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RESEARCH OF HEAVY METALS ON THE AGRICULTURAL LAND IN BAJGORA REGION, KOSOVO

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Abstract: For the first time, a survey about agricultural land focusing on the partitioning of the Pb, Cd, and Zn to the rural part of Mitrovica in northern Kosovo was made. Kosovo's Mitrovica is one of the main industrial sites in the former Yugoslavia and a world-class mining district in Europe. The process of obtaining metals dates since 1927. From this year until 2000, the technological process of acquisition/obtaining has been accompanied by environmental pollution by creating waste landfills. These landfills are located on the outskirts of the city of Mitrovica at a distance from 1 to4 km. In this area high levels of heavy metals in air, water, and earth were noticed. Therefore, these metal residues have a particular impact on air, earth, water, and effects on plants, animals and humans health. This situation became alarming; therefore in 2000 the production process was discontinued. During the period from 2000 to the present day, there is noticed a change of nature. Residents of the area have begun to work on agricultural lands without realizing the potential risk coming up. Despite the stagnation of industrial production, environmental pollution continues even further, especially from the landfill generated by industrial wastes. Widespread and very visible contamination mainly from Pb, Zn, Cd were found on the ground, with the highest concentrations measured near the Zveçan smelter. A significant amount of Cd, Pb, and Zn in contaminated soils/ground was quite movable/changeable, suggesting that these elements may be readily available for plants and soil/ground organisms. The main objective of this work is to address this pollution and take measures for education and information.

Keywords: landfill, heavy metals, pollution, environment, health

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Introduction

Mitrovica area in northern Kosovo represents a zone of great interest for studies on the behavior of heavy elements in contaminated soils. In fact, this area coincides with an important mining site in the former Yugoslavia, where smelting and other industrial activities led to severe contamination of surface soils by heavy elements.

The Mitrovica region with the Trepça industrial complex was one of the most popular metal ores resources in Europe. In Trepça, lead, zinc, and silver have been extracted for a long time. Mineral/ore melting and industrial processing have mainly been focused on the lead smelter in Zvecan, zinc electrolysis and battery factory in Mitrovica. These processes have been developed without any special attention to the environment and the health of the population. For years, the production of ore in the Zveçan smelter has released large quantities of particles and gases from two chimneys, thus accumulating large amounts of industrial waste deposited without any particular care or norm/principle. Thus, only from the Zvecan smelter during 1990, it is estimated to be emitted about 730 [t/year] dust, 438 [t/year] Pb, 83 [t/year] Zn and 3.6 [t/year] Cd, while the total amount of accumulated waste in the landfills of the Mitrovica region was about 40 000 000 tons.[1]

Other reports about level of heavy metal contamination in Kosovo and has shown that there is no significant pollution except for the region of Mitrovica where there are higher levels of pollution, mainly with Pb, Zn, and Cd and according to him, elements of Cd, Zn, and Pb expect that in addition to being above the level they are also quite mobile and this poses a real risk for their inclusion in the food chain.[2]

Air emissions and industrial wastes are the main sources of heavy metal dispersion in the environment. Due to the constant risk of emission from the smelter process, production output ceased in 2000. Despite the cessation or interruption of the process in the lead smelter and battery factory, there are still three large landfills that continue to endanger the environment and the population of this area. This level of pollution is a major concern for the residents of the city of Mitrovica, especially for those living near the Zveçan smelter landfill, the landfill in Kelmend (Lipa), and landfills near the Battery Factory PIM, (Figure 1).

Currently, the pollution of earth and air in this locality is caused by Trepça's industrial and technological waste, unlike the previous periods when the pollution was also caused by chimneys. Most of these wastes with different chemical and physical (granulometric) compositions are deposited in different place sand unfortunately, they are open. This pollution is caused by the transmission and discharge of particulate matter, in the form of dust. In this sense, heavy metals such as Pb, Zn, Cd, Cu, As, Ag etc., being carried by air currents (wind) are sedimented to the ground. The accumulation of metals on earth allows their introduction into the food chain (plant-animal), from which even human takes his part, by including the Pb and Cd as highly toxic.

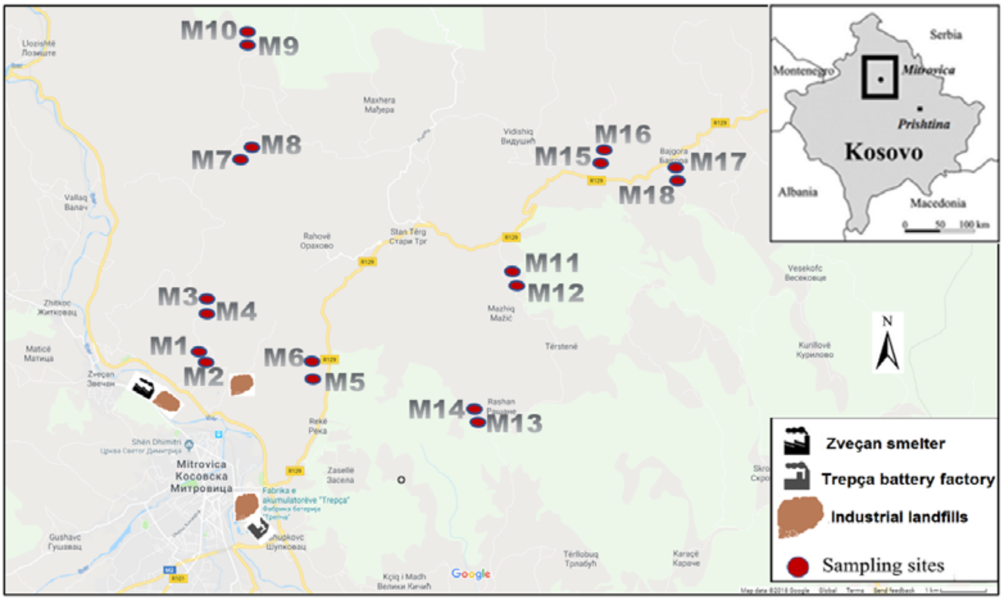

Figure1. Industrial landfills in Zveçan, Kelmend and PIM[2].

Many studies have been conducted in this area in the industrial zones and in the city of Mitrovica, and the results have shown a large pollution of heavy metals in these areas.[3-5] However, in the most remote (rural) areas studies have been scarce, and this paper aims to ascertain the level of contamination of heavy metals, in particular with Pb, Cd, Zn and Cu, and is aimed at to sensitize the residents, but also the competent bodies for the high level of metal pollution in the area with the aim of intensifying the ecosystem protection measures, identifying the risk factors.

Another polluted areas with heavy metals have been identified in some countries in the region. Tarnița is one area in Romania which has been identified with high pollution. These pollutants have been found in surface waters, soil and sterile landfills. A recent report has highlighted the extraction from contaminated soil. This extraction ensures the removal of heavy metals up to 1:10.[6] Determination of heavy metals in waste materials was done by ICP-OES and this demonstrated high accuracy of the developed protocol.[7] Heavy metals concentrations in our investigation were determined by atomic absorption spectrometry (FAAS).

The chemical fractionation of elements in soil is therefore a key factor determining their geochemical mobility, bioavailability and potential toxicity. In this regard, in recent years a number of studies have focused on the fractionation of heavy elements in contaminated.[5,8-13]

Geographical Position–The city of Mitrovica lies at the 42.53° of the northern latitude, and at that 25.52° of the eastern latitude, and at an altitude 508-510 m.

Table 1. Coordinates of sampling.

Samples	Village	COORDINATES			Color	Moisture content
		x	Y	Z		
M1	Kelmend	42°55'1.00"N	20°52'1.46"E	672	Brown	Dry
M2	Kelmend	42°54'57.20"N	20°52'7.76"E	647	Brown	Dry
M3	Boletin	42°55'23.73"N	20°51'57.02"E	778	Brown	Dry
M4	Boletin	42°55'26.29"N	20°51'59.29"E	778	Brown op	Medium
M5	Tuneli i Parë	42°54'40.68"N	20°53'58.54"E	582	Brown	Medium
M6	Tuneli i Parë	42°54'55.35"N	20°54'1.07"E	580	Dark brown	Dry
M7	Zhazhë	42°56'30.34"N	20°52'25.44"E	798	Brown	Dry
M8	Zhazhë	42°56'33.74"N	20°52'36.90"E	843	Brown op	Dry
M9	Vllahi	42°58'31.70"N	20°52'36.12"E	988	Light brown	Dry
M10	Vllahi	42°58'31.23"N	20°52'36.93"E	988	Brown	Dry
M11	Mazhiq	42°55'51.95"N	20°56'50.77"E	958	Brown	Medium
M12	Mazhiq	42°55'51.40"N	20°56'50.34"E	957	Dark brown	Dry
M13	Rashan	42°54'16.85"N	20°56'59.14"E	882	Brown	Dry
M14	Rashan	42°54'15.80"N	20°56'59.87"E	883	Brown op	Dry
M15	Bare	42°57'9.29"N	20°58'29.47"E	1160	Brown	Medium
M16	Bare	42°57'9.22"N	20°58'31.54"E	1161	Brown	Medium
M17	Bajgorë	42°57'7.44"N	20°59'26.00"E	1210	Brown	Medium
M18	Bajgorë	42°57'6.69"N	20°59'26.51"E	1213	Light brown	Dry

From the data of the Hydro meteorological Institute of the Republic of Kosovo, it is shown that in Mitrovica the winds are more commonly winding from northern direction, and rarely in the northeast, while their maximum speed reaches 18.9 m/s, while the average is 2.8 m/s.

The relief of Mitrovica, as well as the surrounding area (the area where research was conducted), is characterized by morphology dominating hilly and mountain formations.

The study was done in the part of the region "Shale of Bajgore" (Table 1). The Zvecan landfill and the PIM landfill are at an altitude of about 510 m, while that of Kelmend at 580 m. The surfaces of the agricultural land from which the samples were taken (M1-M18) occurred at an altitude of about 580-1213 m.

In Table 1 are presented the sampled coordinates in the Shales region, Mitrovica municipality in Kosovo, samples up to 20 cm in depth and with organic content of less than 10%, date of sampling 13.04.2018.

Results and discussion

The results of the research are presented in Tables (2, 3 and 4) as well as Figures (2, 3, 4 and 5). The results obtained from the surveyed samples, related to the concentration of heavy metals, are reflected in Table 2.

Table 2. Tabular analysis results of soil samples up to 20 cm deep.

Designation of samples	Pb(mg/kg)	Cd(mg/kg)	Zn(mg/kg)	Cu(mg/kg)
M1	1520.25	4.3	220.17	12.25
M2	1098.16	4.2	310.34	11.36
M3	495.32	5.2	235.42	17.89
M4	497.12	2.6	232.25	13.36

Table 2. Continued

M5	1252.13	4.7	1056.28	22.39
M6	1234.26	4.3	987.89	26.33
M7	370.54	0.9	250.82	13.23
M8	362.25	1.7	222.32	13.56
M9	211.65	0.8	589.02	20.46
M10	250.08	1.3	214.06	7.38
M11	809.12	5.2	387.92	23.56
M12	806.92	4.8	388.14	18.28
M13	262.44	2	227.04	15.1
M14	214.96	1.7	160.14	25.8
M15	123.12	0.4	400	20.66
M16	186.4	0.8	353.58	17.48
M17	202.28	0.9	411.3	53.66
M18	245.4	1.1	407.7	46.92
The usual content according to Lindsay	**2-200**	**0.01-0.70**	**10-300**	**2-100**

A correlation of heavy metals was applied to analyze the sources and pathways among the heavy metals.[14].The Spearman's correlation coefficients are shown in Table 4. The results demonstrate that the elemental pairs Pb-Cd (0.8) has high positive correlation and Pb-Zn (0.44) had a statistically significant positive correlation at the $p < 0.01$ significance level, which revealed that these elements most likely originate from some common sources.

Table 3. Statistical results of the data of heavy metals in topsoils around industrial sites in Mitrovica.

Elements	Pb (mg/kg)	Cd (mg//kg)	Zn (mg/kg)	Cu (mg/kg)
Mean	563.47	2.606	391.91	21.093
Median	366.4	1.85	331.96	18.085
Standard Deviation	442.83	1.784	252.63	11.862
Range	1397.1	4.8	896.14	46.28

Table 4. Correlation coefficient among different metals in topsoils around industrial sites in Mitrovica.

Elements	Pb	Cd	Zn	Cu
Pb	1			
Cd	**0.807575**	1		
Zn	**0.4447**	0.278112	1	
Cu	-0.21629	-0.20914	0.270975	1

Quantification of Pb concentration in tested samples

The content of Pb in almost of the analyzed samples is very high and sometimes exceeds the maximum permissible values.

Data obtained from analyzed soil sample, presented in Table 2, revealed that the Pb level in soil at depths of 20 cm exceeds the allowed values in several samples, while in others it is at the normal level. Pb had the highest value in the samples: M1, M2, M5, M6, M11 and M12, the value of these samples ranged from 806 mg/kg in the M12 sample, up to 1520.25 mg/kg in the M1 sample, while the other samples were close to the normal level. The samples with lower values were in the samples: M15 and M16, their value ranged from 123.12 mg/kg, sample M15, to 186.4 mg/kg in sample M16, while the value of some samples (M10, M13 and M18) was slightly above the permitted level. As seen from the results, out of 18 samples, the value of 16 samples has exceeded the normal level, while only 2 samples showed results at a normal level (Figure 2).

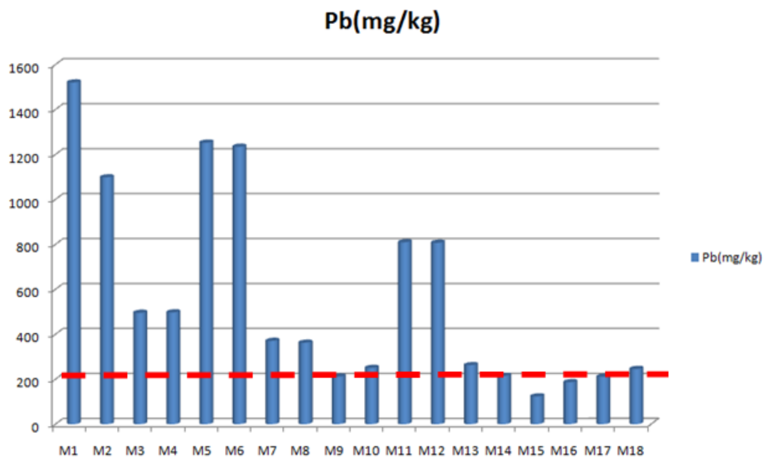

Figure 2. The content of Pb (mg/kg) - in samples with a depth of 20 cm.

Quantification of Cd concentration in tested samples

The content of Cd in all analyzed samples was very high and varied between 0.4 and 5.2 mg/kg (Table 2). Except in the M15 sample, in all other samples, the Cd content was higher compared to the defined boundary values (Figure 3).

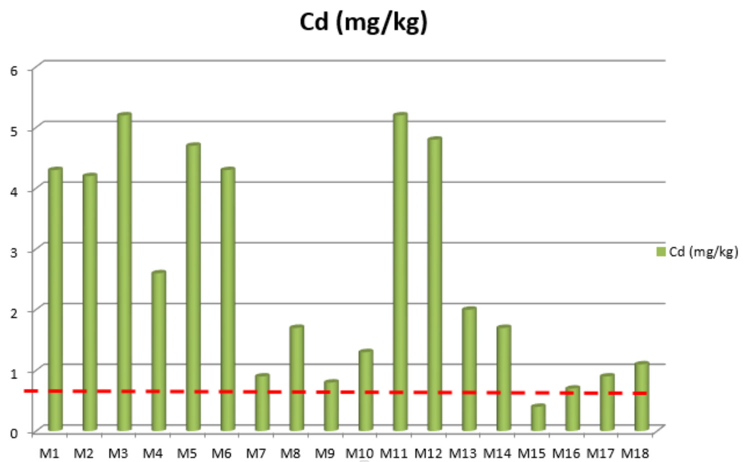

Figure 3. Content of Cd (mg/kg) in samples with a depth of 20 cm.

Quantification of Zn concentration in tested samples

Regarding the content of Zn only in some samples the gained values exceed the maximum permitted values.

The highest content was recorded in the M2, M5, M6, M9, M17, M15, M18, M16, M11 and M12 samples, the value of these samples ranged from 310.34 mg/kg in sample M2, up to 1056.28 mg/kg, in sample M5. The other samples were close to the normal level. The lowest sample values were in the: M14, M10, M1, M8, M13, M4, M3 and M7, their value ranged from 160.14 mg/kg in the M14 sample up to 250.82 mg/kg in the M7 sample. As seen from the results, out of 18 samples, the value of 10 samples is exceeded by the normal level, while 8 other samples do not exceed the maximum allowed values (Figure 4).

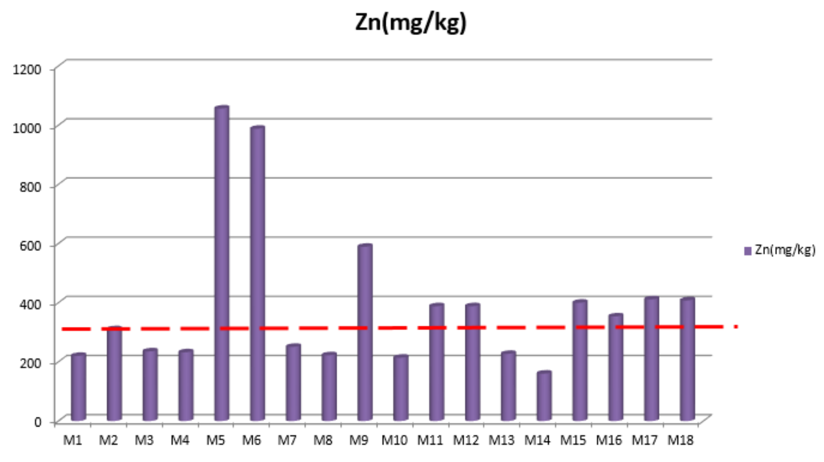

Figure 4. Content of Zn (mg/kg) - in samples with a depth of 20 cm.

Quantification of Cu concentration in tested samples

The results obtained from all analyzed soil samples (Table 2) reveal that the concentration of Cu in ground at 20 cm deep, is at the permissible level. The highest value was of 56.66 mg/kg in the sample M17, whereas the lowest value in the sample M10, 7.38 mg/kg (Figure5).

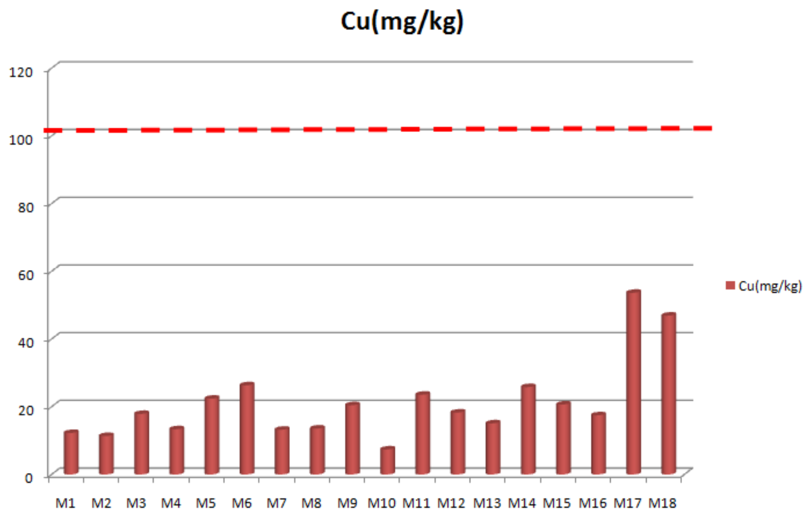

Figure 5. Content of Cu (mg/kg) in samples with a depth of 20 cm.

From the data presented we can conclude that as a result of the most typical position of the landfill in Zveçan, but also the one of Kelmend and PIM, as well as vegetation of topography, the highest content of all the metals explored (Cd, Pb, and Zn), are recorded at points near industrial landfills. The concentration of heavy metals in these samples proportionally decreases with increasing the distance from the landfill. Meanwhile, as a result of the change of the area exposure is observed a proportional decrease of the concentration of the explored metals. The deposition of metals in other analyzed samples was different, depending on the factors mentioned above. This difference can be explained as a result, primarily of the distance from the landfills but also from the micro-relief.

Based on the literature data, the number of heavy metals in the ground areas in the territory of Mitrovica was significantly higher in the period when their emission has happened also by chimneys.

Pollution Indexes of Heavy Metals

A pollution evaluation was conducted for four heavy metals, including Pb, Cd, Zn, and Cu, based on the geochemical background values of Mitrovica.[4,15] The statistical results of PI for each metal are shown in Table 5. Overall, the mean PI for all of the metals were in descending order of Pb (4.58) > Cd (3.72) > Zn (1.91) > Cu (0.49). The PI values of Pb, Cd, and Zn are much higher, ranging from Pb (1.00 to 12.36), Cd (0.57 to 7.43), and Zn (0.78 to 5.15), and Cu (0.17 to 1.25). The mean PI of Pb and Cd, pointed to a high level of pollution, while the mean PI obtained for Zn indicate moderate level pollution. Cu exhibit lower values, ranging from 0.13 to 2.29, this indicated that the topsoil in the area were non-polluted by Cu. Indeed, most of the samples had non-pollution level PI values for Cu, and only 2 samples had PI levels indicating low level of pollution, respectively. However, high PI values (higher than 3) were observed in 50% of the samples for Pb, 44.44% for Cd, 11.11% for Zn, and 0% for Cu. These findings indicate that according to the geochemical background values of Mitrovica-North Kosovo, the topsoils in the vicinity of the industrial sites of Trepça are contaminated by Pb, Cd, and Zn.

Table 5. Statistical results of pollution index (*PI*) of heavy metals in top soils around industrial sites Trepça.

Elements	***PI***			**Number of Samples**			
	Mean	***Min***	***Max***	***h.p*** ***(PI>3)***	***m.p*** ***(2<PI≤3)***	***l.p*** ***(1<PI≤2)***	***n.p*** ***(PI≤1)***
Pb	4.58	1.00	12.36	9	3	5	1
Cd	3.72	0.57	7.43	8	3	6	1
Zn	1.91	0.78	5.15	2	2	13	1
Cu	0.49	0.17	1.25	0	0	2	16

h.p: high level pollution; **m.p**: moderate level pollution**;** **l.p:** low level pollution; **n.p**: non pollution

Experimental

Apparatus

A Varian model SpectrAA 220 atomic absorption spectrometer (Victoria, Australia, http://www.varianinc.com) was used for measuring of heavy metals in air-acetylene flame. The instrumental parameters according to manufacturer's recommendations are presented in Table 6.

A Metrohm 713 pH meter (http://www.metrohm.de) equipped with a combined glass calomel electrode was used for pH measurements.

Table 6. Conditions of metal determination by flame AAS technique.

Metal	Absorbtion wavelength nm	Electric current density, mA	Flame
Pb	283.3	10	Acetylene-air
Zn	213.9	10	Acetylene-air
Cd	228.8	10	Acetylene-air
Cu	324.8	10	Acetylene-air

Reagents

All reagents were of analytical grades. Metals (purity; 99.9%), nitric acid (especially pure), hydrochloric acid (especially pure), were used. For all the aqueous solutions milliQ water was used.

Sampling and Analytical Techniques

All of the soil samples were taken on agricultural land in the region of Shale of Bajgora. The samples were taken according to the standard method, at depths of up to 20 cm. The soil samples were collected using a metal probe, then the same ones were preserved in sterile bags and for each one was made the markings according to sampling-location, as shown in Figure 6.

The obtained samples were dried for three days in consecutively manner at a temperature of the ambient (10-18 ºC). After drying them, these samples were transported to the laboratory for further treatment. Firstly, 1g of the obtained soil was taken from each sample and 10 mL of the so-called royal water ($HCl:HNO_3 = 3:1$) were added. The resulted samples were heated gently to dryness and 10 mL of HCl (1:1) were added. Then, the samples were boiled for few minutes to fulfill the acquired/gained digestion, then filtrated, washed with hot and distilled water, and finally, adjusted to a 100 mL final volume.

Figure 6. Obtaining soil samples by means of a metal probe and marking them.

The degree of concentration of heavy metals in the samples has been measured by technique of FAAS.

Standard stock solutions of metals were prepared by dissolving the metal in the solution of nitric acid (1:1). Standard solutions of metals were prepared by dilution of the appropriate standard stock solution with doubly distilled water just before using.

Analytical quality control was carried out with certified reference materials CRM CZ 7004 – loam soil with higher contents of analysts, (Analytika) using certified contents of metals (Pb 71.7 ± 2.5 mg/g, Cd 1.36 ± 0.01 mg/g, Zn 119 ± 5 mg/g, and Cu 137±4 mg/g). The accuracy and precision of the analytical method were calculated according published reports[16]. The accuracy of the metal concentrations in soils samples determined by FAAS, were ensured through 10 repeated analyses of metal standards and the results in the range ± 5 % of certified values were found. Detections (LOD) and quantifications (LOQ) limits were also calculated (Table 7). Moreover, metal concentrations were determined on three different replicate soils samples in order to assess the homogeneity of sample and the repeatability of the analysis and data were considered acceptable if standard deviation was lower than 10 %.

Table 7. Detection (LOD) and quantification (LOQ) limits of Cd, Pb, Zn, and Cu, in mg/L

Elements	Pb	Cd	Zn	Cu
LOD	0.95	$4.09 \cdot 10^{-4}$	0.26	0.25
LOQ	3.17	$1.37 \cdot 10^{-3}$	0.87	0.95

The procedure for the preparation, treatment and reading of soil samples, in order to determine the concentration of heavy metals, has been developed in the Mining Laboratory with the Flotation "Trepça" in Kishnica.

For the evaluation of the obtained results, regarding the degree of concentration of heavy metals in soil samples, we are based on the reference values according to Lindsay, (Concentration of metals in non-contaminated soil). Concentration of metals on uncontaminated land is related to the base material from which the earth is formed, in co-operation with local geological conditions.

Pollution Indexes (PI)

Pollution indexes (*PI*) of heavy metals were used to assess the degree of metal contamination in the topsoil around the industrial areas of Industrial Park Trepça in Mitrovica, North Kosovo. The *PI* was calculated using the soil environment quality standards of China. The PI was defined as follows:

$$PI = C_n/S_n$$

where *PI* is the pollution index of the element *n,* C_n is the measured concentration of the element n in soils (mg/kg), and S_n is the geochemical background concentration of element *n* (mg/kg).

The degree of heavy metal contamination in the soils can be classified into the following categories: non-pollution (PI≤1); low level pollution (1<PI≤2); moderate level pollution (2<PI≤3); and high level pollution (PI>3).

The geochemical background value is 123 mg/kg for Pb, 0.7 mg/kg for Cd, 205 mg/kg for Zn, and 43 mg/kg for Cu[4,15]. The factor 1.5 was introduced to minimize the effect of possible variations in the background values due to lithological variations.

Conclusions

In most of the cases, elevated concentration of heavy metals, in particular, lead (Pb) and cadmium (Cd) and in some areas of Zn (zinc) were found in the studied soil. This refers to the spread of heavy metals contaminated throughout the study area as well as the high concentration of pollutants. The values of their contents, in many cases, exceed the limits set for the risk of ecosystems. The distribution of the elements such as Pb, Cd, Zn and Cu in the soils of the studied region is influenced by the technological activity. This activity has the consequence in accumulation of Pb, Zn, and Cd with risks on soil ecosystems.

Compared with the North Kosovo background values of these elements in the soil, concentrations of Cd, Cu, Pb, and Zn were elevated in the vicinity of industrial sites in Mitrovica. The Spearman correlation analysis showed that Pb, Cd and Zn originated from common anthropogenic sources.

Higher mean PI values for Pb, Cd, and Zn in this study indicate that there is high level pollution, which mainly originates from industrial emissions. The PI values of Cu was low, indicating that Cu was practically unpolluted in this area

In general, the heavy metal content is reduced with the departure from industrial areas in Zveçan and Mitrovica. The actual state of landfills represents a permanent risk for pollution of the ecosystem, even of agricultural lands in the territory of Mitrovica.

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