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ANALYSIS OF SEDIMENT'S QUALITY OF THE OŁAWA RIVER IN POLAND USING INDICES OF HEAVY METALS AND STATISTICAL TECHNIQUES

This case study reports a dataset enabling the analysis of heavy metal concentrations in sediments collected in the Oława River basin. The focus is on the ecological risk associated with six metals: copper (Cu), zinc (Zn), lead (Pb), chromium (Cr), nickel (Ni), and cadmium (Cd). The key information on the pollution status of the aquatic environment is provided by the single- (*EF*, I_{geo}, and *PI*) and the multi-elemental (*PLI*, PI_{Nem}, *RI*, and *MERMQ*) pollution indices, and statistical techniques such as Spearman's correlation, the principal component analysis, and the cluster analysis. The sediments indicate the presence of Cu, Zn, Pb, and Ni, a smaller input of Cr, and the absence of Cd, according to criteria from the State Working Group on Water in Germany (the LAWA's guidelines) and pollution indices. As assessed by the *RI* index and the sediment quality guidelines (the SQGs), the potential ecological risk is possible to occur at some sampling points as a result of uncontrolled emission of pollutants. This resource supports environmental monitoring, risk management, and comparative research of aquatic environments.

1. INTRODUCTION

River sediments are a natural sorbent of heavy metals and an indicator in aquatic ecosystems, indicating their presence [1–3]. Heavy metals are not biodegradable, but their biotransformation is possible. Through the self-purification process of surface waters, the soluble forms of heavy metals, thanks to sorption and then sedimentation, move to sediments. Then the water quality improves and the concentration of metals in sediments increases. Heavy metals are usually bound to the organic matter in sediments,

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either precipitated as hydroxides, sulfides, and carbonates, or adsorbed on hydrated Fe/Mn oxides and inaccessible to aquatic organisms. The release of heavy metals deposited in sediments is possible by changing the physical and chemical properties of water: pH, salinity, redox potential, and the content of organic chelates. Then sediments become an important source of water pollution, which is best observed in river basins [2]. Natural factors causing the imbalance in the water-sediment system result mainly from geomorphology and climatic conditions, which determine the course of rock weathering processes, migration, and accumulation of elements [4, 5]. Anthropogenic factors most often include the discharge of industrial and municipal sewage, as well as runoff from agricultural areas and roads [1, 3, 6].

In assessing the environmental quality, increasingly the single and multi-elemental pollution indices are used [1, 3, 6–8]. They enable comparison of metal levels at different test sites based on a relatively simple calculation method. The enrichment factor (*EF*) allows one to determine the probability of the natural or anthropogenic origin of detected metals. Other popular indices that allow assessing the effect of the single trace element concentration include the pollution index (*PI*), the geoaccumulation index (I_{geo}), and the contamination factor (C_{f}). All of them characterize the ratio of the detected metal in the tested environment to the average level of this element in the standardized geochemical background. The differences between the individual indices result from modifications to the formulas aimed at eliminating background variability and the adopted evaluation criteria. Some indicators allow the integration of the group of heavy metals and comprehensively assess the actual degree of environmental pollution. These include the pollution load index (*PLI*), the Nemerow pollution index (PI_{Nem}), the contamination severity index (*CSI*), and the modified degree of contamination (mC_{d}). As a result of the constantly growing ecological awareness, indices assessing environmental risk such as the mean effects range median quotient (*MERMQ*) and the potential ecological risk index (*RI*), are becoming more and more common. Classification of indices according to defined criteria allows for the designation of classes with a specific degree of pollution and risk.

Pollution indices analysis is increasingly supported by multivariate analysis techniques in monitoring aquatic environments. Multivariate statistical techniques such as the hierarchical cluster analysis (HCA), the principal component analysis (PCA), and the factor analysis (FA) are used to identify sources and typologies of contaminants, as well as to indicate relationships between samples or variables [1, 3, 6, 8–12].

The chemical analysis of sediments from the Oława River in Lower Silesia, Poland, presented in this article enables the assessment of anthropogenic pressure on the aquatic environment, which may pose a threat to living organisms. The research aims to assess heavy metal contamination (Cu, Zn, Pb, Cr, Ni, and Cd) of surface sediments in the lower section of the Oława River using reliable pollution indices, determine the main sources of the studied metals (anthropogenic or natural) by using statistical techniques, and present the relationships between metals.

2. MATERIALS AND METHODS

Research object. The object of the study is the lower section of the Oława River in SW Poland, with a length of 24 km (Fig. 1). The Oława River has a total length of 99.01 km and a catchment area of 1167.4 km^2. It originates at an altitude of about 315 m above sea level in the Sudeten Foreland in the Kamiennik commune. The Oława River is a left tributary of the Odra River, which has its mouth at 250.4 km in the city of Wrocław and an altitude of approximately 115 m above sea level. The average water flow in the Oława River is 3.43 m^3/s. The catchment area in the middle section of the Oława River runs through agricultural areas with intensive agricultural production, which may cause problems with water quality. In the lower part of the basin, there is a highly urbanized agglomeration of Wrocław, where various types of individual economic activity are carried out. These are a potential source of pollution that may be introduced intentionally or unintentionally through the inflow of Brochówka waters feeding the Oława River in the Wrocław district. Additional sources of water pollution in the Oława River are its tributaries: Krynka (Strzelin commune), Młynówka and Gnojna (Oława commune). The average slope of the catchment area is about 0.62%, the density of the river network is about 0.34 km/km^2, and the forestation of the catchment area is about 19%. The climatic water balance is about 71 mm. The Oława River is of particular importance to the city of Wrocław, as it is the main source of drinking water supply for its residents.

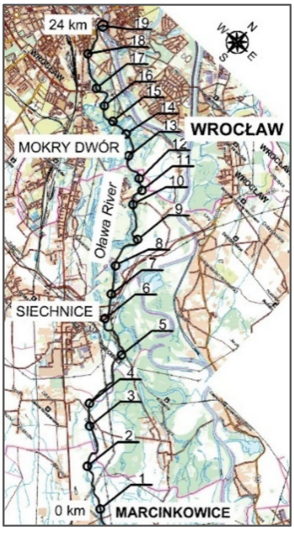

Fig. 1. Map of a monitored area with sampling location numbers

Sampling procedure. The research was carried out on the sediments of the Oława River, on a 24 km long section, up to its confluence with the Odra River in Wrocław. Samples were collected in the summer of 2023 at a low water level in the Oława River at a depth of 0.5 to 1.0 m below the water table, from the surface layer of sediments up to 5 cm. Samples were collected with a sampler, enabling the collection of sediments with an intact structure. The collection was carried out on average every 1.5 km in 19 locations along the lower bank of the Oława River (Fig 1). In each location, three independent places were designated, where two subsamples were taken. Both subsamples of sediment were averaged after drying and sieving through a sieve with 1 mm mesh. A total of 57 sediment samples weighing about 250 g were collected.

Analytical methods. The air-dried sediment samples were digested in 65% nitric acid. The contents of Cu, Zn, Pb, Cr, and Ni were determined using an air-acetylene flame by atomic absorption spectroscopy. The AAS method with the electrothermal atomization in a graphite furnace was used to determine Cd [13]. Background interference in elemental measurements was corrected using a deuterium arc lamp. To determine the precision of the analytical method, triplicate analyses of mineralizate solutions were performed – with no more than 5% exceedances of the standard deviation, the coefficient of variation within 10%, and the confidence interval for the *p*-value of the Student's *t*-test equal 0.05. The measurement readings were controlled using blank samples of certified reagents. The detection limits were estimated based on the standard deviation for three repetitions of the digestion blank. The accuracy of the determinations was controlled by the standard addition method, obtaining 94–98% of the recovery percentage. Validation of the analytical methods was performed using certified metal solutions. All chemical analyses in the Environmental Research Laboratory at the Faculty of Environmental Engineering, Wrocław University of Science and Technology, were performed [8].

Data treatment. Länder-Arbeitsgemeinschaft Wasser (LAWA). The LAWA's guidelines for the quality of sediments according to the classification of the State Working Group on Water in Germany divide them into 7 purity classes depending on the contamination of the aquatic ecosystem with heavy metals [14]. The limit values dividing sediments into purity classes are shown in Table 1.

Table 1

Permissible concentrations of heavy metals [mg/kg] in sediment sample according to the LAWA's classification [14]

Metal	Criteria						
	1	1–2	2	2–3	3	3–4	4
Cu	≤ 20	≤ 40	≤ 80	≤ 160	≤ 320	≤ 640	> 640
Zn	≤ 100	≤ 200	≤ 400	≤ 800	≤ 1600	≤ 3200	> 3200
Pb	≤ 25	≤ 50	≤ 100	≤ 200	≤ 400	≤ 800	> 800
Cr	≤ 80	≤ 160	≤ 320	≤ 640	≤ 1280	≤ 2560	> 2560
Ni	≤ 30	≤ 60	≤ 120	≤ 240	≤ 480	≤ 960	> 960
Cd	≤ 0.3	≤ 0.6	≤ 1.2	≤ 2.4	≤ 4.8	≤ 9.6	> 9.6

1 – unpolluted, 1–2 unpolluted/moderately polluted, 2 – moderately polluted, 2–3 moderately/heavily polluted, 3 – heavily polluted, 3–4 heavily/very heavily polluted, 4 – very heavily polluted.

Sediment quality guidelines (SQGs). The SQGs define allowable concentrations of contaminants. Concentrations corresponding to the 10th and 50th percentiles present the low (*ERL*) and the median (*ERM*) ranges of adverse biological effects, respectively. These two guideline values define three ranges in concentrations, presented in Table 2 [6].

Table 2

The SQGs values [mg/kg] [6]

Metal	Cu	Zn	Pb	Cr	Ni	Cd
ERL	150	34	46.7	20.9	81	1.2
ERM	410	270	218	51.6	370	9.6

< ERL rarely occurs adverse effects. ≥ ERL and < ERM occasionally occur adverse effects. ≥ ERM frequently occurs adverse effects.

Statistical analysis. Calculations of statistical parameters such as average (Avg), median (Med), minimum (Min), maximum (Max), standard deviation (SD), coefficient of variation (CV), skewness, and kurtosis for metal concentrations determined in 57 sediment samples were performed using the Statistica software package. The data were checked for normal distribution using and 95% confidence level. In the absence of a normal distribution, Spearman's rank correlation coefficients were calculated, investigating the associations of metal concentrations in pairs. Correlations were considered very strong for an absolute value above 0.8, strong for the range 0.8–0.6, moderate for 0.4–0.6, and weak below 0.4. The factor loadings in the principal component analysis (PCA) were considered significant for values of 0.7 or greater [15]. The hierarchical cluster analysis (HCA) was performed using Ward's algorithm, the squared Euclidean distance, and the median values of the pollution indices for each location.

Pollution indices. The sediment quality monitoring was performed using single-element indices represented by the enrichment factor (*EF*), the pollution index (*PI*), and the geoaccumulation index (I_{geo}), as well as multi-element indices represented by the pollution load index (*PLI*), the Nemerow pollution index (PI_{Nem}), the potential ecological risk index (*RI*), and the mean *ERM* quotient (*MERMQ*). All formulas and classifications of pollution indices have been presented elsewhere [8]. The median concentrations of elements in stream sediments are the reference values, in mg/kg: 17 (Cu), 71 (Zn), 20.5 (Pb), 63 (Cr), 21 (Ni), 0.28 (Cd) reported by Salminen et al. [16].

3. RESULTS AND DISCUSSION

3.1. CONTENT OF HEAVY METALS

The results of the statistical analysis of sediments are summarized in Table 3. The contents of heavy metals determined in the sediments of the Oława River show variability along the course of the river. The median of metals can be arranged in the following series: Zn > Cu > P ≈ Ni > Cr > Cd. For maximum metal levels, the order of Cr and Ni changes in the series. Strong spatial variability according to the coefficient of variation above 45% was noted for all tested metals, except for Cd (normal distribution), which is confirmed by the Shapiro–Wilk *W*-test, and the deviation from the Gaussian curve in the frequency histograms. The high heterogeneity of metal concentrations in sediments results from the point inflows of pollutants, which are diluted along the Oława River. Positive kurtosis for Cu, Zn, and Pb indicates the presence of a large pool of results clustered around the mean value. Negative kurtosis for Cr, Ni, and Cd indicates dispersion of most results, significantly deviating from the mean value. Positive skewness values noted for all metals indicate that the mean values are higher than the median values.

Table 3

Descriptive statistics of metal contents [mg/kg] in sediment samples

Metal	Avg	Med	Min	Max	SD	CV [%]	Skewness	Kurtosis
Cu	58.71	53.53	15.45	177.45	38.36	65.34	1.49	2.83
Zn	248.75	224.28	19.55	922.63	214.81	86.36	1.63	3.00
Pb	43.54	42.33	0.63	174.08	40.95	94.04	1.66	3.36
Cr	52.63	31.15	1.60	118.40	37.30	70.87	0.36	–1.46
Ni	44.35	42.55	5.15	90.68	28.17	63.51	0.25	–1.47
Cd	0.07	0.07	0.05	0.10	0.01	17.64	0.55	–0.02

In line with the LAWA's criteria [14], the studied sediments were not contaminated with cadmium and chromium (Table 4). In 6 locations, no exceedances of the determined metals were recorded (the 1st or the 1st–2nd purity class). In 11 other locations, increased contents of copper, zinc, lead, and/or nickel were recorded in the sediments at the level of the 2nd class or the 2nd–3rd purity class, which indicates a moderate environmental load of these elements. A significant problem concerning the quality of sediments in the Oława River are two locations heavily contaminated with copper or zinc (the 3rd class). This might be due to the service and production activities, the construction or modernization of roads and small bridges, and the drainage of storm sewage.

Comparison of sediment samples with the SQGs data (Fig. 2) shows that Cu, Pb, and Cr concentrations are between the ERL and ERM in 68%, 32%, and 32% of samples, respectively, indicating that these metals pose an occasional threat to organisms in the study area. All samples for Cd are lower than the ERL, showing that cadmium has no harmful effect on biota. Zn and Ni concentrations are higher than the ERM in 11% and 37% of samples, respectively, showing that these metals have an adverse biological effect on aquatic organisms in the entire studied region. Based on the sediment quality guidelines [6], the obtained results indicate that some metals, especially Zn and Ni, are the most important pollutants in the studied section of the Oława River.

Table 4

Assessment of sediment samples
in studied locations according to the LAWA's classification [14]

No.	Cu	Zn	Pb	Cr	Ni	Cd
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						

1 class	2 class
2–3 classes	3 class
3–4 classes	4 class

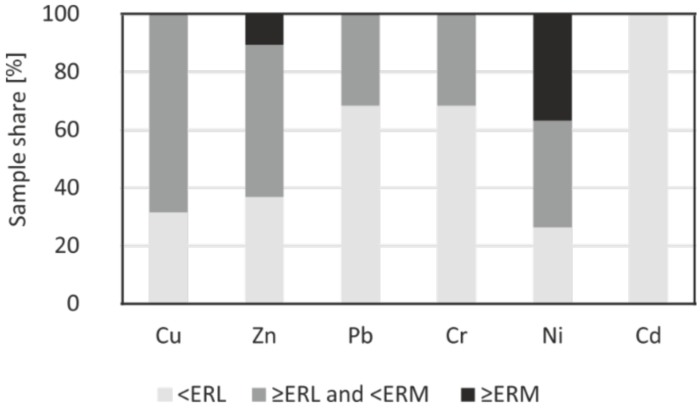

Fig. 2. Comparison between sediment samples
with mentioned SQGs values

Spearman's correlation analysis indicates strong relationships between metal pairs, with three exceptions for cadmium (Table 5). According to the classification, 27% of very strong and 53% of strong correlations were identified. Moderate and weak correlations between metals account for 7% and 13%, respectively.

Table 5

Spearman's correlation analysis ($p < 0.05$)

	Cu	Pb	Cr	Ni	Cd
Zn	0.89	0.88	0.72	0.74	0.61
Cu	–	0.86	0.69	0.65	0.61
Pb	–	–	0.71	0.60	0.48
Cr	–	–	–	0.89	0.20
Ni	–	–	–	–	0.35

The result of the PCA analysis for the elemental composition of the sediments is shown in Fig. 3. According to the adopted criterion, the first two PC components are statistically significant and contribute to 86% of the total variance of the data. The first component (PC1) explains 64.5% of the total variance and shows the correlation of the concentrations of 4 metals in sediments. In this group, there is a stronger relationship between Cu, Zn, and Pb than Cd.

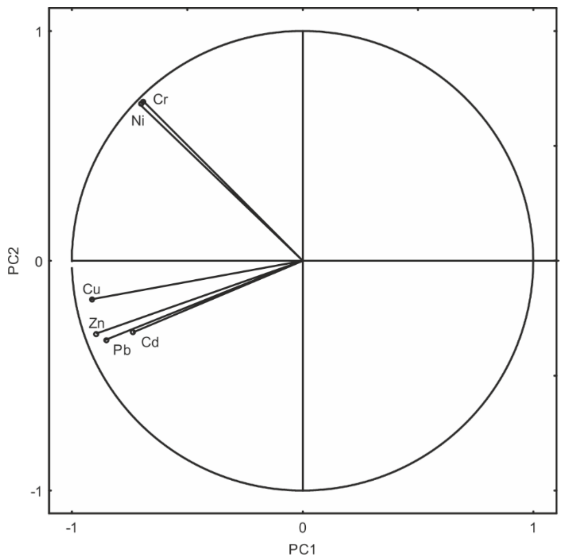

Fig. 3. Projection of metals on the factor-plane

The second component (PC2) explains 21.5% of the total variance for the strongly correlated Cr and Ni. The factor loading of these two elements are similar in both PC's components and statistically significant. The positions of the Cr and Ni vectors concerning the metals from the PC1 group indicate a weak correlation between the elements of both groups (PC1 and PC2) and different sources of their origin. The loading factor of cadmium is also included in the third component of PC3 (9.6%) and suggests different origins of this element in the aquatic environment. The statistically significant contribution of all studied elements suggests the anthropogenic enrichment of sediments from various production and service activities and the discharge of rainwater into the Oława River.

3.2. POLLUTION ASSESSMENT

The pollution indices of heavy metals have a universal character in the evaluation of the environmental quality thanks to the use of the background reference values, in this case representing the content of trace elements in stream sediments [16]. The medians of the single indices of heavy metals do not change their position in the series in comparison with the arrangement of their concentrations (Tables 3, and 6).

Table 6

Descriptive statistics of pollution indices for sediment samples

Index	Metal	Avg	Med	Min	Max	SD
EF	Cu	2.09	1.19	0.77	7.16	1.75
	Zn	1.98	1.14	0.60	14.65	3.03
	Pb	1.18	0.63	0.08	9.68	2.06
	Cr	0.39	0.36	0.07	0.87	0.19
	Ni	0.97	0.97	0.47	1.74	0.24
	Cd	0.21	0.09	0.04	0.77	0.20
I_{geo}	Cu	0.90	1.07	–0.72	2.80	0.96
	Zn	0.63	1.07	–2.45	3.11	1.47
	Pb	–0.55	0.46	–5.62	2.50	2.31
	Cr	–1.37	–1.60	–5.88	0.33	1.46
	Ni	0.09	0.39	–2.61	1.53	1.16
	Cd	–2.65	–2.69	–3.14	–2.07	0.25
PI	Cu	3.45	3.15	0.91	10.44	2.26
	Zn	3.50	3.16	0.28	12.99	3.03
	Pb	2.12	2.06	0.03	8.49	2.00
	Cr	0.84	0.49	0.03	1.88	0.59
	Ni	2.11	2.03	0.25	4.32	1.34
	Cd	0.24	0.23	0.17	0.36	0.04
PLI		1.37	1.59	0.19	3.08	0.78
PI_{Nem}		3.20	3.03	0.68	9.81	2.35
RI		50.91	52.60	12.54	117.86	28.22
MERMQ		0.34	0.36	0.04	0.72	0.21

The only difference is that the values of Zn and Cu are very similar, and the *EF* index value for Ni is higher than that for Pb. Also, the order of the maximum values for single indices does not change, except for the comparable values of Cr and Cd for the *EF* index. The single-element pollution indices confirm the heterogeneous distribution of metals along the studied section of the Oława River.

Table 7

Quality assessment of sediment samples in studied locations according to pollution indices

No.	*EF*						I_{geo}						*PI*						*PLI*	PI_{Nem}	*RI*	*MERMQ*	Legend	
	Cu	Zn	Pb	Cr	Ni	Cd	Cu	Zn	Pb	Cr	Ni	Cd	Cu	Zn	Pb	Cr	Ni	Cd						
1																							*EF*, *PI*	PI_{Nem}
2																							1 class	1, 2 classes
3																							2 class	3 class
4																							3 class	4 class
5																							4 class	5 class
6																							5 class	
7																								
8																							I_{geo}	*RI*
9																							0 class	*MERMQ*
10																							1 class	1 class
11																							2 class	2 class
12																							3 class	3 class
13																							4 class	4 class
14																							5 class	
15																							6 class	
16																								
17																							*PLI*	
18																							1, 2 classes	
19																							3 class	

The *EF* index shows a moderate (the 2nd class) or significant (the 3rd class) enrichment of 37% sediments with Cu, and additionally in some samples with Zn and Pb as shown in Table 7. The *PI* and the I_{geo} indices are practically agreed in assessing the degree of contamination of samples and indicate the contamination of almost all sediment samples. The established criteria for the classification of the single pollution indices indicate greater contamination of sediments according to the *PI* index than the I_{geo} index. The I_{geo} index shows that only two sediment samples are moderately (the 3rd class) or strongly (the 4th class) polluted with at least one metal. According to the *PI* index, two sediments are moderately (the 3rd class), and 11 samples are strongly or very strongly contaminated with heavy metals (the 4–5 classes). Only 4 sediment samples are slightly enriched with Cu, Zn, and/or Ni. Two samples were assessed as uncontaminated by metals. The largest input to contamination of the studied sediments was made by Cu, Zn, Pb, and Ni, the smaller of Cr, and no contamination of Cd was noted. The background exceedance was almost 10.5 times for Cu, 13 times for Zn, 8.5 times for Pb, 4.5 times for Ni, and 2 times for Cr (Table 6).

The *PLI* index shows contamination of 12 sediments with heavy metals regardless of the amount of the background value exceedance (Table 7). In the assessment of the PI_{Nem} index, as many as 15 samples are loaded with trace elements to varying degrees from insignificant (the 3rd class) to high contamination (the 5th class). The potential risk ecological index (*RI*) classifies only two sediments as moderately contaminated with metals. Their values of the toxic-response factor (E_r) for Cu and Pb are in the range of 40–80 for sediment samples Nos. 5 and 9, respectively, and this is also due to the highest *PI* values at these sampling points (Table 7). The *MERMQ* index shows a medium or a high risk level for samples from the Oława River, with a 21% and 49% probability of toxicity for tested sediments.

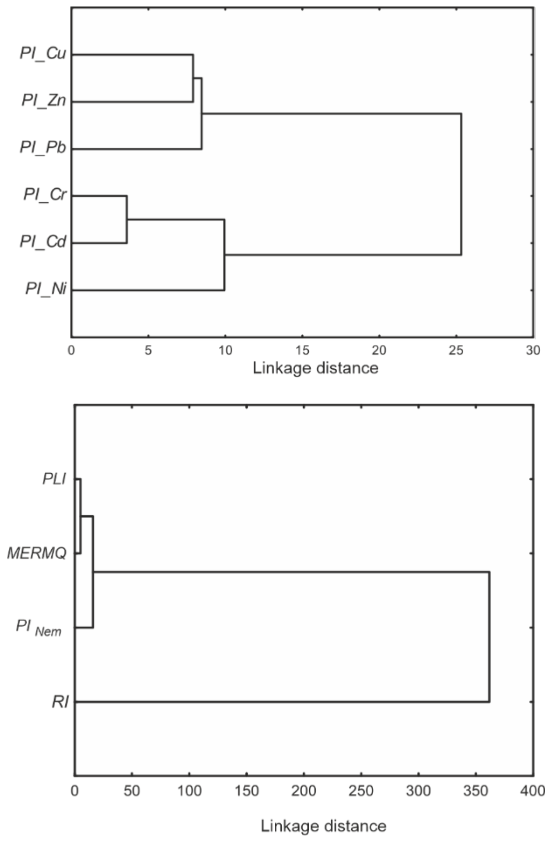

Fig. 4. Dendrograms of pollution indices

Identification of metal clusters by the HCA method shows a strong aggregation of zinc with copper, which is additionally associated with lead (Fig. 4). The second group is formed by the aggregation of chromium with cadmium, which is also associated with nickel. Close bond distances within both metal groups might indicate a common source of sediment contamination. Dendrograms for the multi-element indices show close integration of the *PLI* and *MERMQ* indices together with the *PIN* index. The *RI* index might not be compared with other integrated pollution indices, because its separation is caused by very high values of toxic response factors for studied metals in the calculation formula.

4. CONCLUSIONS

The analysis of the sediment quality of the Oława River provided a comprehensive assessment of heavy metal contamination and indicated various anthropogenic sources. Pollution indices of heavy metals show high variability in their distribution along the course of the river and high element loading of the tested sediments. The lack of homogeneity is the result of the long-term anthropogenic pressure and point emissions of pollutants, in particular those related to runoff from roads and paved areas, service and production activities, and modernization of road infrastructure and small bridges. Studied samples are mainly polluted with Cu, Zn, and Pb, to a lesser extent with Ni and Cr, according to the *PI* and the I_{geo} indices and the LAWA's classification. Sediments were assessed moderately to very strongly enriched with these four pollutants and minimally enriched with Cr. Cadmium does not contaminate the studied samples. The multi-elemental pollution indices of the *PLI* and the PI_{Nem} confirm a high contamination of about 65% of sediment samples. The *MERMQ* index shows a moderate to high ecological risk in locations identified as polluted by the PI_{Nem} index. In general, five heavy metals pose a threat to the biota of the Oława River, with the greatest danger being caused by zinc and nickel in 7 locations with concentrations above the *ERM* value. Correlations between metals in the assessment of the PCA analysis are confirmed by Spearman's correlation analysis. The statistical techniques used in this resource indicate various anthropogenic sources of the studied heavy metals. It is recommended to continue monitoring the elemental composition of sediments in the Oława River to prevent uncontrolled inflow of pollutants that might cause an ecological risk.

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