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Assessment of Soil Heavy Metal Pollution in a Former Mining Area – Before and After the End of Mining Activities

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Abstract

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Toxicity and persistence of heavy metals, which are accumulated in the environment as the result of diverse industrial activities, represent serious environmental problem worldwide. The intense mineral extraction in mining areas has produced a large amount of waste material and tailings, which release toxic elements to the environment. The aim of the study was to determine in two time horizons (1997, 2015) the heavy metal contents of samples derived from ten sampling sites located in the former mining area of Central Spiš (Slovakia). In order to compare the level of contamination, the contamination factor (C_f^i), degree of contamination (C_d), and pollution load index (PLI) were computed. Spearman's correlation coefficient was used in order to detect the relationships among heavy metals. A serious situation was found for Hg, Zn, and Cd, which exceeded limit values at all sampling sites within both studied years. In 1997, the average values of contamination factor have shown very high contamination with all studied heavy metals, and moderate contamination with Co. In 2015, the study area was classified as very highly contaminated with As, Hg, Zn, Cu, considerably contaminated with Ni, Cr, Pb, and Cd, while Co contamination was not detected. Since 1997 till 2015 the pollution load index decreased by about 38%, nevertheless even then almost all sampling sites were classified as heavily polluted. Despite the fact that mining activities were stopped or limited at the beginning of the 21st century, the presence of heavy metals in soils remains at a serious level. The high level of contamination is a result of heavy metal persistence and non-biodegradability.

Keywords: contamination factor; degree of contamination; pollution index; soil toxicity

With the rapid development of mining activities landscape changes as well as environmental pollution have become still more serious. The intense mineral extraction has produced a large amount of waste material accumulated on the heaps or tailings (Ma *et al.* 2015). Without proper management, abandoned mines and tailings are the source of heavy metals, which are washed out by precipitation and can contaminate all environmental components (Liakopoulos *et al.* 2010; Li *et al.* 2014). The extent and degree of heavy metal contamination vary depending upon the mineralogical and geochemical characteristics of both ore and host rocks (Ivazzo *et al.* 2012).

Soil is a critical environment because it is able to accumulate pollutants produced by anthropogenic activities, such as mining and processing of ore, industry, agriculture, traffic, etc. The transport of heavy metals in soil is the result of processes between soil and metal components, which include processes of physical, chemical, and biological nature (Violante *et al.* 2008). However, soil is not only a passive acceptor of heavy metals, polluted soils become a source of contamination for other environmental components and the food chain (Gholizadeh *et al.* 2015). In addition, heavy metals are non-degradable and persistent, their presence in soil is stable and

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long-term (Lizárraga-Mendiola *et al.* 2009). It has been found that the presence of heavy metals in the soil environment significantly influences biological, chemical, and physical soil properties, which results in decreased soil fertility and also ultimately crop contamination.

Several studies have been focused on this issue in Central Europe, including Poland (Jablonska & Siedlecka 2015; Woch *et al.* 2015), Czech Republic (Pavlů *et al.* 2007; Soudek *et al.* 2015), Hungary (Szabó *et al.* 2015) or Germany (Mayanna *et al.* 2015).

In Slovakia, a lot of attention has been paid to the area of Central Spiš which is considered environmentally loaded and unhealthy. Here, high levels of heavy metals were found not only in soil, water or sediment samples (Angelovičová *et al.* 2014; Holub *et al.* 2015; Singovszká *et al.* 2015), but several studies detected extremely high levels of heavy metals in plants (Banasová & Lackovicová 2004; Slávik *et al.* 2016) or animal products (Stanovič *et al.* 2016).

The aim of the study was (*i*) to determine the level of contamination with heavy metals in the area of Central Spiš in two time horizons (1997, 2015), (*ii*) to compare the level of contamination between the studied years using the contamination factor, degree of contamination, and pollution load index, (*iii*) to assess the correlation relationship between heavy metals and significant differences in heavy metal pollution between the studied years.

MATERIAL AND METHODS

Mining activities in the region of Central Spiš were focused on copper and mercury ore mining and processing. Moreover, based on the environmental regionalization, the area is considered environmentally loaded and hazardous for human health. Mining activities started here in the 13th century and peaked during the 19th century. At the beginning of the 21st century, when mining was not as profitable as before, processing plants finished or reduced their production. Abandoned mine areas, heaps of waste material and tailing ponds remained unchanged and became the source of undesirable substances, heavy metals. From the geographical point of view, the area (ca. 375 km^2) is situated in the Hornád River valley. The population (ca. 52 000) is concentrated predominantly in villages. Climatologically the region can be characterized as moderately warm and moderately wet with mean January temperature –5°C to –20°C (Džatko 1989). GPS coordinates and altitude of the sampling sites are listed in Table 1 and their location within the Central Spiš region is shown in Figure 1.

The tailing pond, copper processing plant, and dozens of heaps of waste material randomly distributed over the study area represent the main sources of toxic elements (Figure 1). The tailing pond is localized in the north-eastern part of Slovinky village cadastre. Slag material occurring in the upper part of the pond is of powder consistency. Small particles are wind-transported north-westwards, directly to the inhabited part of the village. The plant located in the Kompachy village cadastre has long been used for ore (especially copper) processing. Nowadays it is still active but, compared to the past, its production has been considerably reduced.

Soil sampling from 10 sampling sites (S) (grasslands) in the former mining area of Central Spiš was carried out in November 1997 and November 2015 from the surface layer (15–20 cm). From each sampling site four samples were collected and mixed. Samples were stored in plastic bags, air dried in laboratory condi-

Table 1. GPS and altitude information about sampling sites

Cadaster of the village	Sampling site	GPS coordination		Altitude (m a.s.l.)
		latitude	longitude	
Hrišovce	1	48°54'20.65''N	20°39'18.53''E	524
Richnava	2	48°55'23.93''N	20°53'59.31''E	350
Krompachy	3	48°55'20.82''N	20°53'58.65''E	353
Krompachy	4	48°55'6.781''N	20°52'51.53''E	394
Kolinovce	5	48°55'23.33''N	20°51'32.24''E	387
Kolinovce	6	48°55'30.62''N	20°51'20.29''E	383
Kolinovce	7	48°55'39.36''N	20°50'48.38''E	368
Kolinovce	8	48°55'15.31''N	20°50'41.44''E	458
Nižné Slovinky	9	48°52'48.92“N	20°50'50.99''E	470

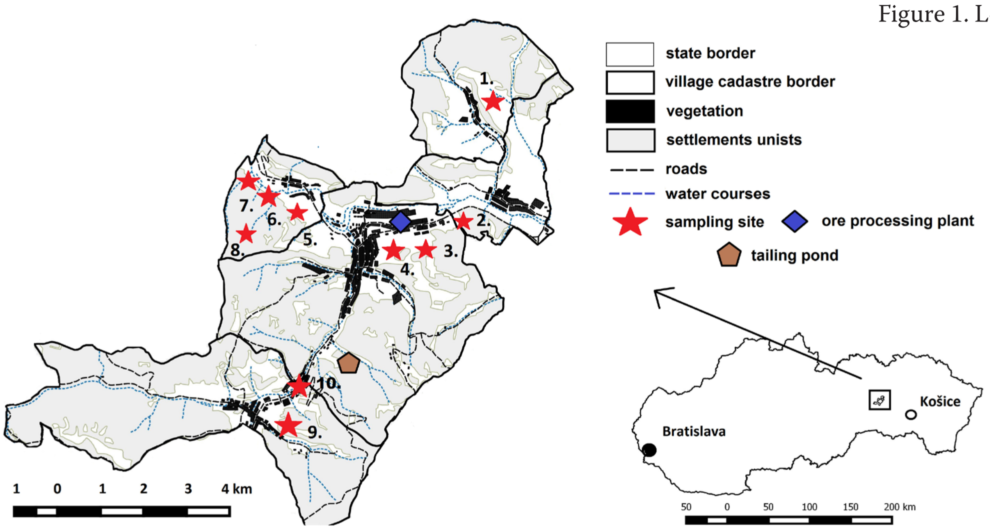

Figure 1. Location of sampling sites

tions, handy crushed, and sieved through a sieve with a 2 mm size opening. All soil samples were classified as sandy-loamy or loamy and Cambisols. The soil samples were digested in a mixture of 2.5 ml of 65% HNO_3 Suprapur® and 7.5 ml of 37% HCl Suprapur® (both Merck, Darmstadt, Germany) using microwave decomposition Mars Xpress 5 (CEM Corp., Matthews, USA). After mineralization samples were filtered and the volume was adjusted to 100 ml with deionized water. The total content of heavy metals (As, Hg, Zn, Cu, Co, Ni, Cr, Pb, Cd) was determined by flame atomic absorption spectrometry(F-AAS) using SpectrAA 240FS (Varian Inc., Mulgrave, Australia). Statistical operations were performed in R software (RStudio Team, Boston, USA). Spearman's correlation coefficient was calculated in order to determine correlation relationships between heavy metal contents in both time horizons. Values of $P \leq 0.05$ and $P \leq 0.001$ were considered statistically significant. Student's *t*-test was conducted to determine differences in metal concentrations between time horizons. Hierarchical cluster analysis (HCA) with squared Euclidean distance was used to identify clusters of heavy metals, for both studied years, with a similar contamination factor. Cluster analysis was performed using Ward's algorithm. Data were log transformed prior to analysis. HCA was used to output natural clusters present in the data. For determination of metal pollution in soils, contamination factor (C_f^i) and degree of contamination (C_d) proposed by Hakanson (1980) were used. The C_f^i is a single-metal index determined by the relation:

$$C_f^i = \frac{C_{0-1}^i}{C_n^i} \qquad (1)$$

where:

C_{0-1}^i – concentration of metal in the sample
C_n^i – background level of metal in upper Earth's crust suggested by Čurlík and Ševčík (1999) and Kabata-Pedias (2011)

The background values of metals (C_n^i) in natural soils were considered as 0.3, 20, 10, 20, 550, 400, 10, 30, 40 mg/kg for Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, and Zn, respectively (Kabata-Pendias & Pendias 1992; Čurlík & Ševčík 1999; Kabata-Pendias 2011). Four classes of were recognized by Hakanson (1980): (*i*) low contamination factor (if $C_f^i < 1$), (*ii*) moderate contamination factor (if $1 \leq C_f^i < 3$), (*iii*) considerable contamination factor (if $3 \leq C_f^i < 6$), and (*iv*) very high contamination factor (if $C_f^i \geq 6$). The C_d is a measure of the degree of overall contamination in a particular sampling site and was defined as the sum of all C_f^i:

$$C_d = \sum_{i=}^{n} C_f^i \qquad (2)$$

The C_d was according to Hakanson (1980) divided into four groups as follows: (*i*) low degree of contamination ($C_d < 8$), (*ii*) moderate degree of contamination ($8 \leq C_d < 16$), (*iii*) considerable degree of contamination ($16 \leq C_d < 32$), and (*iv*) very high degree of contamination ($C_d \geq 32$). Pollution load index (PLI) proposed by Tomlinson *et al.* (1980) is an empirical index that comparatively assesses the level of heavy metal pollution for each sampling site. PLI was calculated by the relation:

$$PLI = (C_{f1} \times C_{f2} \times C_{f3} \times \ldots \times C_{fn})^{1/n} \qquad (3)$$

where:

n – number of assessed metals (n = 9 herein)

C_f – contamination factor of individual pollutant

The value of PLI was classified into four groups (Wang *et al.* 2010): (*i*) no pollution (if PLI < 1), (*ii*) moderate pollution (if 1 ≤ PLI < 2), (*iii*) heavy pollution (2 ≤ PLI < 3), and (*iv*) extreme pollution (PLI ≥ 3).

RESULTS

Mining and subsequent ore-processing activities are always associated with very high levels of heavy metal contamination of the environment (Alloway 2010; Ma *et al.* 2015). The descriptive statistics of the heavy metal concentrations in soils of the Central Spiš area in two year horizons are listed in Table 2. Average values of heavy metals were compared with limit values defined by Act No. 220/2004 Coll. for sandy-loamy and loamy Slovak soils. Limit values are 25, 0.50, 150, 60, 15, 50, 150, 70, and 0.70 for As, Hg, Zn, Cu, Co, Ni, Cr, Pb, and Cd, respectively.

In 1997, total contents of Hg, Zn, Co, Ni, and Cd exceeded limit values at all sampling sites. No contamination by chromium was found. Average value of Hg exceeded limit value more than 29 times. Serious contamination was found for Cu and Zn, which exceeded limit value on average 10.3 and 4.5 times, respectively. Extremely high and above-limit values of Hg and Cd were determined at all sampling sites in 2015. No contamination with Ni and Cr was found. The above-limit value of copper was determined at the same sampling sites as in 1997. The highest values of arsenic were determined at S2 and S5. The presence of arsenic in the Central Spiš area has been reported previously, but usually was attributed to the geochemical origin (Hronec *et al.* 2008). At the sampling sites located close to the processing plant (S3, S5, S6, S7), the highest values of copper, lead, and zinc were determined in both years. Processing plant emissions and dumps of residual material stored in the plant area are considered as major sources of toxic elements. As mentioned above, the processing plant is focused predominantly on copper ore processing. The solid waste from the copper production with residues of lead and zinc (Michaeli & Boltižiar 2010) is the source of increased contents of the three heavy metals around the processing plant. Lastincová *et al.* (2003) and Wilcke *et al.* (2001) identified the region of Central Spiš as a hot spot of copper contamination.

Comparing the research horizons, a significant decrease of all evaluated heavy metals was ascertained. Since 1997 till 2015, the average value of As, Hg, Zn, Cu, Co, Ni, Cr, Pb, and Cd decreased above

Table 2. Average values of heavy metals (mg/kg) in soil samples from the region of Central Spiš and the results of Student's *t*-test indicating significant differences in the heavy metals contents between the evaluated years 1997 and 2015

Heavy metal	Year	Min	SWMX	Max	SWMI	Mean ± SD
As*	1997	12.3	2	153.3	10	52.1 ± 43.4
Hg		3.01	1	59.1	10	13.3 ± 16.5
Zn*		215.6	3	1697.8	8	628.3 ± 474.3
Cu*		17.3	2	1273.7	8	556.7 ± 522.4
Co**		16.3	1	30.2	2	21.5 ± 4.9
Ni*		56.7	10	342.3	4	97.4 ± 86.8
Cr		38.8	10	331.9	2	81.8 ± 88.5
Pb*		32.2	2	503.5	8	207.9 ± 165.4
Cd**		2.23	6	7.4	10	4.12 ± 1.53
As	2015	10.4	2	146.7	10	48.4 ± 41.2
Hg		2.78	1	41.1	8	11.5 ± 11.2
Zn		148.9	6	1208.7	8	482.4 ± 334.2
Cu		22.1	6	705.5	10	303.2 ± 264.9
Co		8.55	1	27.2	7	17.1 ± 6.72
Ni		17.9	10	48.8	5	31.77 ± 7.76
Cr		22.2	10	63.1	2	36.98 ± 14.5
Pb		30.1	3	159.6	8	96.7 ± 46.9
Cd		1.12	5	2.05	10	1.57 ± 0.27

*, **$P < 0.05$, 0.01; SWMI – site with a minimum determined value; SWMX – site with a maximum determined value; SD – standard deviation

Table 3. Correlation relationship between heavy metals in soil samples

	Hg	Zn	Cu	Co	Ni	Cr	Pb	Cd
As	0.35	0.20	0.01	–0.48	–0.52	–0.24	0.01	0.22
Hg		0.95**	0.80*	0.74*	0.81*	0.70*	0.48	0.55
Zn			0.01	0.18	0.57	0.88*	0.01	0.02
Cu				0.03	0.75*	0.17	0.01	0.04
Co					0.01	0.01	–0.12	0.33
Ni						0.03	0.36	0.01
Cr							–0.61	0.03
Pb								0.01

*, ***P* < 0.00, 0.010

7.1, 13.5, 23.2, 45.1, 20.0, 67, 54.8, 53.5, and 61.8%, respectively. Since the beginning of the 21st century, mining activities have become less attractive and low productive, additionally more attention has been paid to environmental protection (Banasová & Lackovičová 2004; Hronec *et al.* 2008) with positive impact on the environmental situation within the study area. But due to the fact, that heavy metals are non-biodegradable, they remain accumulated in the soil for years (Mehrabi *et al.* 2015; Mirzaei *et al.* 2015).

Spearman's correlation analysis between heavy metals in the soil samples is given in Table 3. Mercury gave significant positive correlation with all evaluated heavy metals (except correlation with Pb and Cd). Significant positive correlation was found between Ni and Cu ($r = 0.75$, $P < 0.05$) and Zn and Cr ($r = 0.88$, $P < 0.05$). Correlations between heavy

Table 4. Values of contamination factor (C_f) and degree of contamination (C_d) for each sampling site in both evaluated years 1997 and 2015

Sampling site	Year	C_f									C_d
		As	Hg	Zn	Cu	Co	Ni	Cr	Pb	Cd	
1	1997	8.0	53.7	8.2	4.4	1.5	8.1	6.6	3.4	12.1	106.0
2		21.3	10.2	9.9	59.5	0.8	5.7	3.9	16.8	8.2	136.4
3		9.1	5.5	26.4	63.7	0.9	6.7	4.0	11.8	15.9	143.9
4		2.9	13.0	10.9	15.9	0.8	5.7	6.5	3.9	11.8	71.6
5		12.2	9.2	19.7	36.8	0.9	6.8	4.7	8.8	18.0	117.2
6		7.8	4.3	42.4	63.3	1.2	9.1	6.1	13.4	24.6	172.1
7		2.7	8.4	21.2	23.9	1.0	8.2	5.2	6.4	15.7	92.7
8		3.2	3.0	5.4	0.9	1.0	5.8	5.0	1.1	10.0	35.4
9		3.4	11.2	6.1	8.8	1.3	7.1	6.7	2.0	13.4	59.9
10		1.7	27	6.8	1.1	1.4	34.2	33.2	1.6	7.4	90.2
Average value		7.2	12.1	15.7	27.8	1.1	9.7	8.2	6.9	13.7	102.3
1	2015	8.1	37.3	7.6	3.9	1.4	3.1	5.7	2.9	5.5	75.5
2		20.4	8.0	7.5	32.7	0.6	3.5	2.3	4.3	5.0	84.2
3		7.7	6.4	18.6	31.0	0.5	3.3	2.7	5.3	5.8	81.4
4		2.7	11.4	9.4	10.9	0.6	2.9	3.9	3.0	5.1	50.0
5		10.7	10.5	15.1	17.1	0.7	1.8	4.8	4.9	6.1	71.7
6		7.5	3.3	30.2	35.3	1.1	2.9	2.9	4.9	6.8	94.9
7		2.2	9.1	18.0	10.4	0.4	3.1	3.3	2.9	5.0	54.4
8		3.4	2.5	3.7	1.2	1.1	2.7	2.7	1.0	4.2	22.5
9		3.1	13.0	5.2	8.0	1.0	3.5	2.2	1.8	5.1	42.9
10		1.4	2.7	5.3	1.1	1.2	4.9	6.3	1.3	3.7	28.0
Average value		6.7	10.4	12.1	15.2	0.9	3.2	3.7	3.2	5.2	60.4

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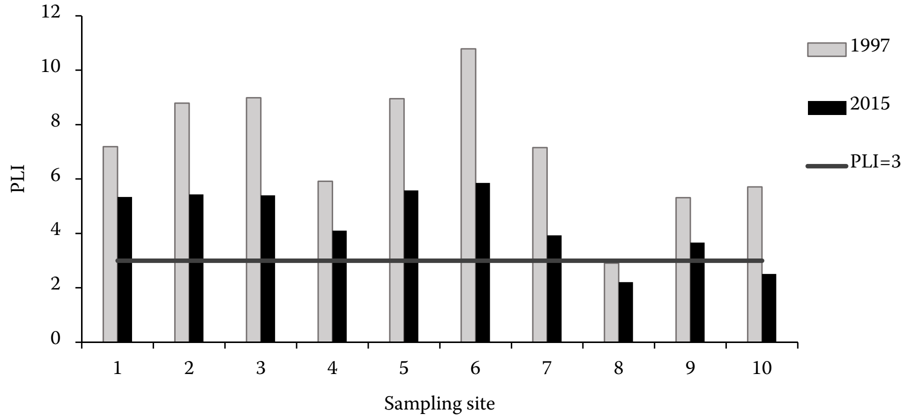

Figure 2. Values of pollution load index (PLI) for 1997 and 2010 at the sampling sites on the Central Spiš area

metals could suggest their identical origin (Rafiei *et al.* 2010; Bekteshi & Bara 2013), but in some cases provide only interesting information regarding the source and the pathways of the metals (Chen *et al.* 2014). No significant correlation was found for arsenic. Numerous studies focused on the environmental pollution of the Central Spiš region have found that here the contamination of soil environment by arsenic is related not only to anthropogenic impact but also to the geochemical effects of mineralized zones (Čurlík & Ševčík 1999; Hronec *et al.* 2008). The presence of all other metals is caused by mining and smelting activities what is confirmed by significant positive correlations.

The soil contamination assessment was based on C_f, which was calculated for each heavy metal and each sampling site, for both studied years (Table 4). In order to compare the results for the two years analyzed, the average value of contamination factor for each evaluated year was calculated. Based on the average values of contamination factor, in 1997 the soils were classified as very highly contaminated with all analyzed heavy metals and moderately contaminated with Co. In 2015 the study area was classified as very highly contaminated with As, Hg, Zn, Cu, considerably contaminated with Ni, Cr, Pb, and Cd, and not contaminated with Co.

Values of contamination degree for 1997 ranged from 172.1 (S6) to 35.4 (S8). Based on the classification by Hakanson (1980), at all sampling sites a very high degree of contamination ($C_d \geq 32$) was determined. In 2015 the values of contamination degree ranged from 94.9 (S6) to 22.5 (S8). At all sampling sites (except S8 and S10) a very high degree of contamination was determined, only S8 and S10 showed a considerable degree of contamination. Since 1997 till 2015 the average value of contamination degree decreased by about 41%. Based on the PLI, which was used in order to assess quality of sampling sites (Wang *et al.* 2010), in 1997 all sampling sites were considered as extremely polluted (PLI > 3). In 2015 the average values of PLI decreased by about 38%, but still, all sampling sites were considered extremely polluted, except S8 and S10, which were found as heavily polluted (Figure 2).

In order to determine the groups of heavy metals, the cluster analysis of contamination factor values was used. The Euclidean distances for similarities in the variables were calculated. The obtained results are presented by a dendrogram (Figure 3), where the distance axis represents the degree of association between groups of variables. The dendrogram results indicate two clusters for both studied years. For the year 1997, group 1 includes only Co, indicating lower contamination with cobalt compared to the other metals; group 2 consists of all other heavy metals, based on the contamination factor classified as very highly contaminated. For the year 2015, group 1 includes Hg, Zn, and Cu; group 2 consists of As, Ni, Cr, Pb, Cd, and Co. Cobalt is standing alone in group 2, what suggests a difference in its contamination factor value, which was (like in 1997) absolutely the lowest if compared to the other metals in the group. Heavy metals included in the first group are, according to the contamination factor, classified as very highly contaminated. According to the contamination factor classification, arsenic should be included in the first group, but because of its only slightly exceeding value (if compared to that of Hg, Zn, and Cu), it was grouped with metals classified as considerably contaminated. Mining and smelting activities in the study area were focused predominantly on copper and mercury production,

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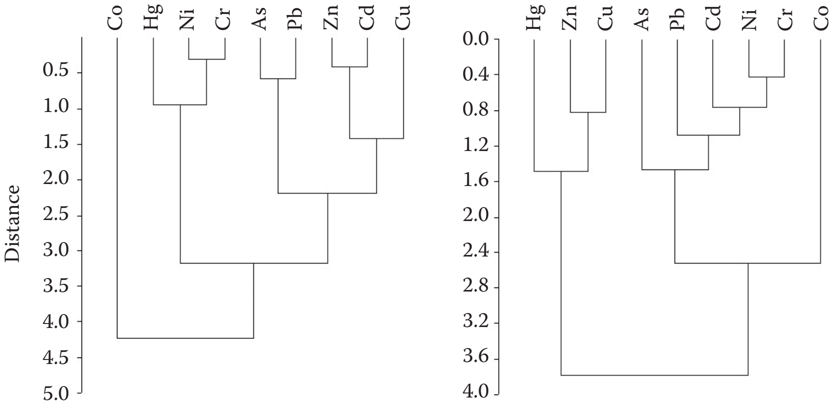

Figure 3. Dendrogram results of contamination factor values of heavy metals for 1997 (a) and 2015 (b) in the former mining area of Central Spiš

therefore contamination with these metals has for long term been considered the most serious (Hronec *et al.* 1992; Bálintová *et al.* 2014). Contents of other heavy metals (zinc, lead, etc.) produced as accompanying material have also been found serious for the environment (Angelovičová *et al.* 2014).

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