

Evaluation of Seasonal Distribution of Heavy Metals in the Water of Shkumbini River, Albania

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Abstract—Heavy metal levels were monitored during 2021–2022 on six campaigns along the Shkumbini river. Water samples were collected from eight stations, covering the whole watershed area through six successive monitoring expeditions. Samples analyses were performed by Graphite Furnace Atomic Absorption Spectroscopy (GF-AAS). The amount of heavy metals in water was found to be in the order Fe> Ni> Cu> Cr> Cd> Pb> Mn> Zn. Principal component analysis and correlation analysis results showed that variation of heavy metals elements in Shkumbini River is related to the heterogeneity of human activities, which is mainly affected by urban industrial and agricultural pollution, and natural environments of the river with different background values. According to the CE Directive (75/440/EEC) and the European Community Directive (EEC/EEAC/EC 78/659) on “Quality of fresh waters supporting fish life” the results obtained show that the content of iron, manganese, lead, nickel, mercury and copper are quite high for stations Sh 4, Sh 5, Sh 6, Sh 7 and Sh 8, above the allowed limits classifying these waters polluted. The results of this study could provide useful information for further understanding of the transportation of heavy metals in the Shkumbini River water system.

Keywords—graphite furnace atomic absorption spectroscopy heavy, metals, Shkumbini River, water quality

I. INTRODUCTION

The Shkumbini River is one of the largest rivers in the country, with a length of 181 km, a catchment area of 2444 km² and an average source height of 753 m [1, 2]. Due to the specificity of the Shkumbini River and the importance of its water use, there is a need for continuous research on water quality and pollution levels, especially in the lower reaches of the river, where agricultural and urban activities are concentrated. Anthropogenic activities, such as municipal and industrial emissions, agricultural activities, and mineral development processes, combined with natural processes, such as precipitation, erosion, and weathering, determine the river water quality [3, 4]. River nutrition and heavy metal contents are important factors affecting river water quality. Industrial and municipal discharges will also have significant impacts on the nutrient elements and heavy metal content in rivers [5, 6]. Heavy metals are the most dangerous pollutants as they are persistent and accumulate in water, sediments and in the tissues of living organisms, through two mechanisms, called “bio-concentration” (uptake from the ambient environment) and “bio-magnification” (uptake through the food chain) [7]. Fish are able to aspire to take up and retain heavy metals dissolved in water by active or passive processes. The toxic effects of metals occur when the mechanisms of extraction, metabolism, storage and detoxification are no longer able to keep up with intake rates

[8]. Toxic heavy metals entering the aquatic environment are adsorbed on particles, although they can form free metal ions and soluble complexes that are available for uptake by biological organisms. Increasing waste levels of heavy metal content in water, sediments and biota has resulted in decreased productivity and increased human exposure to harmful substances. Many of these metals tend to remain in the ecosystem and move from one link to another within the food chain. Therefore, in the conducted study, efforts are made to quantify the concentration of toxic heavy metals in the water of the Shkumbini River [9]. The study was carried out in order to get to know the pollution load, in order to estimate the frequency of pollution due to heavy toxic metals such as Fe, Mn, Ni, Cr, Cd, Cu, Pb and Zn in water.

II. MATERIALS AND METHODS

The survey was conducted in the Shkumbini River Basin during the period, from May 2021 to November 2022, and eight points (Fig. 1) along the Shkumbini River were selected as representative points of the main stream of the Shkumbini River, whose coordinates are given in Table 1. A total of 144 river water samples were collected from 8 sampling sites in the Shkumbini River, Albania. The subject of research focused on heavy metals (Fe, Cr, Cu, Zn, Ni, Cd). Water samples were collected in clean plastic bottles, soaked with HNO₃ 10% and rinsed with distilled water. The samples were filtered immediately upon arrival at the laboratory using 0.45 μm glass filter paper and the filtrate was acidified to pH 2 with ultrapure grade nitric acid, in order to minimize precipitation and adsorption on the walls of the container. Heavy metals were determined by Atomic Absorption Spectroscopy (AAS), Graphite Furnace Atomization and Electrothermal Atomization (GFA/ETA) system, of the NOVA 400 spectrometers type Jena, Novva and AAS [10]. Standard deviation was calculated for three repeated analyses of each sample. Quality control of the obtained results was conducted by analyzing a certified reference material. The correlation between the heavy metals analyzed in the Shkumbini River sediments was performed by calculating the correlation coefficients using the Origin Pro 2023, descriptive statistics, one-sample hypothesis tests and one-way analysis of variance (ANOVA). The results obtained regarding the content of heavy metals were treated statistically with the one way ANOVA method to judge if there are significant differences between the results obtained regarding the content of each metal between the stations as well as in different seasons.

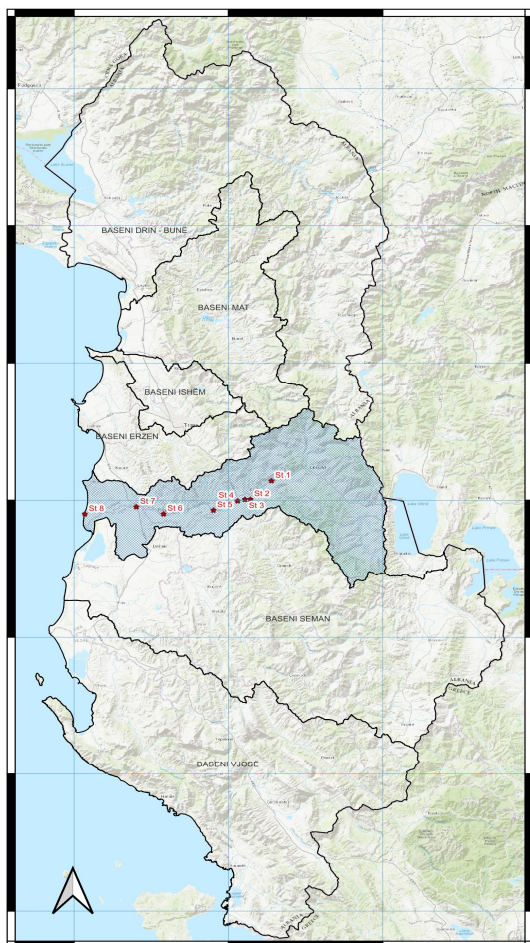


Fig. 1. The watershed of the Shkumbini river and the sampling stations.

Table 1. The coordinates of the stations

Stations	Coordinates WGS84 system	
	N	E
St 1	41°08'53"	20°09'59"
St 2	41°06'02"	20°05'07"
St 3	41°05'15"	20°03'50"
St 4	41°04'57"	20°02'08"
St 5	41°03'08"	19°56'33"
St 6	41°02'23"	19°45'02"
St 7	41°03'46"	19°38'43"
St 8	41°02'18"	19°26'49"

III. RESULTS

Figs. 2–9 presents the descriptive analysis of the results obtained regarding the content of heavy metals in the waters of the Shkumbini River. The figures represent the variation of concentrations during each month, the minimum point represents the minimum value minus the standard deviation value recorded during each month at all stations and the maximum point represents the maximum value plus the standard deviation. The midpoint represents the average value of heavy metals for all stations.

The content of Fe in the stations under study and during different periods varies from 1.1–10.5 mg/L. The results show a significant increase in the content of Fe in the waters of the Shkumbini River mainly during the summer and spring seasons, where the highest values of the content of this element were recorded on stations Sh 1, Sh 2, Sh 4, and Sh 5). The cause of this increase is undoubtedly the high

temperatures of these seasons, as a result of which the assimilation of Fe by organisms decreases [4]. The Fe content was lower during the months of February and November in almost all stations (Fig. 2).

If we refer to the results obtained for each station, we can say that the highest Fe content was recorded at station Sh 4, almost in all sampling periods, followed by stations Sh 2, Sh 3, Sh 5.

On the other hand, manganese has shown a similar distribution trend as iron (Fig. 3), where increased values appear during the hot season which are mainly related to the mobilization of manganese from sediments in water due to the decomposition of organic matter according to microbial activity [6]. The results show a significant increase in the content of Mn mainly during the spring and summer season, where the highest values of the content of this element was recorded. The values fluctuate between the highest value of 0.41 mg/L recorded in July 2021, at station Sh 6 and the lowest value of 0.01 mg/L recorded in November 2022, at station Sh 1. The cause of this increase is undoubtedly the high temperatures of these seasons, as a result of which the assimilation of Mn by organisms decreases [11]. The content of Mn was found to be lower during the months of May, February and November in almost all stations. The highest Mn content was recorded at stations Sh 6 and Sh 7, in almost all sampling periods, followed by stations Sh 3, Sh 4, Sh 8.

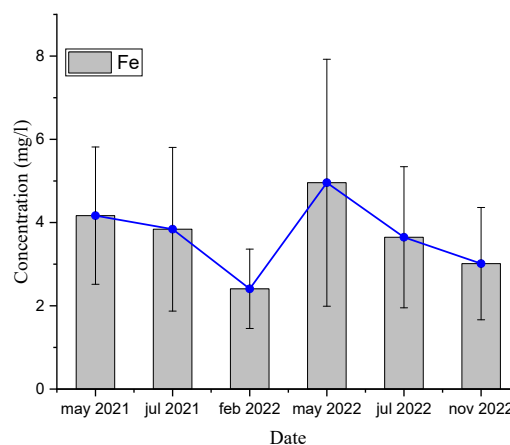


Fig. 2. Average values and standard deviation for Fe.

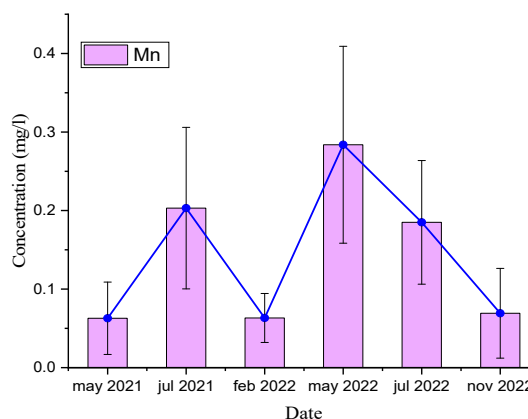


Fig. 3. Average values and standard deviation for Mn.

The content of Ni in the stations under study and during different periods varies from 0.1–8.23 µg/L. The levels of Ni express small variations between the stations (Fig. 4), while its highest levels were recorded in the summer and autumn seasons.

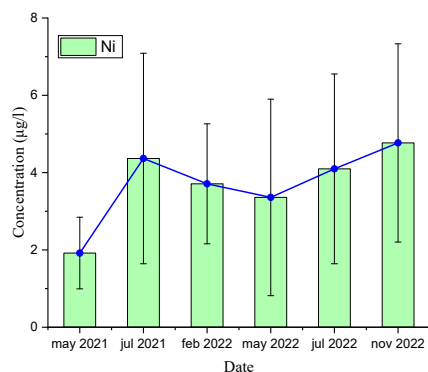


Fig. 4. Average values and standard deviation for Ni.

The maximum value was recorded in July 2021 with a value of 8.23 µg/L at station Sh 5 and the minimum value was recorded in July 2022 with a value of 0.1 µg/L at station Sh 8. The cause of this increase is undoubtedly the high temperatures of these seasons, as a result of which the assimilation of Ni by organisms decreases [12].

Chromium levels increased during autumn and winter, with wide variations in monitoring stations (Fig. 5).

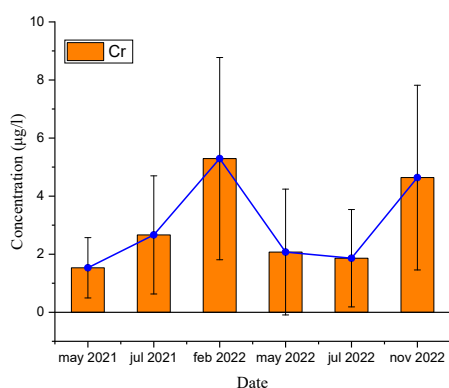


Fig. 5. Average values and standard deviation for Cr.

The maximum value was recorded in February 2022 with a value of 9.65 µg/L at station Sh 6, while the minimum value was recorded in May 2022 with a value of 0.25 µg/L at station Sh 2. The iron-bearing minerals of the region are cut with those of chromium content. This is a specific feature of our country's iron ores, which have a high chromium content ranging from 4%–4.30% CrO₃.

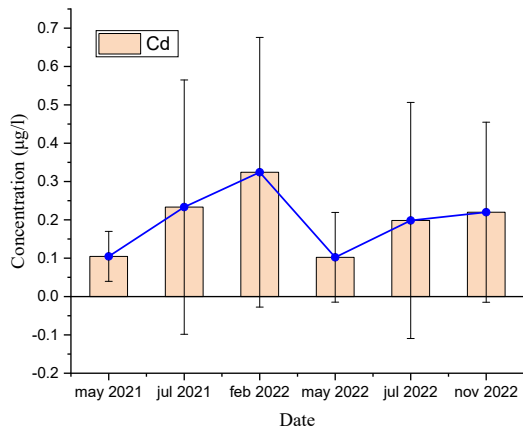


Fig. 6. Average values and standard deviation for Cd.

Cadmium has resulted in wide fluctuations mainly during autumn and winter in all stations, marking the highest value

of 0.95 µg/L in July 2021 at station Sh 7, while the minimum was recorded in May 2022 with a value of 0.1 µg/L at Sh 2 station.

Lead values have shown significant increases during the fall and winter (Fig. 7). The highest value of 0.7 µg/L of lead was recorded during the winter of 2022 at station Sh 5, while other high values were recorded during autumn and winter at stations Sh 4 and Sh 6. The minimum value was recorded at the Sh 1.

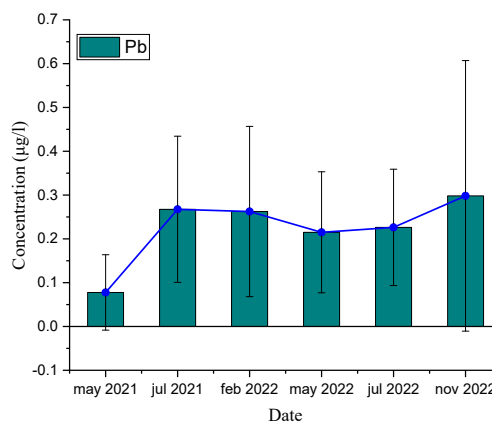


Fig. 7. Average values and standard deviation for Pb.

In contrast to the results of Fe and Mn, copper levels showed a similar trend as Pb, where its values increased during autumn more than spring and summer, which is mainly attributed to precipitation of copper in sediments as a cone at altitude of temperature [5]. The highest value (9.2 µg/L) was recorded during during November 2022 at station Sh 8, while the minimum (0.018 µg/L) was recorded during summer at station Sh 1. The obtained copper results showed a slight increase during the winter and autumn seasons more than during the summer season.

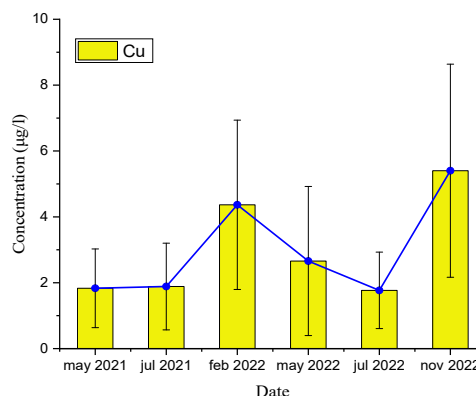


Fig. 8. Average values and standard deviation for Cu.

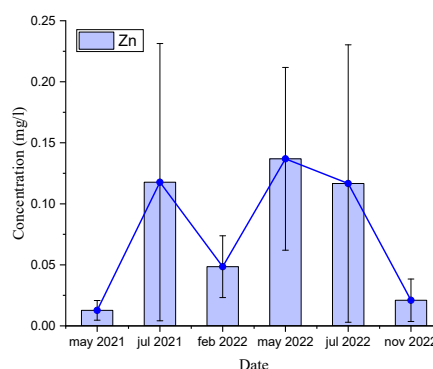


Fig. 9. Average values and standard deviation for Zn.

The decrease in zinc levels during spring and autumn is due to its sedimentation [4], as well as uptake by macrophytes and its adsorption on silt and clay particles. If the amount of organic matter is high, the content of Zn in the water is small as this metal has a high affinity for humic acids, this also explains the low values of Zn that were recorded in the stations of the lower part of Shkumbin. The highest value of zinc (0.3 mg/L) was recorded at station Sh 2 in July 2022 and the lowest value was recorded at stations Sh 1 during May 2021.

The results obtained regarding the content of heavy metals were treated statistically with the one way ANOVA method to judge if there are significant differences between the results obtained regarding the content of each metal between the stations as well as in different seasons. Regarding comparing the F-values and coefficient of variance was aiming the evaluation of the differences in metals levels between months and stations. Results are presented in Table 2.

Table 2. The Anova analyses

	Fe		Mn		Zn		Cu		Cr		Pb		Cd		Ni	
	<i>F_{val}</i>	<i>C. Var</i>	<i>F_{val}</i>	<i>C. Var</i>	<i>F_{val}</i>	<i>C. Var</i>	<i>F_{val}</i>	<i>C. Var</i>	<i>F_{val}</i>	<i>C. Var</i>	<i>F_{val}</i>	<i>C. Var</i>	<i>F_{val}</i>	<i>C. Var</i>	<i>F_{val}</i>	<i>C. Var</i>
Months	1.81	0.51	10.73	0.56	4.40	0.97	4.27	0.71	3.39	0.80	1.42	0.82	0.84	1.31	1.62	0.60
Stations	6.85	0.38	0.65	0.82	1.89	1.07	4.97	0.65	5.50	0.70	8.36	1.44	11.3	0.82	5.25	0.49

F_{val}: variation between sample means / variation within the samples; Coefficient Variation (*C. Var*): the extent of variability in relation to the mean of the population, widely used in analytical chemistry to express the precision and repeatability of an assay

Table 3 presents the results obtained in relation to the values of the correlation coefficients for the metals under study. According to this table, there is no visible correlation between the elements since the highest value is reached between Pb and Cr, Cu, Ni with correlation coefficients of 0.75, 0.65, 0.61, 0.56, respectively; and between Cr and Cu with a correlation coefficient of 0.95. The high correlation values, $r > 0.5$, are related to the same origin of the correlating elements as well as to the chemical or physical processes that these metals undergo during their stay in water, such as the formation of soluble complexes with the same ligands, sedimentation processes, connection with the matter in suspension, etc. The presence of metals in water also depends on other factors such as pH, water redox potential, etc.

Cadmium presence in water is dominated by soluble forms, except for cases where the concentration of particles in suspension is high, which reduce its concentration in water as an ionic form. If the amount of organic matter is high, the content of Zn in the water is small since this metal has a high affinity for humic acids.

Meanwhile, a negative correlation of Fe with Cd, is observed. This is related to the fact of the origin of the metals in the waters of the Shkumbini river. As we can see, Fe, which is known mainly as a metal of natural origin, exhibits a low correlation with metals that are usually of anthropogenic origin.

Table 3. The correlations of heavy metals on stations

Metal	Fe	Mn	Zn	Cu	Cr	Pb	Cd	Ni
Mn	0.40							
Zn	0.35	0.29						
Cu	0.24	0.31	0.09					
Cr	0.22	0.44	0.03	0.95				
Pb	0.24	0.29	0.38	0.90	0.70			
Cd	-0.11	0.28	0.16	0.75	0.56	0.65		
Ni	0.85	0.63	0.52	0.59	0.42	0.61	0.27	

These heavy metals evidenced in the waters of the Shkumbini River, as well as the presence of other elements in them, in smaller concentrations, cause them to accumulate in the soil with the irrigation water, causing its and groundwater pollution. In this way, these heavy elements potentially pose a risk for their transfer to agricultural plants and then to other links in the food chain.

The impacts resulting from the use of the polluted waters of the Shkumbin River are very important, especially in those areas, where the predominance of clay soils, the accumulation of heavy metals is favored by colloidal fractions. Fortunately, in the study area, predominate the alkaline conditions of soils and mobility of heavy metals is low, influencing the precipitation of heavy metals and reducing their availability for plants. The activity of trace elements in the aquatic system, besides factors such as pH, redox potential, etc., also depends on the identity of the element.

IV. DISCUSSIONS

The content of Fe in all the stations under study was several times higher than the levels recommended by UNECE (>600 µg/L) [11, 13]. The content of Fe in each year was about 3 times higher than the maximum value allowed. This is mainly related to the discharges of the mineral industry that occur in this river mainly from the mineral smelting and processing plant in Elbasan, Albania.

Manganese content in all the stations under study was several times higher than the levels recommended by UNECE (>150 µg/L) [11, 13].

The content of Ni was lower during the springs in almost all the stations. This is also related to the mineralogical composition of the region of iron-bearing minerals and the levels through which the river Shkumbin passes. In iron ores, the nickel content varies from 0.9%–1% Ni. Nickel content in all the stations under study was several times higher than the levels recommended by UNECE (>120 µg/L) [11, 13].

The chromium content in all the stations under study was several times higher than the levels recommended by UNECE (1–6; 6–11 µg/L). The cadmium content in all the stations under study was higher than the levels recommended by UNECE (0.07–0.53 µg/L). The lead content in all the stations under study was higher than the levels recommended by UNECE 0.1–1.6 µg/L.

The copper content in stations Sh 4, Sh 5 and Sh 6 was higher than the levels recommended by UNECE (>2 µg/L). The zinc content in all the stations under study was higher than the levels recommended by UNECE (>120 µg/L), [11, 13].

Significant variations were found between stations for metals Cd and Fe. Seasonal variations of heavy metals were

found significant for almost all metals studied, in all stations, except Mn. This is related to the fact that rivers are systems in continuous flow and the content of elements is expected to be approximate at different points in a given moment of time. The ANOVA analysis has shown that there are significant changes in the content of iron in different seasons, since the content of iron depends on many factors such as temperature, pH level of flows, precipitation, assimilation by biota, etc.

The content of heavy metals in the waters of the nearby Shkumbini River resulted in the following order: Fe> Ni> Cu> Cr> Cd> Pb> Mn> Zn.

A. Clusters Analysis Method

The study shows the variance for two components, on 8 monitoring stations and 8 variables were discriminated into three different groups, which were also associated with the industrial, urban, mountainous area and agricultural area. Table 4 shows there were the significant differences in the heavy metals among the different groups. CA1 group is composed of sampling points St1 and St2, with 36 samples, CA2 group is composed of sampling points St3, St4 and St5, with 54 samples. CA3 group is composed of sampling points St6, St7 and St8, with 54 samples.

The CA1 group was located in a rural mountainous area, which were rarely impacted by human activities. The CA2 group was located in towns in the lower reaches of the river basin, or in the plains where the mining industry was relatively developed. The towns along the Shkumbini tributary are relatively developed, and the wastewater from human activities is directly discharged into the river. The results also suggest that slightly polluted rivers have the ability to self-clean. Therefore, the degradation in water quality was caused by anthropogenic influences, such as the mining and processing of the steal combine production in the Shkumbini tributary. The average contents of Fe and Ni were the highest among the three clusters, which were 6.31mg/L, and 5.35µg/L, respectively. This is because the river flows through an iron-nickel rich area. The contents of Cu and Cr in the CA2 group were was the highest, with average contents of 4.82 µg/l and 3.87 µg/L, respectively. This is because the river in this area passes an area where the iron and ferrochrome processing industry is concentrated. The CA3 group was located in a rural area with flat terrain, and human activities were mainly associated with agricultural production.

Table 4. Concentrations, statistics and analysis test of three clusters

	Clusters															
	CA1 (n=36)				CA2 (n=54)						CA3 (n=54)					
	St1		St2		St3		St4		St5		St6		St7		St8	
	Mean	STD	Mean	STD	Mean	STD	Mean	STD	Mean	STD	Mean	STD	Mean	STD	Mean	STD
Fe	2.16	1.37	4.10	1.52	3.96	0.73	6.10	2.45	4.96	1.60	3.42	0.84	2.78	1.13	1.56	0.44
Mn	0.11	0.15	0.13	0.11	0.20	0.15	0.16	0.10	0.12	0.10	0.19	0.15	0.15	0.11	0.10	0.10
Zn	0.04	0.07	0.15	0.13	0.06	0.06	0.05	0.06	0.10	0.08	0.14	0.11	0.05	0.03	0.02	0.01
Cu	0.09	0.08	0.09	0.07	2.43	1.97	3.53	1.70	4.82	1.91	4.65	1.61	4.48	2.52	3.01	3.21
Cr	0.03	0.03	0.21	0.08	2.55	2.16	4.31	2.41	3.87	2.39	5.54	2.35	4.52	2.62	3.07	2.52
Pb	0.10	0.07	0.10	0.07	0.32	0.22	0.41	0.22	0.51	0.17	0.27	0.08	0.22	0.13	0.30	0.31
Cd	2.5·10 ³	3·10 ³	2.5·10 ²	7·10 ³	0.11	0.07	0.05	0.02	0.30	0.20	0.41	0.22	0.61	0.34	0.07	0.05
Ni	1.27	0.58	3.58	2.08	4.47	1.92	5.35	2.18	5.53	2.60	4.93	1.87	3.14	1.16	1.37	1.07

B. The Principal Component Analysis

The study found the Principal Component (PC1) explained 46.5% of the variance variation, in which Fe and Ni had strong positive loads (Table 5). This is because the river passes through an area rich in nickel and iron. The Principal Component (PC2) explained 73.1% of the variance differences, in which the Pb and Zn had positive and negative loads, but also had good correlation, and the Cu had positive loads. The Principal Component (PC3) explained 85% of variance differentiation, in which Fe and Pb had the strong and medium positive loads. These are mainly originated from urban and industrial activities, such as mining, metal

smelting and refining, waste inceneration The Principal Component (PC4) explained 93.3% of variance differences, in which the Mn had strong positive loads. The Principal Component (PC5) explained 98.9% of variance in differences, where the Fe and Cd had moderate positive loads. The Principal Component (PC6) explained 99.6% of variance differences, in which Zn and Cr had positive loads. The Principal Component (PC7) and (PC8) explained 99% of variance differences, in which Fe, Cu, Pb and Cd had the positive loads, could be attributed to a mixture of anthropogenic, domestic sewage, petrogenic and agricultural inorganic fertilizers.

Table 5. Principal component analysis of source contributed and eigenanalysis of correlation matrix

Variable	PC1	PC2	PC3	PC4	PC5	PC6	PC7	PC8
Fe	0.344	-0.340	0.515	-0.065	0.333	-0.039	0.549	0.289
Mn	0.374	-0.129	-0.096	0.641	-0.598	-0.149	0.108	0.181
Zn	0.237	-0.365	-0.343	-0.685	-0.425	0.122	0.046	0.163
Cu	0.385	0.441	0.113	-0.126	0.110	-0.078	-0.483	0.614
Cr	0.400	0.411	0.112	-0.006	-0.105	0.718	0.180	-0.316
Pb	-0.235	0.454	0.433	-0.290	-0.497	-0.366	0.289	-0.049
Cd	0.315	0.359	-0.564	-0.058	0.286	-0.419	0.417	-0.140
Ni	0.472	-0.188	0.279	-0.113	0.006	-0.362	-0.402	-0.599
Eigenvalues	3.7233	2.1237	0.952	0.6672	0.4494	0.0525	0.0318	-0.0000
Proportions	0.465	0.265	0.119	0.083	0.056	0.007	0.004	-0.000
Cumulative %	46.5	73.1	85.0	93.3	98.9	99.6	99.0	1.000

The results of spatial principal component analysis and correlation analysis in the Shkumbini river showed that the heavy metals of the river water varied with different clusters due to the variation in the environment, such as geological sections, anthropogenic and industrial activities.

V. CONCLUSIONS

This study provides information on heavy metal levels in the water of the Shkumbini River of Albania, over a two-year sampling period. The relative content of heavy metals in water is in the order: Fe > Ni > Cu > Cr > Cd > Pb > Mn > Zn. It should be reiterated that monitoring shows that these waters are considered polluted, based on EU standards. The level of heavy metals in the Shkumbini River water is highly dependent on the location of the sampling station as well as the sampling time. Significant variations were found between stations for metals Cd and Fe. According to the obtained results, the level of metals studied in the water of the Shkumbini River is below the level required for their presence in the surface water. The results of this study may provide useful information to better understand the transport and fate of heavy metals in the Shkumbini River water.

The monitoring and the results obtained show that the content of iron, manganese, lead, nickel, mercury and copper are quite high for stations Sh 4, Sh 5, Sh 6, Sh 7 and Sh 8, above the limits allowed, classifying these waters polluted.

The content of iron, copper has been outside the defined limits. Zinc has resulted in content within the limits, except for station Sh 1. Nickel is slightly above the defined limits. In the water of Shkumbini river, the content of manganese has resulted above the defined limits, as well as the content of cadmium and lead.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

AUTHOR CONTRIBUTIONS

All authors have contributed in sampling, analysis, and site inspection. All authors read and agreed to the final version of paper.

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