



Evaluation of heavy metal contamination in soil samples in the region of Mitrovica

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Abstract:

The industry is crucial for the economic development of the country, but at the same time, it is one of the biggest influences on the environment. The Mitrovica region faces numerous environmental problems due to the mining-industrial processes applied in the facilities of the industrial complex "Trepça". As a result of these processes, industrial waste with a toxic composition is created, which is considered a potential polluter of soil, air, water, and vegetation in this area. The study aims to determine the concentration of heavy metals including Pb, As, Zn, Ni, Co, Cr, Cd, and Cu in soil samples. Also, the level of soil pollution with heavy metals was examined, using pollution indicators, such as contamination factor (CF), degree of contamination (Cd), modified degree of contamination (mCd), pollution load index (PLI), and geo-accumulation index (Igeo). To carry out this research, we selected 6 sampling sites in the period May - August 2024, which were characterized and evaluated as the most critical points. The concentration of heavy metals in soil samples was determined using inductively coupled plasma-optical emission spectrometry (ICP-OES Optima 2100 DV, Perkin-Elmer) by standard method US EPA 6010C. From the obtained results it was found that the level of heavy metals analyzed in this work, respectively the level of zinc (197.84 - 3456.14 mgkg⁻¹), lead (204.75 - 3458.16 mgkg⁻¹), and arsenic (30.76 - 634.56 mgkg⁻¹) is relatively high and exceeds the allowed values recommended by Kosovo and European Legislation.

1. Introduction

The rapid development of urbanization and industrial activities around the world has caused great environmental degradation and disruption of the ecological balance [1,2]. The pollution of the soil ecosystem by heavy metals is a global problem, including in our country Kosovo. Heavy metals should be given special importance and attention because, unlike other environmental pollutants, they are considered systemic toxicants, resistant to biodegradation, and known to cause multiple organ damage, even at lower exposure levels [3,4,5]. Numerous studies have proven that heavy metals accumulated in the soil can infiltrate into underground water and aquifers, can also

threaten the entire food chain so that they can pose a serious threat to human health [6, 7].

Trepça Mine is an ancient source of lead, zinc, silver, crystals, and various minerals in Kosovo. The mining-industrial processes applied in the wards of the industrial complex "Trepça" have resulted in enormous environmental pollution. In addition to the metallurgical and chemical working activities of these departments, industrial waste has also been created, which is considered a potential polluter of the soil, air, water, and vegetation in this region [8].

The study focuses on the research and assessment of the environmental condition of the soil in this region by selecting locations close to the industrial area where there are open dumps of industrial

waste. It has been established that activities related to the production of lead and zinc generally lead to significant pollution in the surrounding areas [9]. The limits of heavy metals in soil have been determined through various standard methods of digestion based on the total concentrations of metals. In the assessment of soil pollution caused by heavy metals, different pollution indicators are used: such as the pollution load index (PLI), the geo-accumulation index (Igeo), and the enrichment factor (EF) [10,11,12].

The aim of the present work is: a) to determine levels and distribution of toxic heavy metals in soil samples in selected locations; b) to explore the extent of pollution and impacts of pollution using the following pollution indicators such as contamination factor (CF), contamination degree (Cd), modified pollution degree (mCd), pollution load index (PLI) and geo-accumulation index (Igeo); c) to suggest a regular monitoring network work in accordance with proper land management.

2. Material and Methods

2.1 Study area

In the north of Kosovo, respectively in the Mitrovica region, there is the "Trepça" Industrial Complex. As a result of the mining and industrial processes that have been applied and continue to be applied, have resulted in enormous pollution of air, soil, water, and plants with heavy metals [13,14]. To determine the level of soil pollution with heavy metals in our research area, a total of six sampling locations were collected during the period from May to August 2024. Each sample site presents special characteristics that correspond to the purpose of our research. Numerical coordinates were measured at each sampling location with a Garmin 35X GPS device. The ArcGis program was used to create the map, as well as the conversion of numerical coordinates into geographic ones. Figure 1 presents the positions of the sampling sites.

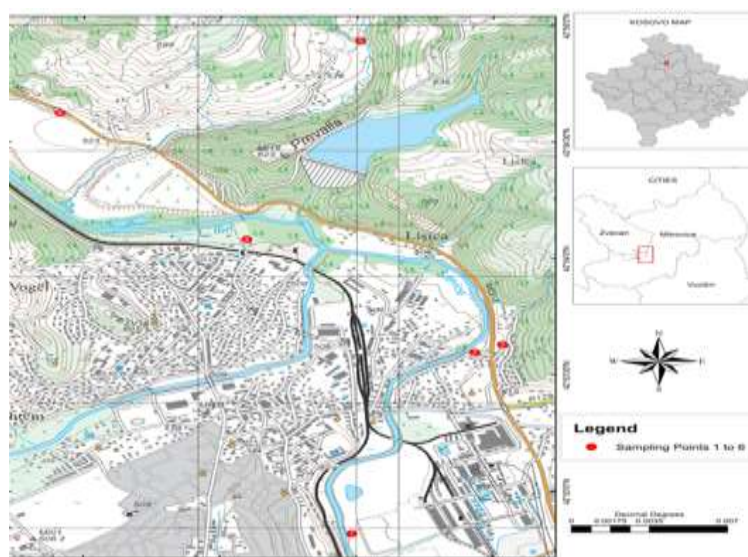


Figure 1. Sampling sites

2.2 Soil sampling, preparation and chemical analysis

The planning, method, and tools for taking soil samples, the amount of the sample, conservation for determining individual parameters, as well as the method of transportation to the laboratory, are done in accordance with the standard ISO 11464 (2006) [15]. After sending to the laboratories, the samples were placed in a drying container and placed in a thermostat for 3 - 4 hours at a temperature of 105°C. First, the sample is ground to obtain particles smaller than 2 mm, then grinding is continued in a ball mill until particles smaller than 75 µ are obtained. After grinding and homogenizing the sample, 0.5 grams of each sample is weighed on an analytical

balance, where it is treated according to the EPA 3052 method, according to which 7.5 ml of HCl and 2.5 ml of HNO₃ are added to each sample [16]. In this case, these soil samples are ready for microwave digestion-mineralization (model: BERGHOF Speedwave MWS-3+). After mineralization, the samples were filtered and concentrations of Pb, As, Zn, Ni, Co, Cr, Cd, and Cu in these soil samples were determined using optical emission spectrometry with inductively coupled plasma (ICP-OES Optima 2100 DV) by EPA standard method 6010 C [17].

Also, in the soil samples, the pH value was measured according to ISO Standard 10390 (2005), using Eutech Instruments pH 1500 [18].

All experiments were done in triplicate, data are presented as mean and standard deviation and were calculated using Microsoft Office Excel 2023. Pearson's r correlation matrix and Scatter Plot Matrix (using OriginPro statistical software) were used to clarify the data to find the relationship between heavy metals in soil samples in Mitrovica region.

2.3 Determination of Contamination Indicators

Several methods of pollution indicators are used to assess the degree of soil contamination with heavy metals, such as contamination factor (CF), degree of contamination (Cd), modified degree of contamination (mCd), pollution load index (PLI), and geo-accumulation index (Igeo).

2.3.1 Contamination factor (CF)

The contamination factor is used to estimate heavy metal contamination in the respective samples [19], the (Eq. 1) used to calculate the pollution factor is:

$$CF = C_{sample}^i / C_{reference}^i \quad (1)$$

where:

CF – is the contamination factor for heavy metal,
 C_{sample}^i – is the measured value of the heavy metal in the soil [$mg \cdot kg^{-1}$],

$C_{reference}^i$ is the parameter for calculation, regarding the background values for heavy metals in soil recommended by Administrative Instruction No.11/2018 on Limited Values of Emissions of Polluted Materials into Soil [$mg \cdot kg^{-1}$] [20].

The following classification is used to describe the contamination factor: $CF < 1$, low contamination factor; $1 \leq CF < 3$, moderate contamination factors; $3 \leq CF < 6$, high contamination factors; and $CF \geq 6$, very high contamination factor [21].

2.3.2 Degree of contamination and modified degree of contamination

According to Özkan (2012), the degree of pollution (Cd) is defined as the sum of all contamination factors of selected heavy metals, therefore it is calculated using the (Eq. 2) [22]:

$$Cd = \sum_{i=1}^{n=8} CF_i \quad (2)$$

The classification proposed by Håkanson is used to describe the contamination degree for analyzed elements: $Cd < 6$: low contamination degree, $6 \leq Cd < 12$: moderate contamination degree; $12 \leq Cd < 24$:

considerable contamination degree; $Cd \geq 24$: very high contamination degree [23].

The modified degree of contamination (mCd) is calculated based on the (Eq. 3) proposed by Abraham and Parker.

$$mCd = \frac{\sum_{i=1}^{n=8} CF_i}{n} \quad (3)$$

Also, they proposed the classification of this soil pollution indicator: $mCd < 1.5$, Nil to very low degree of contamination; $1.5 \leq mCd < 2$, Low degree of contamination; $2 \leq mCd < 4$, Moderate degree of contamination; $4 \leq mCd < 8$, High degree of contamination; $8 \leq mCd < 16$, Very high degree of contamination; $16 \leq mCd < 32$, Extremely high degree of contamination; $mCd \geq 32$, Ultrahigh degree of contamination [24].

2.3.3 Pollution Load Index (PLI)

The pollution load index (PLI) is defined as the n^{th} root of the product of the n pollution factor (CF). Based on this index, the degree of soil contamination with heavy metals can be estimated. The pollution load index is calculated using the Tomlinson equation [25]. This indicator is calculated using the following (Eq. 4):

$$PLI = (CF_1 \cdot CF_2 \cdot CF_3 \cdot \dots \cdot CF_n)^{\frac{1}{n}} \quad (4)$$

where:

CF – pollution factor,

n - number of analyzed elements (n = 8 in this study).

A value of $PLI > 1$ means contamination, while a value of $PLI < 1$ means no contamination [19].

2.3.4 Geoaccumulation index (Igeo)

The geoaccumulation index was first proposed by Müller and is still used by other researchers to assess the degree of soil pollution by heavy metals [21,26]. The equation (Eq. 5) is given for calculating this index:

$$I_{geo} = \log_2 \frac{C_n}{1.5 \cdot B_n} \quad (5)$$

where:

Factor 1.5 - to minimize the effect of variation of reference values,

C_n - measured metal concentration (n) in the soil,
 B_n : reference value of the corresponding metal (n).

Classification of the geo-accumulation index and description of soil quality proposed by Müller [21], is presented in Table 1.

Table 1. Classification of geo-accumulation index and description of soil quality

Igeo value	Igeo class	Designation of sediment quality
>5	6	Extremely contaminated
4-5	5	Strongly to extremely contaminated
3-4	4	Strongly contaminated
2-3	3	Moderately to strongly contaminated
1-2	2	Moderately contaminated
0-1	1	Uncontaminated to moderately contaminated
≤0	0	Uncontaminated

3. Results and Discussions

Soil samples were taken at six locations to examine the level of heavy metal pollution in the Mitrovica region. Table 2 and Figure. 2 shows the concentration values of heavy metals in soil samples, the average concentrations and standard deviation are also calculated. Based on the results obtained from the analysis of soil samples, it was proven that the concentration of Lead, Arsenic, Zinc, Cobalt, Chromium, Cadmium, and Copper is quite high, exceeding the maximum permissible values recommended by Administrative Instruction No. 11/2018 and Council Directive 86/278/EEC on the protection of the environment, and in particular of the soil [20,27]. The following results were observed: Pb (78.64 – 3458.16 mg kg⁻¹), As (30.76 – 757.14 mg kg⁻¹), Zn (197.84 – 3456.14 mg kg⁻¹), Ni (47.48 – 205.68 mg kg⁻¹), Co (72.18 – 278.58 mg kg⁻¹), Cr (53.19 – 219.34 mg kg⁻¹),

Cd (8.78 – 97.84 mg kg⁻¹), Cu (60.79 – 537.12 mg kg⁻¹). Higher values of the concentration of metals have resulted in the samples that were taken in the vicinity of the area of the industrial waste dumps, respectively in S1, S4, and S6. Therefore, the open landfill of industrial waste affected by unsuitable meteorological conditions has a significant effect on increasing the concentration of heavy metals in the environment of the Mitrovica region [8]. Also, the pH in the soil samples measured in the sampling sites has shown different values, from 3.4 to 7.2. Lower pH values in soil samples were recorded in the vicinity of industrial landfills, respectively in S1, S4, and S6 (Table 2). High acidity in soil can have two harmful consequences: inhibiting the activity of bacteria that cause microbial degradation of organic pollutants and increasing the mobility and bioassimilation of many divalent metal cations [28]. The contamination factor (CF) was calculated as

Table 2. The average concentrations of heavy metal in soil samples, pH values, and limited values (concentration unit is in mgkg⁻¹ dry weight)

Samples (mgkg ⁻¹)	Pb	As	Zn	Ni	Co	Cr	Cd	Cu	pH
S1	1034.24 ±0.07	634.56 ±0.32	3456.14 ±0.59	205.68 ±0.27	175.14 ±0.02	197.21 ±0.35	52.34 ±0.48	537.12 ±0.57	4.5 ±0.03
S2	538.79 ±0.09	214.64 ±0.36	1037.65 ±0.39	86.47 ±0.39	99.78 ±0.18	84.75 ±0.29	24.57 ±0.82	148.28 ±0.24	6.7 ±0.05
S3	78.64 ±0.13	30.76 ±0.67	197.84 ±0.29	56.19 ±0.85	72.18 ±0.91	53.19 ±0.09	10.49 ±0.39	97.54 ±0.12	6.8 ±0.02
S4	1753.43 ±0.25	405.34 ±0.68	1459.98 ±0.94	157.63 ±0.94	109.84 ±0.76	124.37 ±0.49	97.84 ±0.59	312.26 ±0.25	4.8 ±0.01
S5	204.75 ±0.56	76.53 ±0.85	245.96 ±0.53	47.48 ±0.39	84.56 ±0.42	92.15 ±0.19	8.78 ±0.27	60.79 ±0.24	7.2 ±0.06
S6	3458.16 ±0.31	757.14 ±0.40	2037.15 ±0.26	104.75 ±0.95	278.58 ±0.72	219.34 ±0.39	67.43 ±0.72	457.83 ±0.08	3.4 ±0.04
A. I. No.11/2018	200	30	300	300	20	300	3	200	/
86/278/EEC	50-300	20	150-300	30-75	50	50-100	1-3	50-140	/

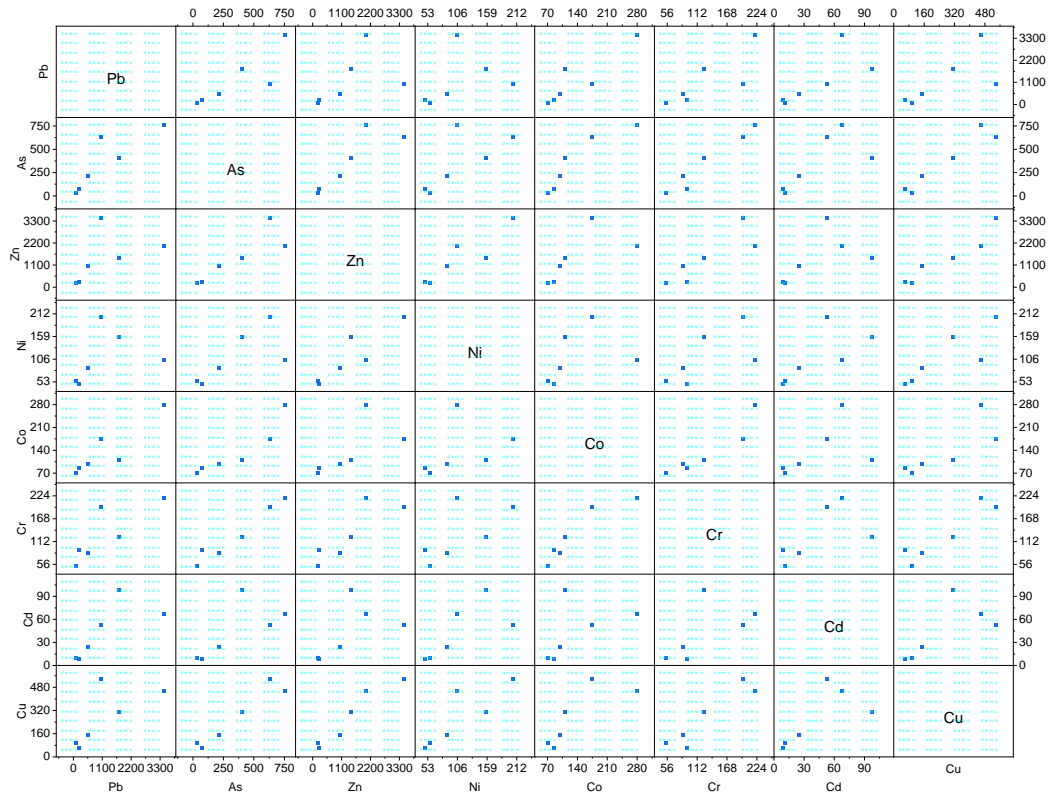


Figure 2. Scatter Plot Matrix of heavy metals in soil samples

Table 3. The values of the contamination factor (CF) of soil samples in Mitrovica region

CF	Pb	As	Zn	Ni	Co	Cr	Cd	Cu
S ₁	5.17	21.15	11.52	0.68	8.75	0.65	17.45	2.68
S ₂	2.69	7.15	3.45	0.28	4.98	0.28	8.19	0.74
S ₃	0.39	1.02	0.65	0.18	3.61	0.18	3.5	0.48
S ₄	8.76	13.51	4.86	0.52	5.49	0.41	32.61	1.56
S ₅	1.02	2.55	0.81	0.15	4.22	0.31	2.93	0.3
S ₆	17.29	25.23	6.79	0.35	13.93	0.73	22.47	2.29

the total concentration of metals regarding the national limit values for the content of heavy metals in soil [20]. Table 3 and Figure. 3 shows contamination factor (CF) values for heavy metals recorded in the selected sampling sites. The contamination factor resulted to be very high for As, Pb, Cd, and Zn followed by Co, while for Ni, Cr, and Cu the values are much lower. The values for As range from (1.02 – 25.23), Pb (0.39 – 17.29), Cd (2.93 – 32.61), and Zn (0.65 – 11.52). Using the classification proposed by Müller [21], the value of the contamination factor for the studied elements resulted mainly in very high levels of soil contamination in S₆, S₄, and S₁, respectively in the area near the lead smelter, near the landfill with industrial waste in Zvečan and Mitrovica Industrial Park. The values of mCd show that the anthropogenic factor has a significant impact on all study points (Table 4). Also, the values show a high

degree of soil contamination with the analyzed metals, especially in the S₆ sampling site, it turns out to be the highest value of this indicator with 11.13. According to the classification $8 \leq mCd < 16$, it turns out to be a point with a very high degree of soil contamination [24]. The Pollution Load Index (PLI) is another method we used to assess the level of soil pollution with heavy metals. The values of this indicator are presented in Table 4. Figure 4 is the Cd, mCd, and PLI in soil samples in Mitrovica region. According to Min [19], if the value of PLI is greater than 1, it means that metal pollution is present, while if the value is less than 1, then there is no metal contamination. So, from the values obtained for this indicator, it turns out that we have metal pollution with special emphasis on points S₁, S₄, and S₆. The geo-accumulation index (I_{geo}) is a method that evaluates the degree of metal pollution in seven classes (Table 1). The

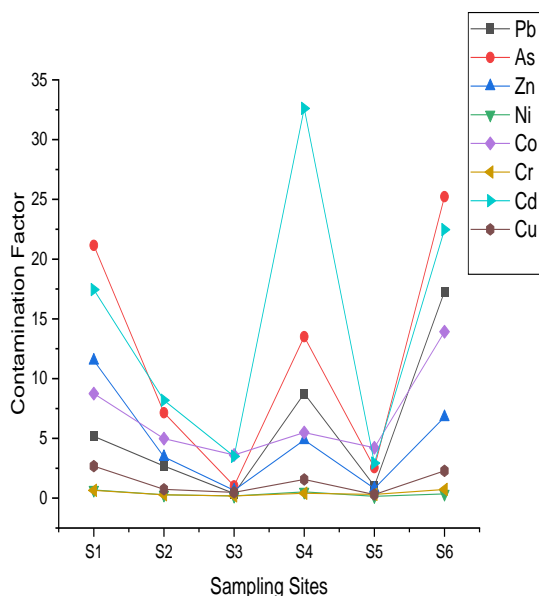


Figure 3. Contamination factor (CF) of heavy metals in soil samples

Table 4. The values of the Cd, mCd, and PLI of soil samples in Mitrovica region

Samples	Cd	mCd	PLI
S ₁	68.05	8.5	4.67
S ₂	27.76	3.47	1.88
S ₃	10.01	1.25	0.68
S ₄	67.72	8.46	3.68
S ₅	12.29	1.53	0.88
S ₆	89.08	11.13	5.21

sampling sites S₁, S₂, S₄, and S₆ belongs to class 6, which turns out to be extremely polluted soil with this heavy metal. The low values of this indicator (Igeo), for nickel (Ni), chromium (Cr), and copper (Cu), show that the concentration of these metals in the soil is low or there is very little metal pollution.

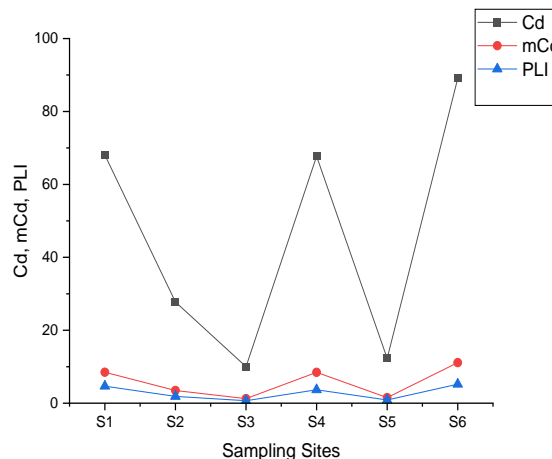


Figure 4. Cd, mCd, and PLI in soil samples in Mitrovica region

values of Igeo are presented in Table 5 and Figure 5. Based on Muller's classification, lead (Pb) in sampling site S₆ belongs to class 4 indicating extremely high contamination. Followed by sample site S₄, which belongs to class 3 with a medium to strong soiled level. Arsenic (As) based on the obtained Igeo values belongs to class 6, a strongly polluted class for sampling sites S₁, S₄, and S₆. For sampling location S₁, zinc (Zn) belongs to class 6, respectively extremely polluted classification, as well as location S₆ belongs to class 5, strongly to an extremely polluted classification. Cobalt (Co) in sampling sites S₁ and S₆ belongs to class 6, which is characterized by extreme soil contamination. Based on the results, we emphasize that cadmium (Cd) in

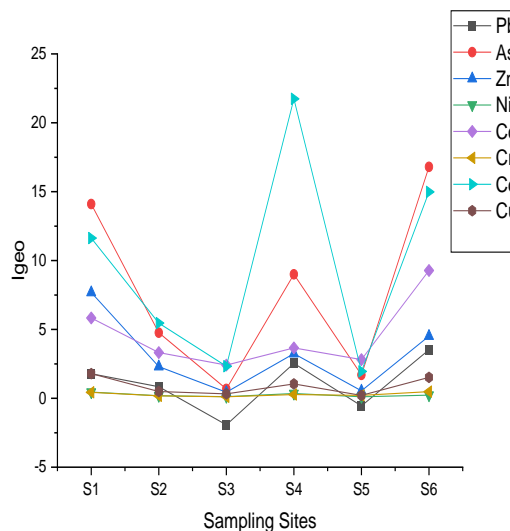


Figure 5. Index of geo-accumulation (Igeo) of heavy metals in soil samples

Table 5. Index of geo-accumulation (Igeo) of heavy metals in soil samples in Mitrovica region

Igeo	Pb	As	Zn	Ni	Co	Cr	Cd	Cu
S ₁	1.78	14.1	7.68	0.45	5.83	0.43	11.63	1.79
S ₂	0.84	4.76	2.3	0.19	3.32	0.18	5.46	0.49
S ₃	-1.93	0.68	0.44	0.12	2.41	0.11	2.33	0.32
S ₄	2.54	9	3.24	0.35	3.66	0.27	21.74	1.04
S ₅	-0.55	1.7	0.54	0.11	2.81	0.21	1.95	0.21
S ₆	3.52	16.8	4.52	0.23	9.28	0.48	14.98	1.52

Table 6. Pearson's correlation coefficient between heavy metals in soil samples

Pearson's r	Pb	As	Zn	Ni	Co	Cd	Cr	Cu
Pb	-	0.854	0.494	0.350	0.899	0.740	0.809	0.701
As	0.854	-	0.871	0.713	0.918	0.718	0.975	0.959
Zn	0.494	0.871	-	0.907	0.664	0.567	0.854	0.956
Ni	0.350	0.713	0.907	-	0.386	0.710	0.653	0.852
Co	0.899	0.918	0.664	0.386	-	0.500	0.928	0.803
Cd	0.740	0.718	0.567	0.710	0.500	-	0.609	0.696
Cr	0.809	0.975	0.854	0.653	0.928	0.609	-	0.932
Cu	0.701	0.959	0.956	0.852	0.803	0.696	0.932	-

In Table 6, we have presented Pearson's correlation matrix of heavy metals in soil samples. The significant correlations during the period from May to August 2024 between the analyzed parameters represent the expected correlation for all analyzed samples.

A reciprocal reflection of the correlation of Pb with a positive coefficient to the elements As, Co, Cd, Cr, and Cu, which implies such a relationship that it is explained that with the increase in the concentration of Pb, the concentrations of As, Co, Cd, Cr and they grow and conversely.

Another reciprocal reflection of the correlation with a negative coefficient is seen between the concentration of Pb with Zn and Ni, such a relationship can be explained that with an increase in the concentration of Pb, the concentration of Zn and Ni decreases and conversely.

The correlation of As has a positive coefficient with all the elements Pb, Zn, Ni, Co, Cd, Cr, and Cu. The correlation of Zn with a positive coefficient is with As, Ni, Cr, and Cu, while it has a moderate correlation coefficient with Cd and Co. At the same time, with Pb, there is a relationship with a negative coefficient. The correlation ratio of Ni with As, Zn, Cd, and Cu represents a positive correlation coefficient, while it has a negative correlation coefficient with Pb, Co, and Cr. Cobalt has a positive correlation coefficient with Pb, As, Zn, Cr, and Cu, while a negative correlation with Ni and Cd. The last elements Chromium and Copper are singled out because they have a positive correlation coefficient with all the elements found in the samples of the soil.

4. Conclusions

The region of Mitrovica has a long industrial history, especially in the exploitation and processing of minerals. These mining-industrial activities contribute to environmental pollution with heavy metals in this region. To examine the level of soil contamination with heavy metals, concentrations of Pb, As, Zn, Ni, Co, Cr, Cd, and Cu were analyzed at six sampling locations.

Measurements of the concentration of Pb, Zn, As, and Cd in soil samples show a high level of concentration, which exceeds the limit values of emissions of polluted materials into the soil recommended by Administrative Instruction No. 11/2018.

To assess the degree of soil contamination with heavy metals, the following pollution indicators were applied: pollution factor (CF), pollution degree (Cd), modified pollution degree (mCd), pollution load index (PLI), and geo-accumulation index (Igeo). Considering all assessment methods, it was found to be very high contamination for As, Pb, Cd, and Zn, followed by Co.

Statistical analysis of experimental data was performed using Pearson's correlation coefficient (r) to obtain relevant data on metal concentration in six soil samples collected in the Mitrovica region.

The results also show that there is a growing need for further research, mainly focused on the mechanisms of the rehabilitation process. The application of phytorehabilitation as a green, prospective, and sustainable method can offer an effective and natural solution for soil decontamination from various pollutants, including heavy metals. The plant *Salix purpurea* can play an important role in the remediation of soils with heavy metals. Our future research work will focus on the use of *Salix purpurea* for the remediation of soil contaminated with heavy metals, and the impact of this plant on the environment will be published in a future paper.

Author Statements:

- **Ethical approval:** The conducted research is not related to either human or animal use.
- **Conflict of interest:** The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper
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- **Data availability statement:** The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

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