrivena luka	10	0.693	134.6	36.6	11.0	77	<0.1	6.6	<4.0	<0.5
6	Barije	10	0.396	153.6	24.4	11.4	55	<0.1	5.9	<4.0	<0.5
7	Lučica	10	0.591	119.7	83.0	22.6	46	<0.1	6.0	<4.0	<0.5
8	Sv. Mihovil	10	0.360	111.4	17.2	7.0	43	<0.1	5.0	6.2	<0.5
9	Zaklopatica	10	0.341	62.4	16.6	7.9	126	<0.1	2.5	<4.0	<0.5
10	Kručica	10	0.588	116.3	30.6	12.3	182	<0.1	3.5	<4.0	<0.5
11	Malo Logo	10	0.512	105.6	21.6	50.3	132	<0.1	3.4	6.2	<0.5
	Average value		*0.446*	*99.8*	*33.8*	*15.2*	*93.7*	*<0.1*	*4.0*	*<4.4*	*<0.5*

Table 2 The content of heavy metals in the sediment of island Mljet (M)
Tablica 2. Sadržaj teških metala u sedimentu otoka Mljeta (M)

No	Location	Layer cm	Fe %	Mn mg kg^{-1}	Zn mg kg^{-1}	Cu mg kg^{-1}	Cr mg kg^{-1}	Hg mg kg^{-1}	As mg kg^{-1}	Pb mg kg^{-1}	Cd mg kg^{-1}
1	Lemoni	10	1.666	292	49.1	12.5	99	<0.1	9.1	6.8	<0.5
2	Salpunara	10	1.388	370	53.5	6.3	164	<0.1	4.7	<4	<0.5
3	Podškoji	10	1.154	300	45.8	26.5	108	<0.1	6.0	<4	<0.5
4	Okuklje	10	1.073	193	62.4	14.7	89	<0.1	7.5	<4	<0.5
5	Prožura	10	1.708	245	67.3	17.8	233	<0.1	5.9	<4	<0.5
6	Sobra-uzgoj	10	1.104	257	59.4	19.4	125	<0.1	7.3	<4	<0.5
7	Sobra-uvala	10	0.529	188	36.6	24.4	74	<0.1	5.9	5.2	<0.5
8	Blatina-Sobra	10	0.481	155	40.0	28.0	83	<0.1	1.7	<4	<0.5
9	Blatina-Prožura	10	3.665	625	72.7	6.3	74	<0.1	5.5	<4	<0.5
10	Slatina-Kozarica	10	3.332	235	41.1	39.8	199	<0.1	4.5	<4	<0.5
11	Kozarica-uvala	10	1.660	307	69.2	26.3	125	<0.1	20	12.5	<0.5
	Average value		*1.674*	*288*	*54.3*	*20.2*	*125*	*<0.1*	*7.1*	*<5.1*	*<0.5*

Table 3 The content of heavy metals in the sediment of Dubrovnik (GD)
Tablica 3. Sadržaj teških metala u sedimentu Dubrovnika (GD)

No	Location	Layer cm	Fe %	Mn mg kg^{-1}	Zn mg kg^{-1}	Cu mg kg^{-1}	Cr mg kg^{-1}	Hg mg kg^{-1}	As mg kg^{-1}	Pb mg kg^{-1}	Cd mg kg^{-1}
1	Izvor Omble	10	1.800	250	32.8	33.7	88	1.25	7.50	<4	<0.5
2a	Marina	10	1.750	198	58.5	26.2	109	2.50	10.00	12.5	<0.5
2b	Marina	5	1.542	197	87.1	35.1	135	2.40	9.25	6.3	<0.5
3a	JK Orsan	10	2.042	124	137	121	83	1.00	10.50	62.5	<0.5
3b	JK Orsan	5	1.845	127	107	109	90	0.50	7.75	52.5	<0.5
4a	Uvala	10	0.863	111	28.4	18.7	91	<0.1	7.50	<4	<0.5
4b	Uvala	5	0.953	117	31.7	25.0	86	<0.1	6.50	<4	<0.5
5a	Petka	10	1.329	76.1	48.3	56.3	43	<0.1	<0.2	<4	<0.5
5b	Petka	5	1.590	71.7	48.8	62.5	21	<0.1	<0.2	<4	<0.5
	Average value		*1.524*	*141.3*	*64.4*	*54.2*	*82.9*	*<0.9*	*<6.6*	*<17.2*	*<0.5*

IRON (Fe): Measured iron concentrations in the Adriatic Sea coastal sediment range from 0.008% to 5.55%, with a mean value of 0.3% [12]. Our measurements showed significantly higher values of iron content in the southern part of the Adriatic Sea (average value for the island of Mljet is 1.614%, and for the city of Dubrovnik is 1.524%), than the concentration in the waters of the island of Lastovo (average value is 0.444%). This is completely in agreement with the results of other authors [14] according to which that is the consequence of different content of the source rocks.

MANGANESE (Mn): The measured manganese concentrations in the Adriatic Sea coastal sediment range from 14.9 mg kg^{-1} to 896.5 mg kg^{-1}, with a mean of 67.1 mg kg^{-1} [12]. Our measurements showed a similar trend as for iron concentrations, with probbable influence of human activity in the seawaters of the city of Dubrovnik, because the manganese content is constantly decreasing from the source of the river Ombla to the location of Petka. The mean values for Lastovo, Mljet and the city of Dubrovnik are 99.9 mg kg^{-1}, 378.8mg kg^{-1} and 141.4mg kg^{-1}.

3.1. Ecotoxic elements / *Ekotoksični elementi*

In present study, we used guidelines on marine sediment quality for ecotoxic elements [7], namely ERL (effect range low) and ERM (effect rage medium).

	Zn	Cu	Cr	Hg	As	Pb	Cd
ERL-mg kg^{-1}	150	34	81	0.15	8.2	46.7	1.2
ERM-mg kg^{-1}	410	270	370	0.71	70	218	9.6

ZINC (Zn): Measured concentrations of zinc in the sediment of the Adriatic Sea coastal belt range from 3.9 mg kg^{-1} to 16.800 mg kg^{-1} [11]. The concentration distribution deviates significantly from the mean value (35.8 mg kg^{-1}) with clearly visible anthropogenic activity. Significantly increased concentrations are found in marinas and in ship service zones.

In the examined sediment samples from the seawaters of the islands of Lastovo and Mljet and the city of Dubrovnik, zinc concentrations are significantly below the ERL value. It is only at the JK Orsan (GD) site that it approaches this value. This is a consequence of the work of the sailboat service and so far has no negative impact on living organisms.

COPPER (Cu): The measured concentrations of copper in the sediment of the Adriatic Sea coastal belt range from 1.8 mg kg^{-1} to 31.100 mg kg^{-1} [12]. The concentration distribution deviates significantly from the mean value (17.1 mg kg^{-1}) with clearly visible anthropogenic activity.

In the examined sediment samples from the seawaters of the islands of Lastovo and Mljet and the city of Dubrovnik, copper concentration is in most cases below the ERL value. However, at several locations, copper concentrations are above

the ERL values, namely: Malo Logo (L); Slatina-Kozarica (M); and Petka (GD). At the location of JK Orsan (GD), this value is significantly higher and is indisputably the consequence of the activity of the sailboat service. This observed concentration can have a negative effect on living organisms.

CHROME (Cr): Measured concentrations of chromium in the sediment of the Adriatic Sea coastal range from 6.1 mg kg^{-1} to 418 mg kg^{-1} [12]. The authors found increased concentrations in the area of Elafiti, Ston Bay and the southern part of the island of Mljet and stablished that this was due to different geological bases.

In our tests, the mean values of chromium content in the sediment of the islands of Lastovo, the island of Mljet and the city of Dubrovnik are successively 93.6 mg kg^{-1}, 124.8 mg kg^{-1} and 67.8 mg kg^{-1}. The concentrations are around or above the ERL value and are the highest for the island of Mljet, which agrees with the literature and results causedby different geological bases. These concentrations of chromium do not have a detrimental effect on living organisms.

MERCURY (Hg): In the Adriatic Sea, the presence of mercury in the sediment was discovered at small concentrations in the ports of Rijeka [5], Šibenik [7] and Split [13], and only at Sokolan location in Kaštela Bay the value was as much as 150 times higher of ERM value [14].

Our tests have shown that in the sediment of the seawaters of the islands of Lastovo and Mljet there is no presence of mercury, i.e. concentrations are below the sensitivity of the method of 0.1 mg kg^{-1}. In the seawaters of the city of Dubrovnik, we found the presence of mercury at three locations: at the source of the river Ombla 1.25 mg kg^{-1}, Marina 2.5 mg kg_{-1} and JK Orsan 1.0 mg kg^{-1} for the layer of 10 cm and 0.5 mg kg^{-1} for the layer of 5 cm. These concentrations are above the ERM value and are the consequence of human activities (the source of the river Ombla is affected by the leachate from Grabovica landfill and the other two locations the service activates for ships and sailboats have been in course). These concentrations of mercury can have a very negative effect on living organisms, as well as on the people who are the last in the food chain.

ARSENE (As): Measured concentrations of arsenic in the sediment of the Adriatic Sea coastal arearange from 1.1 mg kg^{-1} to 938 mg kg^{-1} [12]. The distribution of concentrations deviates significantly from the mean value (7.3 mg kg^{-1}), with a clearly visible anthropogenic contribution, especially in the marina zone. According to research in the seawaters of the cities of Rijeka [5] and Šibenik [7], the arsenic content was below the ERL value, while in the coastal area of Split [14] the average concentration was 36.8 mgkg^{-1}, which is significantly higher.

In our tests, arsenic concentrations at all locations off the coast of the island of Lastovo were below the ERL value. In the marine waters of the island of Mljet at several locations the concentration was slightly above the ERL value and it stands out only at the location Kozarica-bay where it is 20.0 mg kg^{-1}. In the seawaters of the city of Dubrovnik, arsenic concentrations are above the ERL values at all locations except Petka location. All concentrations up to an ERL value of 8.2 mg kg^{-1} can be considered a natural source of arsenic and those slightly higher are caused by human activities. These concentrations of arsenic cannot significantly affect living organisms.

LEAD (Pb): The measured concentrations of lead in the coastal sediment of the Adriatic Sea range from 2.1 mg kg^{-1} to 2.307 mg kg^{-1} [12]. The concentration distribution deviates significantly from the mean value (9.9 mg kg^{-1}) with clearly visible anthropogenic contribution. According to the research in the seawaters of Split-Dalmatian County [13], the lead content is around the ERL value. This was confirmed in later work for the coast of Split [14].

In our tests, the lead content in the seawaters of the islands of Lastovo and Mljet was at the limit of sensitivity of the method (4 mg kg^{-1}) or slightly above which is significantly lower than the ERL value. Similar concentrations were obtained in the sewaters of the city of Dubrovnik, except for the location of JK Orsan, where the concentration was slightly above the ERL value. These concentrations are considered harmless to living organisms.

CADMIUM (Cd): According to tests for Split-Dalmatian County [12] as well as according to recent studies in the seawaters of the city of Split, the cadmium content is significantly below the ERL value [14].

In our tests, we did not establish the presence of cadmium in the sediment of the seawaters of the islands of Lastovo and Mljet and the coast of Dubrovnik. Therefore, cadmium can only be present atthe concentrations below 0.5 mg kg^{-1} which is the sensitivity limit of the method. This is in accordance with the results for the National Park Mljet [15] where determined maximum concentration is 0.30 mg kg^{-1}. These are quantities from natural sources and they are significantly lower than the ERL value, and cannot negatively affect living organisms.

4. CONCLUSION / *Zaključak*

In the sediment of the seawaters of the city of Dubrovnik, the total metal content was measured in a layer of 10 cm and 5 cm. In most cases, small differences have been observed, confirming that the content of heavy metals has not changed significantly in recent years. However, a decrease in the concentration of the most toxic metals mercury and lead in the upper sediment layer is visible, which indicated that over time there is less pollution with these elements.

Furthermore seawater of the islands of Lastovo and Mljet are not contaminated with tested ecotoxic metals, while in the area of Dubrovnik at three locations increased values of ecotoxic metals with Cr, As and Pb above ERL values and Hg above ERM limits were found. The contamination factor [16,17] is successively 1.1, 0.4, 0.6 and 6.1, i.e. it is increased only for mercury content. Therefore, at the locations of the rivers Ombla, Marina and JK Orsan there is a great possibility of environmental impact, so all activities that may adversely affect human health should be limited.

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