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Geochemical distribution of selected trace elements in the soil-plant system from Mănăila mining area, Romania

Simona Petronela Iftode[1], Ramona Huzum[2], Doina Smaranda Sirbu-Radasanu[2], Nicolae Buzgar[1], Ovidiu Gabriel Iancu[1], Andrei Buzatu[1]

[1] "Alexandru Ioan Cuza" University of Iaşi, Department of Geology, 20A Carol I Blv, 700505 Iaşi, Romania
[2] "Alexandru Ioan Cuza" University of Iaşi, Department of Research, 20A Carol I Blv, 700505 Iaşi, Romania

Abstract

This study presents the geochemical distribution of several trace elements (TE) (Ni, Cu, Zn, As, Pb) in a soil-plant system, taking into account the lingonberry, a shrub most widely spread around the Mănăila mining region in Suceava County. A number of 34 samples (soil and the plant above), collected arround the open pit, was prepared for the analysis of soils – using ED-XRF method and separated plant organs (roots, leaves, fruits) – using ICP-MS, after HNO_3 microwave digestion.
Compared with Romanian legislative values, the soil from the surroundings of the Mănăila open pit present contents for Ni and Zn within the normal values; Cu ranges between normal and alert threshold, while As and Pb exceed the alert threshold. The geo-accumulation index (I_{geo}) average values point out to a moderate contamination with As, Pb and Cu and support a higher potential toxicity risk for these elements. Based on the bioconcentration factor (BCF), the lingonberry behaves as an indicator for Cu and Zn and as an excluder for Ni, As and Pb. The transfer factor (TF) values show an accumulation at the aboveground plant parts for Ni and Cu.

Keywords: TE, TF, BCF, soil-plant system, mining area, *Vaccinium vitis idaea*.

1. Introduction

Nowadays, the contribution of geogenic and anthropogenic sources to the trace elements loading in soil is generally accepted. The geogenic component of soil is associated with the nature of parent material, while the anthropogenic component results from various human activities related to the studied area. The large amount of waste materials, tailings and acid mine drainage are potentially sources for

Corresponding author's e-mail: iftode_simona@yahoo.com

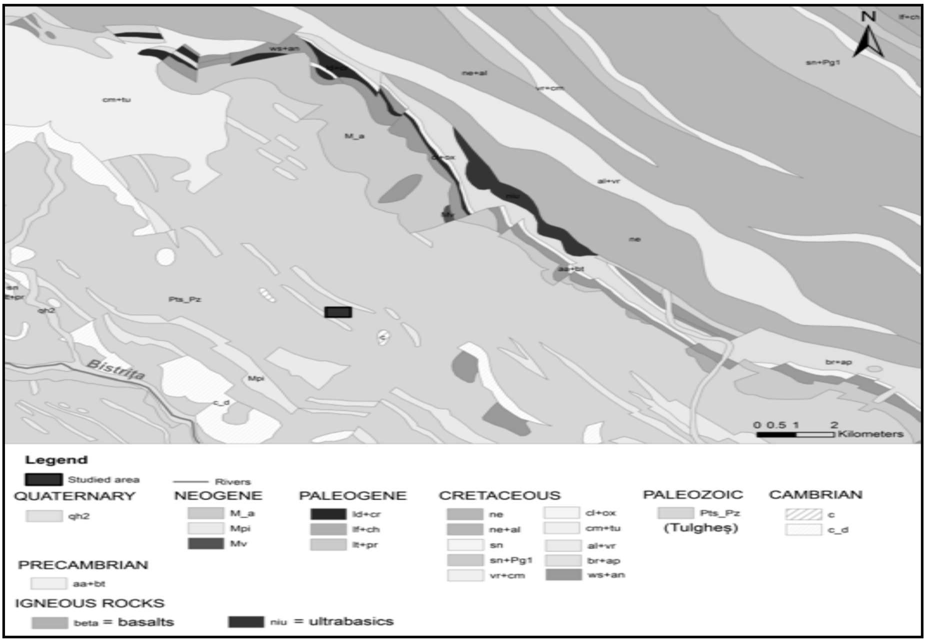

Fig. 1 Geological map of the studied area (modified after Ianovici et al., 1968).

the release of trace elements, whose accumulation in water and soil for a long period of time can lead to the environmental pollution.

Plants consume water and essential elements from soil and when they grow up in a contaminated soil they can develop different behaviours regarding the TE uptake. Generally, a healthy growth of plant involves low soil contents of available Cu and Zn as microelements, which at higher content could generate toxic effects. There are elements such as Ni that have a minimal contribution in plant nutrition while others such as As and Pb can induce toxic effects even at low concentrations. The plant ability to uptake TE from soil depends on the nutrition necessities, varying from species to species. The native plants from mining areas can show high tolerance for different TE content in soil. The taken up TE by plants from soil can accumulate at the roots level or translocate from root to the above ground tissues (stems, leaves, fruits). Thus, the plants can be grouped in three categories: excluder, indicator and accumulator (Sheoran et al., 2011). The excluder plants restrict the translocation of TE to the above ground parts, but can contain large amount in the root. The indicator plants accumulate TE in the above ground parts and reflect the substrate soil concentration. The accumulator plants concentrate TE in the above ground parts independently on their content in soil (Sheoran et al., 2011; Wahsha and Al-Rshaidat, 2014). The study of the trace elements in native species can be used to evaluate their availability in soil and to assess the toxicity risk for plants (Chopin and Alloway, 2007; Gjengedal et al., 2015).

The aim of the present study was to evaluate the distribution of Ni, Cu, Zn, Pb and As within the soil-plant system from Mănăila mining area (Eastern Carpathian Mountains), where the exploitation of ore in open pit and the improper waste dumps disposition can enhance the environmental pollution risk. The native plants growing on the mountain thin soil layer and extreme climatic conditions specifically for alpine steppe (1400 m altitude) is represented by lingonberry evergreen dwarf shrub (*Vaccinium vitis-idaea)*. Lingonberries are one of the most important shrubs that grow in forests and mountain hills across the Carpathians, especially on shady slopes and humid forest. The genus *Vaccinium* belongs to the *Ericaceae* family and consists of more than 150 species (Leahu et al., 2014). The leaves and fruit (found single or in clusters at the tips of branches) are harvested for medicinal extracts with antiseptic and diuretic properties. The berries rich in minerals and antioxidants are widely used for teas, jam, syrup and sauces.

2. Geological setting

The Mănăila ore deposit is located in Suceava County, North-East of Romania (Eastern Carpathians) and it is associated with Tg3 formation from the Tulgheş area which is a metamorphosed rhyolitic volcano-sedimentary unit (Fig. 1).

The Tulgheş Group represents an island arc complex related to the Easternmost part of the Avalonian microcontinent that rifted off Gondwana in the Lower Ordovician and was docked to the East European craton in the late Ordovician-Lower Silurian (Munteanu and Tatu, 2003). It was divided by Kräutner (1984) in five units, designated (from bottom to top) as Tg1–Tg5. The significant accumulations of metallic sulphides were associated with Tg3 and are shaped as massive strata, lenses, or bodies with disseminated metallic minerals (Kräutner, 1984). The mineralization of the studied area is characterized by the dominance of pyrite (FeS_2), associated with other ore minerals (sulphides), the most important of which are chalcopyrite ($CuFeS_2$), sphalerite (ZnS) and galena (PbS) (Moldoveanu et al., 2010).

3. Materials and methods

In August 2014, a number of 34 soil and plant samples was collected around Mănăila open pit (47°35’ N and 25°13’ E) (Fig. 2).

Soil samples, about 1 kg each, were collected at a depth of 0–15 cm, stored in ziplock bags than dried at 60°C and passed through a 2 mm sieve. An amount of soil and binder (synthetic raisin) at a ratio of 5:1, was mixed in an agate ball mill, for 10 minutes at a constant speed of 180 rpm in order to achieve the pellets (pressed powder disks). A pressed powder disk of 9 g weight was obtained for each sample. Total contents of trace elements were determined through energy-dispersive X-ray fluorescence spectroscopy (ED-XRF), using an Epsilon 5 Spectrometer model. The standardization was performed using 24 CRM (LKSD1–4, STSD1–4, Till1–4 SO1–4, JLk1–3, RT, RTH, GSD etc.). The exposure time was

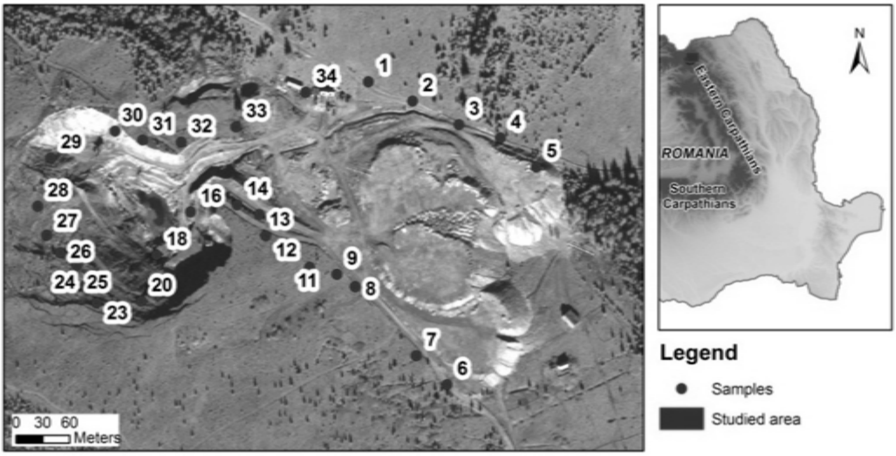

Fig. 2 Sample location–Mănăila ore deposit (Eastern Carpathians, Romania).

50 s, with the exception of As in which case the exposure time was 100 s. The lower limit of detection for measured elements is cca 2 $mg \cdot kg^{-1}$ (Cu, Pb), 1 $mg \cdot kg^{-1}$ for As. Quality control and quality assurance were assessed using the SO-4 certified reference material. The standard was measured after each 10 sample measurements.

Plant samples were collected from the same points as the soil samples and then were separated according to the organ type: roots, leaves and fruits. After separation, each sample was dried in an oven for 4 hours at a constant temperature of 105ºC. Before analysis, the dried plant material was finely grounded using an agate mill.

About 300 mg of each plant organ was weighed and placed into a Teflon reaction vessel with 10 ml of HNO_3. The microwave digestion method applied a temperature slope of 200ºC with a hold time of 10 min. The digests were diluted with deionized water up to 50 ml and stored in polyethylene containers until analysis. An ICP-MS 7700X (Agilent, Japan) was used to determine the trace elements. The optimal conditions indicated by the supplier were: radiofrequency power of 1550 W, carrier gas flow-rate of 1.01 $l \cdot min^{-1}$, plasma gas flow-rate of 15 $l \cdot min^{-1}$, auxiliary gas flow-rate of 0.70 $l \cdot min^{-1}$, integration time 0.3 s.

The geoaccumulation index (I_{geo}) was introduced by Müller (1969) and is computed by the following equation:

$$I_{geo} = \log_2 (C_n/1.5B_n) \qquad (1)$$

where: C_n – the concentration of the element in the enriched samples;

B_n – the background concentration of the element in the upper Earth's crust, according to Taylor and McLennan (1995).

According to Müller (1969) there are six descriptive classes of I_{geo}, as data of Table 1 show.

A plant ability to extract TE from soils can be estimated using the biocon-

Tab. 1 The I_{geo} classes used for interpretation

Class	Value	Soil quality
1	$I_{geo} \leq 0$	Uncontaminated
2	$0< I_{geo}< 2$	Uncontaminated to moderately contaminated
3	$2< I_{geo} <3$	Moderately contaminated
4	$3< I_{geo} <4$	Moderately to heavily contaminated
5	$4< I_{geo} <5$	Heavily contaminated
6	$5< I_{geo}$	Extremely contaminated

centration factor (BCF), which is defined as the ratio of metal concentration in roots and that in soil.

$$BCF = [TE_{roots}]/[TE_{soil}] \quad (2)$$

Baker (1981) classified plants in three groups based on the extent to which the concentration of an element in soil is reflected by the plant:

- *excluders*: BCF < 1;
- *indicators*: BCF = 1;
- *accumulators*: BCF > 1.

The ability of plant to transfer and accumulate TE from the roots to the above ground tissues is measured using the translocation factor (TF), which is defined as the ratio of TE concentration in leaves and that in roots (Mehes et al., 2013). Translocation of metals from roots to aboveground tissues is a crucial physiological process in an effective utilization of plant to remediate polluted sites (Majid et al., 2012, Rajoo et al., 2013).

$$TF = [TE_{aboveground}]/[TE_{roots}] \quad (3)$$

4. Results and discussions

4.1 Soil

The soil surrounding the ore deposit is young, poorly developed and populated by local spontaneous vegetation as herbs and shrubs specifically to the mountain area. The principal characteristic of the soil is the presence of the parent material at 15–20 cm below the surface. Soil acidity ($3.07 \leq pH \leq 5.11$) can be related to the weathering products of the parent material minerals (in particular pyrite) with a low amount of carbonates which should control the soil reaction.

The concentrations of Cu, As and Pb vary in a larger range than those of Ni and Zn in soil as standard deviation shows (Tab. 2), having a non-homogenous distribution. The TE abundances decrease in the following order: Zn > Pb > Cu > Ni > As.

In order to assess the potential toxicity risks, the soil TE concentrations were compared to the normal and threshold values for sensitive soils recommended by the Romanian legislation, given by the Order no. (756/1997) (Tab. 2). Nickel and zinc concentrations are included in the range of the legislative normal values of soil, while copper fits within the range between normal and alert values. Arsenic and lead exceed the alert threshold for most of the samples (41 % and 65 % of samples for As and Pb respectively). Thus, the potential toxicity risk can be considered higher for As, Pb and Cu especially for samples with values closed to AT.

The I_{geo} average values (Tab. 2) point out to a moderate contamination for As, Pb

Tab. 2 TE concentrations in soil from Mănăila area compared with Romanian legislative values (mg·kg^{-1}) and geo-acummulation index (Igeo)

	Ni	Cu	Zn	As	Pb
min	16.00	18.00	50.00	7.20	40.00
average	16.29	**42.65**	78.41	**14.97**	**69.44**
max	19.00	92.00	109.00	24.70	151.00
stdev	0.80	21.11	14.19	5.00	28.80
NV	20	20	100	5	20
AT	75	100	300	15	50
I_{geo}	0.94	1.08	0.79	**2.15**	**2.52**

NV = normal values, AT= alert threshold.

and Cu, while for Ni and Zn the contamination is not supported.

4.2. Plant

Several studies have investigated the impact of TE on wild plants growing in more or less polluted areas (e.g. Gentscheva et al., 2014; Lazăr et al., 2014; Gjengedal et al., 2015) and in most of the cases the contents vary in different parts of the plant. The process of metal uptake is different among species or varieties of the same species bringing to the different level of accumulation among plant tissues (Kabata-Pendias, 2011; Lazăr et al., 2014). Plant roots are expected to accumulate higher concentration of TE than the other organs of the same plant regardless of

Tab. 3 Selected TE concentrations (mg·kg^{-1}) in roots and leaves of *Vaccinium vitis idaea* collected from Mănăila area

	Ni	Cu	Zn	As	Pb
Roots					
minimum	8.56	21.35	37.06	0.14	8.03
average	**14.63**	**39.08**	**77.63**	**0.91**	**18.69**
maximum	36.06	55.52	108	1.89	30.35
median	10.6	39.52	76.17	0.97	17.55
Leaves					
minimum	5.3	12.65	35.3	0.01	2.77
average	**12.19**	**25.29**	**51.95**	**0.42**	**11.43**
maximum	39.8	52.44	88.5	1.49	31.16
median	8.14	22.55	50.68	0.32	8.01
NV*	0.1–5	5–30	27–150	1–1.7	5–10
TV*	10–100	20–100	100–400	5–20	30–300

* Kabata – Pendias (2011); NV= normal values, TV= toxic values.

their soil content (Gallagher et al., 2008).

The TE accumulation trend is almost the same for all lingonberry organs and decreases in the following order:

Zn > Cu > Pb > Ni > As for roots;
Zn > Cu > Ni > Pb > As for leaves and
Cu > Zn > Ni > Pb > As for fruits (Tab. 3).

The distribution of selected TE from soil to plant roots depends on the specificity of plant, nutritional needs and also bio-availability of elements from soil. The TE uptake from soil solution by roots can be both passive (nonmetabolic) and active (metabolic) (Kabata-Pendias, 2011). The high phytoextracted contents of Zn and Cu by lingonberry roots from Mănăila mining area (Tab. 3) could be related to the active uptake due to nutrition role of these elements. The high content of Ni at the roots level could be explained by a passive uptake from soil although its nutritional role is non-essential. Arsenic is retained in soil, with a slight uptake in the roots. Despite of the soil enrichment with Pb, the root uptake is less than 1/3 from total soil content.

The leaves and fruits of lingonberry show lower TE contents with respect to roots (Fig. 3). The Cu and Zn contents in leaves fit to the range of normal values for various species found in literature (e.g., Gjengedal et al., 2015). The Pb content in lingonberry leaves (Tab. 3) is slightly higher than the normal values, while those of As are lower. Exposed to higher soil Pb concentration, *Vaccinium vitis idaea* sp can restrict the transfer from roots to above ground parts. The potentially toxic effect can be presumed in case of Ni which average content exceed the lower limit of toxic range for leaves of different plant species (Tab. 3).

The berries are enriched in Cu, Zn and Pb exceeding the allowed limits (5 mg·kg^{-1} for Cu and Zn, 0.1–0.2 mg·kg^{-1} for Pb) for fruits as part of the food chain (Order no. 293/640/2001-1/2002). The contents of Ni and As in berries are lower than the allowed limits.

The lingoberry fruits from Mănăila show typically high TE contents comparable with those of other ever green shrubs from *Ericaceae* family (blueberry, bilberry and crowberry) from different mining and smelter areas (Demczuk and Garbiec, 2009; Pavlova and Karadjova, 2013; Gentscheva et al., 2014) (Tab. 4). Thus, also the berries, annual part of the perennial shrub, reflect the TE abundance in soil on which the plant is adapted to grow without any visual symptom of deficiency or toxicity effect.

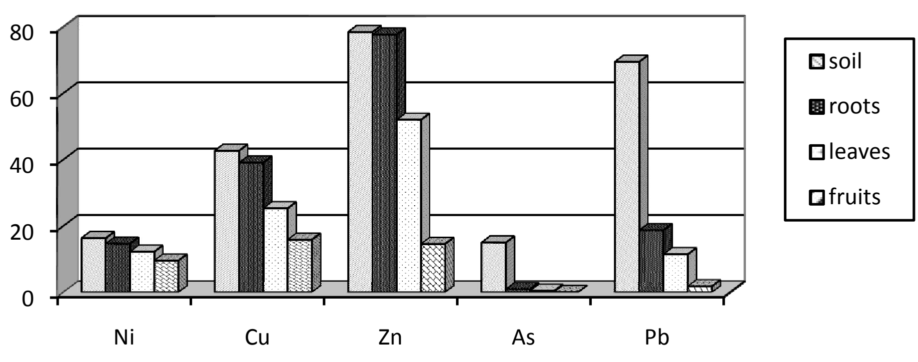

Fig.3 Distribution of selected TE (mg·kg^{-1}) among soil and lingonberry organs.

Tab. 4 Selected TE concentrations ($mg \cdot kg^{-1}$) in berries from Mănăila and other areas

		Ni	Cu	Zn	As	Pb
Mănăila, Romania						
	lingonberry					
	minimum	4.66	8.56	3.11	0.01	0.39
	average	**9.52**	**15.83**	**14.56**	**0.08**	**1.76**
	maximum	23.9	31.63	32.7	0.19	5.73
Plovdiv, Bulgaria						
	lingonberry	0.12	3.25	7.39	-	0.13
SW Poland						
	blueberry	0.52	6.87	10.3	-	1.34
Rila Mt., Bulgaria						
	crowberry	8	8.4	37.5	-	1.3

The accumulation trends of the selected TE in soil and plant materials (root and aboveground tissues) (Tabs. 3 and 4) decrease in the following order: Soil > root > leaf > berry (Fig. 3).

The mechanism of transfer and accumulation behaviors for plants have been described and discussed by several authors (Baker, 1981; Pilon-Smits, 2005; Sheoran et al., 2011), concluding the relevance of using two different ratios:(1) Bioconcentration factor (BCF: roots/soil) provides information on the availability of a certain element in soil; (2) Translocation factor (TF: leaves/roots) gives information about elemental mobility among plant organs.

According to Baker (1981) a bioconcentration factor higher than unity is characteristic for the accumulator plants. Representative extraction from soil was observed only for Cu and Zn (Tab. 5) and therefore *Vaccinium vitis idaea* can be considered an indicator plant for these TE. The average BCF values for Ni, As and Pb are lower than unity (Tab. 5) and reflect the excluder behaviour of lingonberry. Bioconcentration factors for Cu, Zn and subordinately Ni suggest a higher availability in soil than for As and Pb. Taking into account the acidity of soil reaction ($3.07 \leq pH \leq 5.11$) and the soil abundances, the availability of Cu and Zn is supported in the studied area.

Plant species exhibiting TF greater than one are suitable for phytoextraction (Pilon-Smits, 2005). The TF average value determined for the roots to aboveground system indicates that Zn, As and Pb are accumulated to the root level, and their translocation to the others organs is restricted. Consequently, the lingonberry is a tolerant plant (Yoon et al., 2006) for Zn, As and Pb. For Ni and Cu the TF value points out an accumulation at the aboveground plant parts (Tab. 5).

Both BCF and TF higher than unit suggest the plant potential for phytoextraction (Yoon et al., 2006), condition fulfilled only for Cu in our study. When

BCF > 1 and TF < 1, the plant has phytostabilisation potential, as lingonberry shows for Zn.

The low BCF (Ni, As, Pb) and TF (Zn, As, Pb) values denote the limited ability of lingonberry to accumulate and translocate these TE from soil to above ground parts.

Tab. 5 Average values of bioconcentration and translocation factors in lingonberry

	BCF	TF
Ni	0.85	**1.88**
Cu	**1.05**	**1.17**
Zn	**1.03**	0.83
As	0.07	0.64
Pb	0.31	0.85

5.Conclusions

Plants growing on metal-enriched soils denote a particular interest since are genetically tolerant to high metal concentrations and have the ability to adapt excellently to extreme conditions.

According to the Romanian law in force, Cu and As soil concentrations from the surroundings soils of the Mănăila ore deposit are higher than the normal values but still below the alert threshold, while Ni and Zn concentrations are in the range of the normal values. Lead was the most abundant soil contaminant surrounding the mine, exceeding the ranges considered toxic to normal soils.

The presence of TE (Cu, Pb, As) higher than the normal concentrations in soil is a consequence of natural geochemical background upon soil formation and development and subsequently of the mining activities from the area. For special cases such as the ore deposits, the legislative values should be reviewed and restored for a smaller well-delimited perimeter.

The soil TE enrichment as response of parent material composition is also reflected within the soil-plant system.

The *Vaccinium vitis idaea* plant species with curative properties, natively and widely distributed in the mining area was selected as part of the studied soil-plant system. According to BCF values, the lingonberry behaves as excluder for Ni, As and Pb, being an indicator for Cu and Zn. The tolerance capacity is found for Zn, Pb and As on the base of TF values. The translocation of Ni and Cu in above ground parts denotes a higher mobility of these TE among different plant organs, while Zn, As and Pb are preferentially retained in the roots. The TE accumulation in plant is not always correlated with toxicity, especially in the case of essential elements for plant metabolism. The Cu, Zn and Pb enrichment above the normal values for food chain can hinder the curative properties of berries. The lingonberry from Mănăila area confirms the good capacity of adaptation under the conditions of acid soil reaction and high content of TE.

The present work supplies the first report on selected TE accumulation in *Vaccinium vitis idaea* from the studied area, providing a starting point for the potentially use of natural phytostabilisation.

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