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Heavy Metal Contamination and Accumulation in Soil and Plant from Mining Area of Mitrovica, Kosovo

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Abstract

This study assesses the total concentration of eight toxic metals As, Cd, Co, Cr, Cu, Ni, Pb, and Zn in the soil and plant of *Salix purpurea* samples collected from Mitrovica mining region in Kosovo. The concentration, accumulation and transfer from soil to roots, stem and leaves were assessed using bioconcentration factor (*BCF*) and translocation factor (*TF*). The total metal content in the soil and plant was high following the trend of Zn > Pb > Ni > Cd > Cu > As > Co > Cr. The relevance between soil and accumulation of heavy metals in *Salix purpurea* was assessed using correlation matrix and principal components analysis. The results indicate that *Salix Purpurea* can be used in phytoremediation and stabilization of soil contaminated by heavy metals.

Keywords Heavy metals · *Salix purpurea* · Bioconcentration · Translocation factors

The presence of mining waste, originating from the zinc extraction industry in Kosovo, is considered hazardous due to the presence and the mobility of toxic metals that it contains (Deconta 2009; Frese et al. 2004; Kerolli Mustafa et al. 2015a, 2015b; Rydergren and Montelius 2004; Zoi 2010). Its open disposal in many tailing damps close to the urban areas has become a major environmental concern. Contamination of urban lands and other natural habitats by heavy metals has become a severe hazard to the environments (Huszar et al. 2020). Among various heavy metal contaminants, cadmium (Cd), lead (Pb), zink (Zn) and arsenic (As) are identified as some of the most significant pollutants due to their strong bio-toxicity and high transfer risk (Houri et al. 2020). The dependence between metal concentrations in the soil and plants was observed in individual plant species due to the different degrees of absorption observed in individual plant species (Mleczek et al. 2009). The willow material was mainly used to assess the levels of heavy metal pollution (Mleczek et al. 2009). The presence and the form of metals in soil depend on the chemical forms and the affinity to influence their reactivity and mobility (Brininstool 2010; Caporale and Violante 2016). Thus, the presence of metals in the soil also defines their possible transfer into water or plants, known as "bioavailability". Therefore, plants remain an important indicator in determining the bioavailability of heavy metals and pollution in a particular area. According to Rosselli et al. (2003), different genotypes of the same species uptake different amounts of heavy metals. If plants accumulate > 1000 or > 10,000 mg kg^{-1} of metals are categorized as metals hyperaccumulators (Yoon et al. 2006). Researchers report that metal and plant interact in a very specific way that is linked with soil type, plant, growth conditions and the presence of other ions (Yoon et al. 2006; Rosselli at al. 2003; Caporale and Violante 2016). Several methods such as bioaccumulation, translocation, principle component analysis and correlation matrix have been used to assess soil and plant contamination levels with heavy metals (Kerolli Mustafa et al. 2015b). The purpose of this study is to assess the heavy metal pollution levels of soil and plants in the vicinity of mining area in Mitrovica, Kosovo by applying the following methods such as the bioconcentration factor (*BCF*), translocation factor (*TF*),

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principal component analysis and correlation matrix, and to make a comparison of the obtained result with reference limited values and other research outcomes (Sijakova-Ivanova et al. 2017).

Materials and Methods

Six sampling locations were chosen for our study situated in the Mitrovica region in Kosovo close to the Trepca mining complex. The selected area is comprised of industrial (S1, S2, S3, S5), vegetation (S4) and residential areas (S3, S6) (Fig. 1).

Six soil samples at 20 cm depth were collected with a hand soil sample auger allowing reaching depth. Samples were air-dried at room temperature, sieved through a 2 mm size sieve, mixed and homogenized (from 0 to 20 cm) using coning and quartering method and stored in polyethylene containers until analyzed. Soil and plant samples were analysed was following guidelines from the ICARDA's manual (Estefan et al. 2013) and Sijakova et al. (2017). Willow material (six samples of *Salix purpurea*) was collected from June 2018 to October 2019. Determination of elements (As, Cd, Co, Cr, Cu, Ni, Pb, and Zn) in soil samples was carried out using the assisted microwave digestion method (HCl/HF/HNO_3/H_3BO_3) followed by dissolution in aqua regia (HCl: $HNO_3 = 3:1$). A triacid wet digestion method was used to analyse the heavy metal content in plants. The means of element content in soil and plant samples were determined using inductively coupled plasma—optical emission spectrometry (ICP–OES Optima 2100 DV, Perkin-Elmer). Certified reference materials (S JR-3 and S Jsy-1) for soil and the NIST 1575a (Pine Needles) and NCS DC 73,350 (Leaves of Poplar) for plants were analyzed to test the accuracy of the applied method for the determination of total metal concentrations in investigated soil and plants samples (Table 1).

The means and standard deviations (*SD*) for the calculated results were calculated using Microsoft Office Excel 2019. The pH value in soil samples was measured following ISO 10,390 standard (2005). The background values and thresholds for heavy metals are used from *Administrative Instruction No. 11/2018 from Kosovo, w*hile Vamerali et al. (2010) and Yanitch et al (2017) are used for heavy metals thresholds in plants. To evaluate the metal accumulation efficiency in plants the bioconcentration factor (*BCF*) and translocation factor (*TF*) were calculated. As per Wu et al. (2008) the *BCF* is the ratio of the metal concentration in the roots to that in soil:

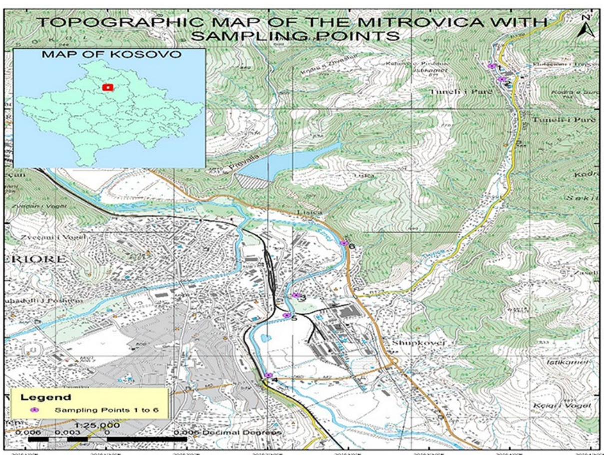

Fig. 1 Sampling location in Mitrovica Region, Kosovo

Table 1 Results of total metal content for certified reference material S JR-3, S Jsy-1, SRM 1575, NCS 73,350

CRM		S JR-3		S Jsy-1	
Elements	Unit	Certified value	Measured value	Certified value	Measured value
Pb	mg/kg	52.8 ± .5	50.04 ± 3	149 ± 10	148,05 ± 10
Zn	mg/kg	209 ± 3	200.89 ± 7	3.8 ± 0.9	3.79 ± 0.5
Cr	mg/kg	3.5 ± 0.4	3.49 ± 0.3	2 ± 0.2	2.55 ± 0.4
Cu	mg/kg	12.9 ± 1.2	12.86 ± 1.5	3.3 ± 0.7	3.12 ± 0.3
Co	mg/kg	21.1 ± 0.104	21.05 ± 1.01	12.1 ± 0.202	11.57 ± 0.9
As	mg/kg	1.1 ± 0.01	1.105 ± 0.101	0.90 ± 0.2	0.85 ± 0.1
CRM		SRM 1575		NCS 73,350	
Pb	mg/kg	10.8 ± 0.5	9.84 ± 0.03	1.5 ± 0.3	1.47 ± 0.11
Zn	mg/kg	65 ± 10	65.76 ± 05	37 ± 3	37.9 ± 2.25
Cr	mg/kg	2.6 ± 0.2	2.53 ± 0.5	0.55 ± 0.07	0.52 ± 0.05
Cu	mg/kg	3.0 ± 0.3	3.55 ± 04	6.3 ± 1.0	6.5 ± 0.07
Co	mg/kg	0.1	0.109 ± 0.01	0.42 ± 0.03	0.44 ± 0.06
As	mg/kg	0.21 ± 0.04	0.20 ± 0.1	0.37 ± 0.06	0.38 ± 004

BCF = metal concentration in plant root (mg kg^{-1})/metal concentration in soil (mg kg^{-1}).

while *TF* is the ration of metal concentration in the stem to the roots:

TF = metal concentration in plant stem (mg kg^{-1}) / metal concentration in plant roots (mg kg^{-1}).

If plants have *BCF* and *TF* greater than one they have the potential to be used for phytoextraction. In this study, response surface plots concentration characteristics, Pearson's correlation matrix r and principal components analysis (using Minitab 19 Statistical Software) was used to elucidate the data to find the relationships between heavy metals in soil, roots, leaves, stem from the Mitrovica Region (Kerolli Mustafa et al. 2015a, b). Statistical comparison of obtained data was performed by two-paired *t*-test at a significance level of $p = 0.05$. A calculated *t*-value of 0.432 was lower than a critical value of 2.25 which implies the two sets of values were not significantly different.

Results and Discussions

The results of heavy metal concentration in soil and plant accumulation patterns of selected metals (As, Cd, Co, Cr, Cu, Ni, Pb, and Zn) are presented in Table 2. Mostly, the higher uptake of metals from the soil and plant *Salix purpurea* was recorded in the area close to mining flotation and Mitrovica Industrial Park. The mean of total contents of eight heavy metals in all six soil samples were found in the range of: As 48.91 – 881.26 $mgkg^{-1}$, Cd 5.48–238.57 $mgkg^{-1}$, Co 17.83–31.25 $mgkg^{-1}$, Cr 34.98–168.56 $mgkg^{-1}$, Cu 58.93–943.8 $mgkg^{-1}$, Ni 82.86–282.33 $mgkg^{-1}$, Pb 359.26–4662.22 $mgkg^{-1}$ and Zn 386.21–4482.26 $mgkg^{-1}$. As can be seen from Table 2, the concentrations of metals in sample 5 were higher than the concentrations of metals in all other samples. By comparing the results with the Kosovo limited

Table 2 The average concentrations of heavy metal in soil and Salix purpurea samples from Mitrovica region, ± se (range) and limited values (concentration unit is in $mgkg^{-1}$ dry weight)

Samples	($mgkg^{-1}$)	As	Cd	Co	Cr	Cu	Ni	Pb	Zn
	Soil	351.69 ± 1.5	29.36 ± 2.1	27.29 ± 0.4	42.39 ± 2.1	187.34 ± 1.4	94.52 ± 2.4	388.38 ± 2.4	658.73 ± 2.3
1	Roots	12.63 ± 0.1	6.36 ± 0.7	8.68 ± 0.02	9.67 ± 0.53	22.91 ± 1.3	61.25 ± 2.1	30.92 ± 2.6	546.13 ± 0.02
	Stem	5.22 ± 1.2	2.67 ± 0.5	3.73 ± 0.4	4.56 ± 0.12	5.2 ± 0.4	17.46 ± 1.7	12.68 ± 0.7	395.52 ± 0.3
	Leaves	0.35 ± 0.05	2.29 ± 0.02	4.07 ± 0.6	2.44 ± 0.3	16.3 ± 0.3	7.49 ± 0.13	30.79 ± 0.06	886.99 ± 0.05
	Soil	640.05 ± 1.3	76.65 ± 0.5	17.83 ± 0.9	58.23 ± 0.9	148.1 ± 2.6	195.24 ± 1.5	4692.22 ± 1.5	2867.78 ± 2.1
2	Roots	25.89 ± 0.06	13.67 ± 0.5	8.73 ± 0.7	5.98 ± 0.05	32.90 ± 0.8	32.87 ± 0.07	1986 ± 2.5	3045.65 ± 1.6
	Stem	16.97 ± 0.9	7.98 ± 0.01	4.32 ± 0.6	3.76 ± 0.8	14.87 ± 0.6	18.65 ± 1.8	387.28 ± 0.4	1095.86 ± 2.6
	Leaves	17.75 ± 0.06	10.93 ± 0.5	6.45 ± 1.7	2.95 ± 0.12	20.63 ± 0.16	27.72 ± 0.19	598.96 ± 0.25	2950.9 ± 0.02
	Soil	452.21 ± 1.9	21.73 ± 1.8	18.32 ± 1.9	34.98 ± 1.4	116.66 ± 1.8	82.86 ± 1.2	1985.64 ± 2.1	387.35 ± 2.8
3	Roots	2.09 ± 0.07	19.98 ± 0.09	17.54 ± 2.3	5.74 ± 1.75	21.98 ± 0.09	17.85 ± 2.1	28.92 ± 0.09	221.54 ± 0.06
	Stem	0.98 ± 0.4	8.65 ± 1.5	8.63 ± 0.06	1.95 ± 0.32	5.87 ± 1.01	4.74 ± 0.07	7.98 ± 1.4	108.98 ± 2.7
	Leaves	0.18 ± 0.05	15.21 ± 0.8	5.69 ± 0.2	2.39 ± 0.17	10.97 ± 0.17	6.98 ± 0.03	19.94 ± 0.14	194.95 ± 0.21
	Soil	48.91 ± 1.2	5.45 ± 2.1	16.53 ± 1.4	94.67 ± 1.9	58.93 ± 1.8	154.90 ± 2.1	428.85 ± 2.3	586.21 ± 2.4
4	Roots	1.09 ± 0.04	6.54 ± 1.6	6.58 ± 1.6	5.74 ± 2.8	17.9 ± 0.06	18.9 ± 0.07	21.9 ± 0.06	376.2 ± 0.08
	Stem	0.04 ± 0.9	4.39 ± 1.09	3.87 ± 0.08	3.74 ± 0.04	5.95 ± 0.5	6.98 ± 1.8	5.98 ± 0.5	196.8 ± 0.05
	Leaves	0.06 ± 0.05	3.82 ± 0.21	4.06 ± 0.3	2.26 ± 0.03	8.24 ± 0.03	8.52 ± 0.36	9.54 ± 0.27	238.39 ± 0.17
	Soil	881.26 ± 0.9	238.57 ± 1.3	31.25 ± 1.2	168.56 ± 0.15	943.8 ± 0.4	282.33 ± 2.8	2091.74 ± 2.6	4482.26 ± 2.5
5	Roots	13.9 ± 0.06	37.2 ± 2.5	8.76 ± 0.05	6.87 ± 0.09	31.8 ± 1.5	53.9 ± 2.6	296.98 ± 0.08	4747.8 ± 2.4
	Stem	5.64 ± 0.18	10.8 ± 0.04	3.87 ± 0.54	1.98 ± 0.23	9.09 ± 2.8	23.9 ± 0.08	108.9 ± 2.6	1876.4 ± 0.5
	Leaves	8.82 ± 0.3	17.7 ± 0.25	4.32 ± 0.4	2.56 ± 0.03	18.54 ± 0.21	31.36 ± 0.25	197.38 ± 0.18	2043.07 ± 0.19
	Soil	320.17 ± 0.6	14.26 ± 1.4	21.98 ± 0.5	67.9 ± 1.3	98.95 ± 1.5	124.59 ± 2.1	259.26 ± 2.6	2956.23 ± 3.1
6	Roots	2.87 ± 0.08	12.9 ± 0.05	7.98 ± 0.4	6.54 ± 0.98	27.18 ± 0.45	16.4 ± 2.3	27.9 ± 0.65	2794.9 ± 0.05
	Stem	1.09 ± 1.7	17.8 ± 0.4	2.89 ± 0.18	1.95 ± 0.5	10.83 ± 0.09	4.98 ± 0.67	3.98 ± 2.6	886.8 ± 1.8
	Leaves	0.08 ± 0.25	10.39 ± 0.06	4.39 ± 0.2	2.45 ± 0.12	18.53 ± 0.18	9.92 ± 0.32	9.74 ± 0.28	1559.75 ± 0.5
	No. 11/2018 A-B*	30–55	3–12	20–240	300–600	200–300	300–600	200–300	300–500
	HM Threshold in plants ***	< 5	2	15	2	40	30	20	150

*Republic of Kosovo/Ministry of Environment and Spatial Planning, Administrative instruction No. 11/2008, A-B values from A clean to B acceptable contamination values but further needs investigations; Vamerali et al. (2010) and Yanitch et al (2017) for arsenic*** values (GRK No. 11/2018), the obtained results showed to be quite high and above the limits in the areas close to mining and industrial zones. The industrial activities and opened tailing damps in Mitrovica, Kosovo presents a serious threat to the environment and human health of the population in that area. The mobility of heavy metals from mining solid waste including to urban and vegetation areas originate from multiple sources, including weathering, mining, metallurgy, manufacturing of metal-containing product and vehicle emissions (Wahsha et al. 2012; Rengel 2015). As reported in other publications Mitrovica Industrial Park is also 3 km away from the lead smelter and the largest lead tailing in the country, it is also characterised by the influence of strong winds which have an important effect on the increase of heavy metal concentration in the area (Ferat et al. 2015; Rengel 2015; Brereton et al. 2016). A similar trend is also in the samples for *Salix purpurea*. The roots, stem and leaves of *Salix purpurea* have been analysed in six identified locations. The means of total metal content in all six Salix purpure samples for roots/stem/leaves were found in the range of As 1.09/0.04/0.06 – 25.89/16.97/17.75 $mgkg^{-1}$, Cd 6.36/2.67/2.29–37.2/10.8/17.7 $mgkg^{-1}$, Co 6.58/3.37/4.06–17.54/8.63/5.69 $mgkg^{-1}$, Cr 5.74/1.95/2.39–9.67/4.56/2.44 $mgkg^{-1}$, Cu 17.9/5.95/8.24–32.9/14.87/20.63 $mgkg^{-1}$, Ni 17.85/4.74 /6.98–61.25/17.46/7.49 $mgkg^{-1}$, Pb 21.9/5.98/9.54–1986 /387.28/598.96 $mgkg^{-1}$ and Zn 3045.65/1095.86/2950.9 $mgkg^{-1}$. The results in Table 2 shows a very high concentration of As, Cd, Pb and Zn in Sample 5 which is located close to Mitrovica Industrial Park and S2 located close to the mining flotation area in Tuneli i Pare. These results are consistent with *other* studies that demonstrate substantial levels of heavy metals in soil and air in this area (Kerolli Mustafa et al. 2015a, 2015b; Ferati et al. 2015).

Three—dimensional surfaces were plotted for the heavy metal accumulation in *Salix purpurea* samples(roots, stem and leaves) for all six locations in the Mitrovica region in Kosovo (Fig. 2). When comparing the plot of the results obtained for the results of roots, stem and leaves of *Salix Purpurea*, it is possible to visualize that the region where the concentration of heavy metals is maximized is not the same in all graphs for each analyzed elements. The surfaces presented in the following figures describe the variations of the roots, stem and leaves for *Salix purpurea* grown in that region.

The pH values of soil range between 3.4 and 7.2 which present a suitable condition for the mobility of metals around the mining complex Trepca where acidity is low. Also, the bioconcentration factor (*BCF*) and translocation factor (*TF*) values > 1 have been used to evaluate the potential of plant species for phytoextraction and phytostabilization of metals in soil (Sijakova-Ivanova et al. 2017; Rengel 2015; Wu et al. 2008). By comparing *BCF*and *TF* we can observe the ability of Salix purpurea grown close to the mining area in taking up metals from soils and translocating them into other parts of the plant. The results for the bioconcentration factor (*BCF*) and translocation factor (*TF*) for the ratio roots/soil and stem/roots are presented in Table 3.

The highest increase of *BCF* value was observed for zinc followed by cadmium and other selected metals: Zn 1.1, Cd 1.2, Co 0.95, Ni 0.65, Cu 0.27, Pb 0.42 As 0.04 and Cr 0.22. The translocation factor (*TF*) results show the similar trend that this factor increases for the stem/roots ratio for As, Pb, Cd, Cr and Z. The highest value of *TF* factor was observed for sample 2 for Zn, while the lowest value of the translocation factor (0.02) was calculated for As in samples 6. Observed interactions and the response surface plots, correlation matrix and principal components analysis confirmed that the uptake of metals by *Salix purpura* as well as the degree of accumulation was high in the region close to Mitrovica Industrial Park. Correlations matrix between the contents of the element in the investigated soil and *Salix purpurea* were established using bivariate statistics. The revealed strong correlation between Cd and As, Cr and Cd, Cu and Cd, Ni and Cd, Cu and Cr, Ni and Cr, Ni and Cu and Zn and Ni is shown in Table 4. In general, the correlation coefficient used for analysing the correlation between elements in the investigated samples showed a very positive correlation between metals in soil and *Salix purpurea* samples coming from the common source of pollution. The statistical results confirm that there is the original relationship between the heavy metals analysed and the same anthropogenic and lithogenic sources.

To assess the relationship between soil and bioconcentration of metals in *Salix purpurea* a principal components analysis (*PCA*) was applied to evaluate the possible relevance of the major elements with the highest environmental impact such as Zn, Pb, Cd, Cu, Co, Cr, Ni and As (Fig. 3). Two principal components (PCA 1 and PCA 2) were extracted through principal components analysis of each heavy metal of samples analyzed.

As shown in Fig. 2, the determination of the relevance among heavy metals in soil and plant were grouped into two classes of components. The closest relevance was shown between Cd, Cu, Cr, Co and Zn and Ni and As and Pb on the other side. The relevance of the heavy metals supports the fact that Pb and As are more concentrated close to the Lead smelting area, while other elements are more concentrated on flotation and zinc smelter in Mitrovica Industrial Park. The research showed that the soil and plant contamination in the Mitrovica region, Kosovo is highly affected by mining activities. Mitrovica urban area is facing a huge threat after the deposition of mining waste in open tailing damps without proper management.

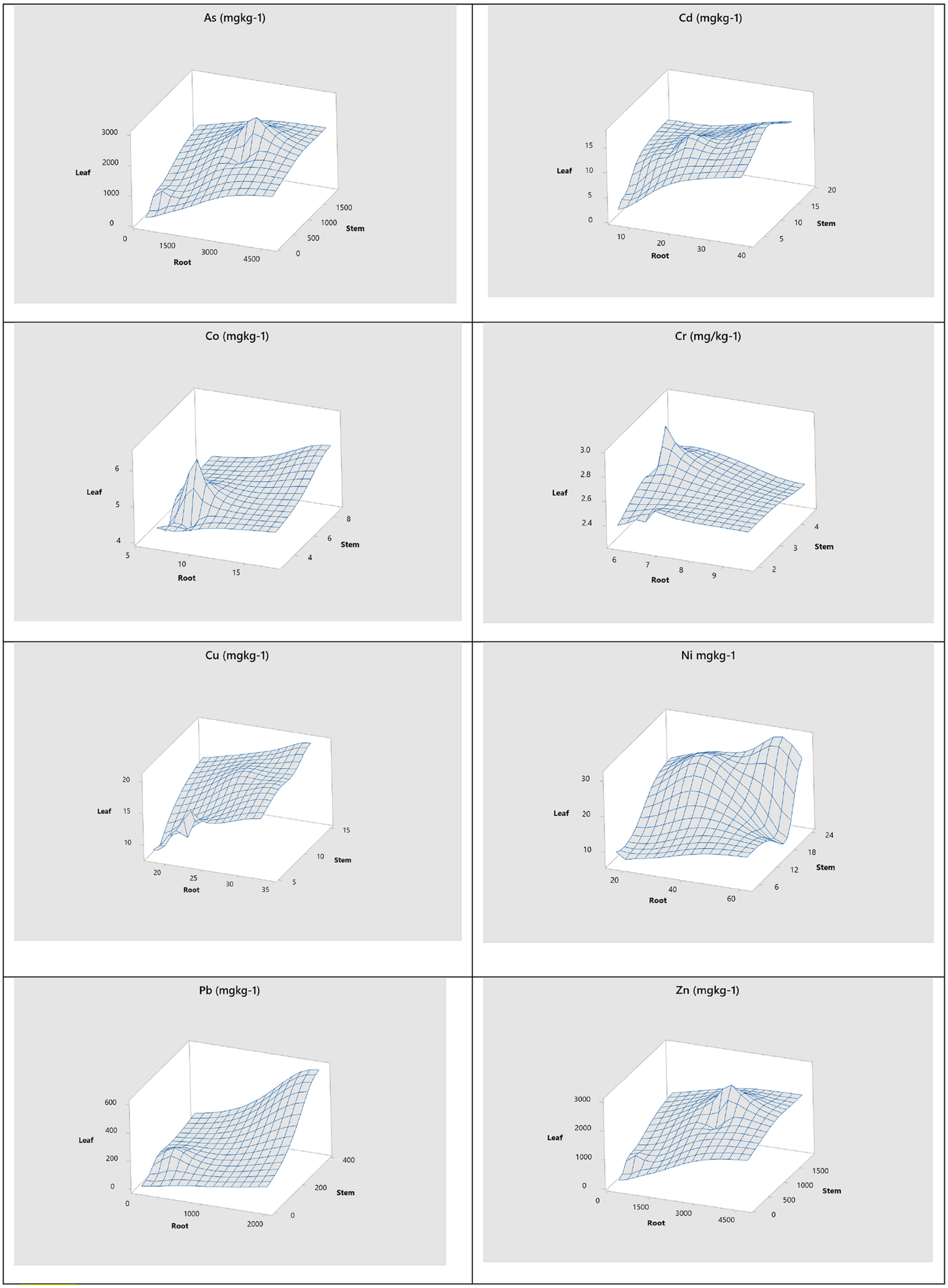

Fig. 2 Response surface plots of the concentration characteristics of the heavy metals in roots, steam and leaves

Table 3 *BCF* and *TF* for *Salix purpurea* from the Mitrovica locality

Samples		As	Cd	Co	Cr	Cu	Ni	Pb	Zn
1	BCF	0.036	0.21	0.31	0.22	0.12	0.65	0.08	0.82
	TF	0.41	0.41	0.43	0.47	0.23	0.28	0.41	0.72
2	BCF	0.04	0.17	0.49	0.1	0.22	0.17	0.42	1.1
	TF	0.64	0.58	0.5	0.62	0.43	0.56	0.19	0.35
3	BCF	0.004	0.91	0.95	0.16	0.18	0.21	0.015	0.58
	TF	0.47	0.43	0.5	0.33	0.26	0.26	0.27	0.5
4	BCF	0.022	1.2	0.39	0.06	0.3	0.12	0.05	0.64
	TF	0.03	0.67	0.58	0.65	0.33	0.36	0.27	0.52
5	BCF	0.015	0.15	0.28	0.04	0.03	0.2	0.14	1.06
	TF	0.4	0.3	0.45	0.29	0.28	0.44	0.37	0.39
6	BCF	0.0089	0.9	0.9	0.09	0.27	0.13	0.1	0.95
	TF	0.37	1.37	0.36	0.3	0.4	0.3	0.14	0.31

Table 4 Correlation matrix for r values of heavy metals in soil, roots, leaves, Stem from the Mitrovica Region

	As	Cd	Co	Cr	Cu	Ni	Pb	Zn
As	1.00							
Cd	0.87	1.00						
Co	0.58	0.71	1.00					
Cr	0.47	0.83	0.55	1.00				
Cu	0.79	0.97	0.81	0.86	1.00			
Ni	0.68	0.89	0.44	0.89	0.81	1.00		
Pb	0.64	0.38	−0.19	0.01	0.16	0.43	1.00	
Zn	0.75	0.79	0.52	0.70	0.72	0.83	0.37	1.00

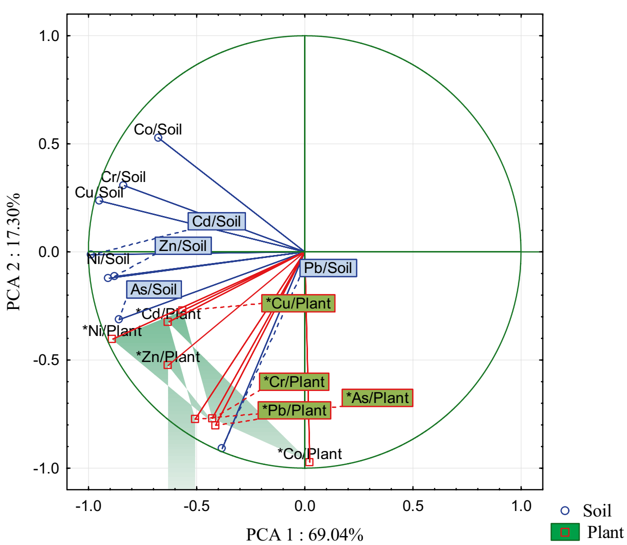

Fig. 3 Principal components analysis (*PCA)* diagram for heavy metals in selected samples from Mitrovica Region

The results also indicate that there is an increasing need for further research mainly focused on the mechanisms of remediation of the contaminated soil where the plants such as *Salix purpurea* can survive in mining areas.

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