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Trace metal contamination of sediments along the open coast of Montenegro

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ABSTRACT

The coastal region of Montenegro faces significant environmental challenges due to ongoing pollution from various sources. This study investigates trace metal contamination in sediment samples collected from the open coast of Montenegro in 2019 and 2020. Elevated contents of some elements, found at the location S4, but also at S1, were predominantly attributed to the pollution from nearby holiday cottages and restaurants. Multivariate statistical analysis, including cluster analysis and canonical component analysis, was used to identify trace metal grouping and variations in trace metal pollution in sediments from different locations. The contamination factor values indicated predominantly low to moderate contamination along the Montenegrin coast, with notable exceptions such as S4, showing very high contamination for Cr in 2020, and significant contamination for Cr in 2019, as well as Ni and Cd in 2020. Pollution load index values showed the presence of pollution at locations S3 and S4, while contamination severity index values revealed varying degrees of pollution severity across different locations, with S4 exhibiting ultra-high contamination levels. Regarding trace metal contamination, only sediments from S2 showed low risk to marine organisms despite its proximity to the tourist center of Budva. These findings underscore the importance of continued monitoring and effective management strategies to address trace metal contamination in Montenegrin coastal ecosystems.

Keywords: trace metals, marine sediments, pollution indices, Montenegrin open coast

INTRODUCTION

Metals are naturally found in the Earth's crust (Briffa *et al*., 2020; Masindi *et al*., 2021). However, human activities, especially metal-based industries and fossil fuel combustion, have significantly elevated trace metal levels in the environment (Ashayeri *et l*., 2023; Masindi *et al*., 2021), including marine environments, thus posing hazards to marine organisms (Bandara & Manage, 2022; Zhao *et al*., 2023).

Coastal areas, such as the Montenegrin coastline, are experiencing increased trace metal accumulation due to urbanization, industrialization, and maritime traffic (Bhuyan *et al*., 2023; El-Sorogy *et al*., 2022; Lipizer *et al*., 2022). This accumulation raises concerns about metal contamination in marine sediments, which often exceed concentrations in the overlying water (Joksimović *et al*., 2020; Radomirović *et al*., 2021a). Sediments serve as both reservoirs and secondary sources of trace metals, providing insights into the prevailing quality of marine systems and human impacts (Batayneh *et al*., 2015; Joksimović *et al*., 2017).

Understanding the spatial distribution, sources, and ecological implications of trace metals is crucial for effective environmental management. However, the bioavailability of metals from sediments is affected by complex interactions, encompassing their complexation with soluble inorganic and organic ligands in water, along with external factors such as temperature (Chapman *et al*., 1998, Eggleton & Thomas, 2004). As a result, the concentrations of metals in sediments may not accurately reflect their biologically available fractions (Wang *et al*., 1996). Considering this, the assessment of sediment quality regarding metal contamination and the risks posed to marine organisms is essential. Various approaches, including contamination indices, can be utilized for this purpose (Radomirović *et al*., 2021b; Pejman *et al*., 2015). Therefore, this study aims to assess metal contamination and determine trace metal sources in marine sediments along the open coast of Montenegro. By employing two different contamination indices calculated for each location (pollution load index (*PLI)* and contamination severity index (*CSI*)) and one index calculated for each element at the location (contamination factor (C_f)), the study seeks to provide insights into the risks posed by trace metals. Multivariate statistical techniques (cluster analysis and canonical component analysis) will assist in characterizing and distinguishing observed samples.

MATERIAL AND METHODS

Sampling and sample preparation

The studied area is illustrated in Figure 1. Surface sediment samples (upper 5cm) were taken from four locations along the open coast of Montenegro, Adriatic Sea: Žanjice (S1), Budva (S2), Bar (S3), and Ada Bojana (S4). Sampling was conducted in spring of 2019 and autumn of 2020. While the sampling site S1 was situated near the Žanjice beach, at the entrance to Boka Kotorska Bay, its proximity to the open sea strongly influences the site. The remaining sites were located at the open coast, near popular tourist destinations (S2 and S4) or ports (S3).

Sediment samples were collected using a petite ponar grab (Wildco). Prior their freeze-drying at -40 °C for 48 hours (CHRIST, Alpha 2-4 LD plus), they were homogenized and frozen. Fraction smaller than 63 µm, obtained by sieving with a vibratory sieve shaker (Retcsh, AS 200 digit), was used for trace metal analysis. Prepared samples (0.2 g) were digested in a closed vessel microwave digestion system (Anton Paar Multiwave Pro). After adding 5 ml of HNO_3 (> 68%, PrimarPlus–trace analysis grade, Fisher Chemical) and 2 ml of HF (47–51%, superpure for trace analysis, Carlo Erba), the samples were left at room temperature overnight. After room temperature digestion, 2 ml of H_2O_2 (> 30%, analytical reagent grade, Fisher Chemical) were added to the samples for the first stage of the microwave digestion. This was followed by the addition of 10 ml of 4% (w/v) H_3BO_3 (99.97%, trace metals basis, Sigma- Aldrich) in the second stage of the microwave digestion (IAEA, 2011).

Concentrations of Zn, Cu, Ni, Pb, and Cr were determined using flame technique, while Hg contents were determined using a hydride vapor generation technique of an atomic absorption spectrometer (Shimadzu AA 7000). Analysis of As was conducted using an inductively coupled plasma optical emission spectrometer (ICP-OES, Thermo iCAP7400 Duo), while Cd concentrations were measured using a graphite furnace atomic absorption spectrometer (GF-AAS, Shimadzu AA 6800). To ensure the accuracy of analytical methods, certified reference materials (IAEA 158, IAEA 456, and IAEA 458) were used. Recovery values ranged from 88% to 115% for all elements except Ni in IAEA 158, which was 72.6%. Recalculation was performed using recovery values. Trace metal concentrations were expressed in milligrams per kilogram of sample dry weight (mg/kg dw).

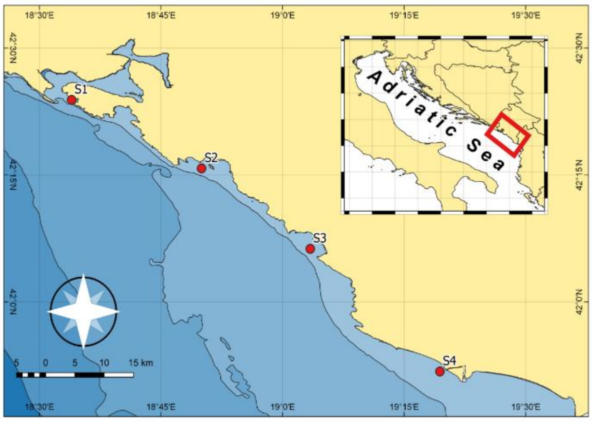

Figure 1. Map of studied area

Statistical analysis

Statistical analysis of the obtained trace metal concentrations was performed using Statistica 14 Software Inc (PaloAlto, CA, USA). Cluster analysis (CA) was utilized to explore the similarities and potential mutual sources of trace elements in sediments samples. In this study, a hierarchical agglomerative procedure was employed on the data using the complete linkage algorithm with "1-Pearson *r*" distance measurements. The comparison of sites using canonical correlation analysis (CCA) aimed to clarify the variations in trace metal pollution and accumulation in sediments across different locations.

Contamination factor (C_f)

The contamination factor serves as a valuable indicator of sediment contamination level compared to preindustrial conditions. It is calculated by dividing the measured concentration of a specific trace metal by the background concentration, which represents the preindustrial reference value for the same metal (Håkanson, 1980; Tomlinson *et al*., 1980) (equation 1):

$$C_f = \frac{C_{metal}}{C_{background}} \quad (1)$$

This factor is classified into four categories:

- $C_f < 1$ indicates a low contamination factor,
- $1 \leq C_f < 3$ suggests a moderate contamination factor,
- $3 \leq C_f < 6$ indicates a significant contamination factor, and
- $C_f \geq 6$ signifies a very high contamination factor (Håkanson 1980).

In this study, the average trace element concentrations in surficial sediments from the Southern Adriatic (Dolenec *et al*., 1998) were used as background concentrations for all the elements except Cd, for which data from the Central and Southern Adriatic Sea were used (Ilijanić *et al*., 2014).

Pollution load index (*PLI*)

To evaluate an overall trace metal contamination at the specific site, the pollution load index (*PLI*) was used (Nagarajan *et al*. 2019). The *PLI* was calculated from the individual contamination factors (n-C_f) obtained for all metals within the sample, using the formula (Tomlinson *et al*., 1980) (equation 2):

$$PLI = \sqrt[n]{C_f^1 \times C_f^2 \times C_f^3 \times \ldots \times C_f^n} \quad (2)$$

A *PLI* value higher than 1 indicates the presence of pollution, while a value below 1 means the absence of any pollution load.

Contamination severity index (*CSI*)

The contamination severity index is a relatively new index, proposed by Pejman *et al*. (2015), which can be used to evaluate the severity of metal contamination in sediments in the specific area. Besides effects range-low (ERL) and effects range-median (ERM) values, corresponding to concentrations below which adverse biological effects rarely occur and concentrations above which adverse effects frequently occur (Long *et al*., 1995), respectively, this index also uses a site specific factor, the weight of trace metal (Wi). This factor is obtained from the results of principal component analysis/factor analysis for each trace metal in the studied area and is calculated by the equation (equation 3):

$$W_i = \frac{(loading\ value_i \times eigen\ value_i)}{\sum_i^n (loading\ value_i \times eigen\ value_i)} \qquad (3)$$

To calculate *CSI* values, the following formula was used (equation 4):

$$CSI = \sum_{i=1}^{n} W_i \left[\left(\frac{C_i}{ERL_i} \right)^{\frac{1}{2}} + \left(\frac{C_i}{ERM_i} \right)^2 \right] \qquad (4)$$

where W_i is the calculated weight of the specific trace metal, C_i is the concentration of the specific metal in the sediment sample, n is the number of trace metals that were analyzed.

CSI values are classified as follows: *CSI* < 0.5 (uncontaminated), 0.5 ≤ *CSI* <1 (very low severity of contamination), 1 ≤ CSI < 1.5 (low severity of contamination), 1.5 ≤ CSI < 2 (low to moderate severity of contamination), 2 ≤ *CSI* < 2.5 (moderate severity of contamination), 2.5 ≤ *CSI* < 3 (moderate to high severity of contamination), 3 ≤ *CSI* < 4 (high severity of contamination), 4 ≤ *CSI* < 5 (very high severity of contamination), *CSI* ≥ 5 (ultra high severity of contamination), as proposed by Pejman *et al*., (2015).

RESULTS AND DISCUSSION

Concentrations of examined trace metals in sediment samples from the open part of the Montenegrin coast are presented in Table 1.

The concentrations of Cr and Ni in sediments from S4 were significantly higher compared to other samples, while As level was notably elevated in sediment sample from S1, collected in 2019 (38,1 mg/kg). For Zn, Cu, and Cd, concentrations were generally higher in samples from S3 and S4 compared to other locations. In contrast, Pb and Hg contents were lowest in samples from S4. Additionally, Hg concentrations were below the quantification limit in samples from S2, whereas the highest contents of both elements were observed in samples from the location S3 (Tab. 1).

The site S4, Ada Bojana, lies downstream of the Bojana River, facing trace metal contamination from waste and sewage discharged by holiday cottages and restaurants. Domestic wastewater effluents, particularly, are a major source of trace metal pollution, notably As, Cr, Cu, Mn, and Ni, in aquatic ecosystems (Cempel&Nikel, 2006). This also explains the high As content in sediment samples from location S1, Žanjice, in 2019 (Tab. 1), given the presence of numerous restaurants along the beach and holiday houses nearby. The site S3, Bar, was located near the Port of Bar, which contributed to higher levels of Cu, Pb, and Hg.

Figure 2 presents the results of CA analysis, obtained as a dendrogram of trace metal clustering. The dendrogram shows two distinct groups of metals defined by two branches. The first branch is divided into two clusters, first one consisting of Cr and Ni, and the second one consisting of Zn, Cu, and Cd, notably from mutual or similar pollution sources. The second branch consists of one cluster (Pb and Hg) and one isolated branch (As), indicating different sources of these non-

Table 1. Trace metal concentrations in sediment samples collected in 2019 and 2020 (mg/kg dw)

Location	Sampling year	Cr	Ni	Zn	Cu	Pb	As	Hg	Cd
S1	2019	172	127	58.8	20.0	24.7	38.1	0.098	0.14
S2	2019	216	20.7	31.4	12.4	28.3	15.0	<0.03	0.245
S3	2019	207	102	85.4	25.6	33.4	15.1	0.086	0.246
S4	2019	492	328	86.9	22.9	19.0	14.8	<0.03	0.211
S1	2020	73.6	63.5	30.5	12.5	23.1	12.8	<0.03	0.103
S2	2020	59.0	23.0	39.9	19.5	29.6	15.2	<0.03	0.115
S3	2020	154	103	107	31.0	36.5	14.0	0.095	0.277
S4	2020	3238	499	109	26.0	22.1	15.4	<0.03	0.322

Table 2. Contamination factor (C_f) values for examined trace metals, and pollution load index (*PLI*) and contamination severity index (*CSI*) values in sediment samples from the Montenegrin coast in 2019 and 2020

Location	Sampling year	C_f								*PLI*	*CSI*
		Cr	Ni	Zn	Cu	Pb	As	Hg	Cd		
S1	2019	1.56	0.99	0.77	0.58	2.25	4.19	0.74	1.46	1.09	2.30
S2	2019	1.96	0.16	0.41	0.36	2.57	1.65	0.11	2.55	0.62	0.90
S3	2019	1.88	0.80	1.12	0.74	3.04	1.66	0.65	2.56	1.19	1.93
S4	2019	4.47	2.56	1.14	0.66	1.73	1.63	0.11	2.20	1.25	9.31
S1	2020	0.67	0.50	0.40	0.36	2.10	1.41	0.11	1.07	0.53	1.09
S2	2020	0.54	0.18	0.53	0.56	2.69	1.67	0.11	1.20	0.53	0.75
S3	2020	1.40	0.80	1.40	0.89	3.32	1.54	0.71	2.89	1.24	1.93
S4	2020	29.4	3.90	1.43	0.75	2.01	1.69	0.11	3.35	1.90	32.8

essential elements, especially As, in relation to the rest of the examined elements. Elevated levels of toxic elements, such as As and Hg, in sediments primarily result from human activities along the Montenegrin coast. Sources include municipal and agricultural runoff, industrial effluents, ports, and shipping areas. Atmospheric deposition also contributes to metal contamination (Khan *et al*., 2017). Human-induced activities have notably increased Hg levels in the biosphere, with a threefold rise attributed to anthropogenic sources (Mason *et al*., 2012). Furthermore, tourism and recreational activities along the coast exacerbate pollution in Montenegrin coastal waters (Joksimović *et al*., 2020).

CCA plot of collected sediment samples in relation to trace metal concentrations in 2019 and 2020 is presented in Figure 3. It can be seen a notable similarity of sediments from S1 and S3 in both years, in contrast to samples from S4, characterized by higher Cr and Ni contents in 2019, especially Cr in 2020, as well as samples from S2, characterized by generally the lowest contents of various trace metals, such as Ni, Cu, Zn, and Hg (Tab. 1).

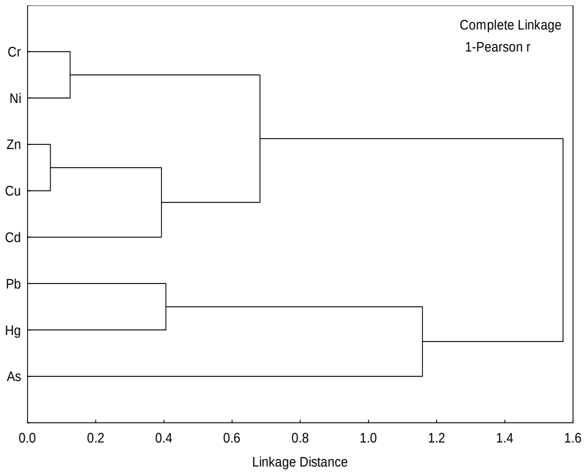

Figure 2. Dendrogram of sediments' trace metal clustering

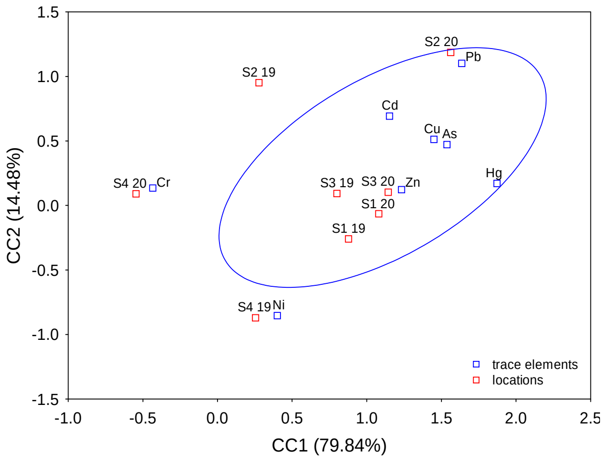

Figure 3. CCA plot of trace metal concentrations in sediments

Contamination factor (C_f) for all analyzed trace metals in sediments, as well as *PLI* and *CSI* values for sediment samples collected along the Montenegrin coast in both 2019 and 2020 are presented in Table 2. The C_f values indicated mainly low or moderate contamination of the sediments from the Montenegrin coast. Only one extreme value, found for Cr in sediment sample from location S4 in 2020, suggested very high contamination. Considerable contamination by Cr in 2019, and by Ni and Cd in 2020, was also noted at the same location. Additionally, considerable contamination of sediments by Pb was found at location S3, and by As at location S1 in 2019.

PLI values indicated the presence of pollution in sediments at locations S3 and S4, while in sediments at locations S2 in both years and S1 in 2020 *PLI* values showed no pollution. Although the *PLI* value for S1 in 2019 slightly exceeded the limit indicating the presence of pollution, on average, this location can be classified as unpolluted. According to the obtained *CSI* values, location S4 exhibited ultra high severity of contamination in both years. In contrast, very low severity of contamination was recorded for location S2 and low to moderate at location S3. Moderate severity of trace metal contamination was found for sediment sample from S1 in 2019, while in 2020, sediments at the same location showed low severity of contamination. Based on these values, only location S2 can be classified as an area without risks to marine organisms (Tab. 2). Given its proximity to the major Montenegrin tourist center, Budva, these findings suggest that the high tourist population and increased recreational activities in the area do not significantly contribute to trace metal contamination of sediments.

CONCLUSION

The coastal region of Montenegro faces ongoing issues due to the continuous discharge of sewage, industrial effluents, and waste from ports, shipping areas, domestic sources, and agriculture. These challenges are worsened by the increasing popularity of recreational activities. Furthermore, the long-term effects pollution from former industries persist. Although sediments from open coastal locations are generally not classified as high polluted due to significant impact of the open sea, this study reveals that considerable contamination is still present in sediments from these areas. Specifically, regarding trace metal contamination, only location S2, Budva, was categorized as an area without risks to marine organisms. Hence, it is crucial to maintain consistent monitoring and implement effective management strategies to address environmental issues caused by trace metal contamination in the area.

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Zagađenje sedimenta metalima u tragovima duž otvorene obale Crne Gore

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SAŽETAK

Obalni region Crne Gore suočava se sa znatnim ekološkim izazovima zbog kontinuiranog zagađenja iz različitih izvora. Ova studija istražuje nivo zagađenja metalima u tragovima u uzorcima sedimenta prikupljenim duž otvorene obale Crne Gore u 2019. i 2020. godini. Povišeni sadržaji nekih elemenata pronađeni na lokaciji S4, ali i na S1, uglavnom se mogu pripisati zagađenju iz obližnjih vikendica i restorana. Multivarijantna statistička analiza, uključujući klaster analizu i kanoničku komponentnu analizu, korišćena je za identifikaciju grupisanja metala i varijacija zagađenja metalima u tragovima u sedimentima sa različitih lokacija. Vrijednosti faktora kontaminacije (C_f) ukazale su na pretežno nisku do umjerenu kontaminaciju duž crnogorske obale, sa uočljivim izuzecima kao što je S4, gdje je nađena visoka kontaminacija Cr u 2020. godini i značajna kontaminacija Cr u 2019. godini, kao i Ni i Cd u 2020. godini. Vrijednosti indeksa opterećenja zagađenjem (*PLI*) pokazale su prisustvo zagađenja na lokacijama S3 i S4, dok su vrijednosti indeksa ozbiljnosti kontaminacije (*CSI*) otkrile različite stepene zagađenja na različitim lokacijama, pri čemu su za lokaciju S4 pronađeni ultra-visoki nivoi zagađenja. Što se tiče zagađenja metalima, samo sedimenti sa S2 su pokazali nizak rizik za morske organizme uprkos blizini turističkog centra Budve. Ovi rezultati naglašavaju važnost kontinuiranog praćenja i efikasnih strategija upravljanja kako bi se riješilo zagađenje izazvano metalima u tragovima u crnogorskim obalnim ekosistemima.

Ključne riječi: metali u tragovima, morski sedimenti, indeksi zagađenja, otvorena obala Crne Gore