



RISK OF CONTAMINATION OF WILD BERRIES FROM UPPER ORAVA REGION BY CADMIUM

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ABSTRACT

The upper Orava region is located at the North Slovakia, near of potential sources of environmental contamination due by mining of coal, zinc and lead ores. The aim of the study was to evaluate the risk of consumption of wild forest fruit from Upper Orava region from the aspect of cadmium content. Ten sampling points were found by random search. From these points samples of soil, leaves and fruits of wild berries (9 samples of blueberries *Vaccinium Myrtillus* and 1 sample of strawberries *Fragaria Vesca*) were collected. In soil samples the active soil reaction (pH/H₂O) ranged from 3.53 (strong acidity) to 4.56 (extremely strong acidity), and the determined percentage of humus ranged from 1.66 (low humic soil) to 4.90 (high humic soil). In two soil samples the total content of cadmium determined in soil extracts by *aqua regia* exceeded limit 0.70 mg.kg⁻¹ given by the legislation in the Slovak Republic. In three soil samples the determined content of cadmium mobile forms determined in soil extracts by NH₄NO₃ exceeded the limit 0.10 mg.kg⁻¹. The content of Cd determined in leaves as well as in fruits was evaluated according to Food Codex of the Slovak Republic. Only in one sample of leaf samples the limit 1.00 mg.kg⁻¹ was exceeded. The other leaf samples are safely when used as an ingredient in tea mixtures. On the other hand even in 7 fruit samples the limit 0.05 mg.kg⁻¹ was exceeded. This fruit can pose a risk for the human organism when is directly consumed as well as may negatively affect the human health when is used as raw materials in the food industry.

Keywords: Upper Orava; heavy metals; cadmium; soil; wild berries; leaves

INTRODUCTION

The upper Orava region is located on the North Slovakia, surrounded by Orava reservoir, near South Poland border (under 20 km).

In South Poland, close to the Silesia-Cracow region, seven of twenty-seven ecological hazardous areas of Poland are located. Mining of coal, zinc and lead ores cause a great additional threat to the natural environment and the human population. A considerable set of data is available on the air pollution in the city of Cracow and the whole area of Katowice voivodship (Godzik, 1993).

These two towns are from the investigated area 120 km respectively 140 km far. So, the air pollution caused by long-distance transfer from these industrial centres can be a potential source of contamination by heavy metals in the environment of the Upper Orava region.

Heavy metal pollution is released into the environment by various anthropogenic activities, such as industrial manufacturing processes, domestic refuse and waste materials (Guala et al., 2010). Soils contaminated with heavy metals cause many environmental and human health problems calling for an effective technological solution. Many sites around the world remain contaminated because it is expensive to clean them up by available technologies.

Anthropogenic pollution caused by heavy metals entering into the plant is subsequently passed into the food chain with the consequence in hazards to human health (Křížová, 2009).

Cadmium, a by-product of zinc production, is one of the most toxic elements to which man can be exposed at work or in the environment. Once absorbed, Cd is efficiently retained in the human body, in which it accumulates throughout life. Cd is primarily toxic to the kidney, but it can also cause prostate and renal cancer as well as the bone demineralization (Bernard, 2008).

In Slovakia there are many areas with natural resources of some forest fruit, such as raspberries, blueberries, blackberries, lingonberries, etc. These fruits contain vitamins, minerals and polyphenolics compounds which are resistant against unsuitable climatic conditions and they can adapt to more severe soil-climatic conditions. Upper Orava region belongs to the Slovakian areas with an occurrence of wild forest berries. Forest fruit such as blueberries, forest strawberries, raspberries, cranberries etc. are often collected by people for their flavor, color and bioactive components which have a positive effect to the human health (Nile and Park 2014). According to Häkkinen et al., (1999) small forest fruits, both wild or bred, are traditional part of Finnish consumers, with significant content of biological active non-nutrients, but also of essential nutritive components. The essential elements (K, Ca, P, Mg, Al, B, Cu, Fe, Na, Mn and Zn) are important components of highbush blueberries, while suitable fact for human organism is low content of Na (Bushway et al., 2006). Prior et al., (1998) consider blueberries as one of the richest sources of antioxidant

phytonutrients, while composition and content of phenolic compounds in blueberries have changed in relation to variety, period, as well as to locality of growing (Giovannelli and Burati, 2009).

The aim of the study was to evaluate the risk of consumption of wild forest fruit from Upper Orava region from the aspect of Cd content.

MATERIAL AND METHODOLOGY

The experiment was realized in region Upper Orava, in cadasters of villages: Malé Borovce, Habovka, Zábiedovo, Brezová, Vitanová and area near Orava reservoir (Figure 1). Samples were collected in June 2014. The exact coordinates of sampling sites are presented in Table 1. The average annual temperature is 6 °C (12.5 °C during vegetation) and the average annual rainfall is 800 – 900 mm (550 mm during vegetation).

Samples of soil, fruits (9 samples of blueberries (*Vaccinium Myrtilus*) and 1 sample of wild strawberries (*Fragaria vesca*) and leaves (can be used as a tea mixture) were taken from individual sampling points. The soil samples were taken from the upper horizon.

The active soil reaction pH/H₂O was determined electrometrically (691 pH Meter, Metrohm, Swiss), and content of oxidizable carbon (C_{OX}, %) was determined using volumetric method according to Tjurin (H₂SO₄: Merck, Germany, K₂Cr₂O₇: Merck, Germany; (NH₄)₂Fe(SO₄)₂•6H₂O: Merck, Germany) while a content of humus (Hum., %) was calculated from C_{OX} content.

Pseudototal content of cadmium including all the form besides residual metal fraction was assessed in soil extract by *aqua regia* (HCl: CentralChem, Slovakia, HNO₃: Merck, Germany) and content of mobile forms in soil extract by NH₄NO₃ (c = 1 mol.dm⁻³, Merck, Germany)). Used analytical method was flame AAS (AAS Varian AA

Spectr DUO 240 FS/240Z/UltrAA, Varian, Australia).

The determined values were compared with limits given by European Commission Regulation no. 1881/2006 as well as Slovak decree no. 220/2004 of coll.

Homogenized berry samples (4 g) were mineralized in a closed system of microwave digestion using Mars X-Press 5 (CEM Corp., USA) in a mixture of 5 mL HNO₃ (Suprapur, Merc, Germany) and 5 mL deionized water (0.054 μS.cm⁻¹) from Simplicity 185 (Millipore, UK). Metal determinations were performed in a Varian AA240Z (Varian, Australia) atomic absorption spectrometer with Zeeman background correction. The graphite furnace technique was used for the Cd determination. The obtained results were expressed as mg.kg⁻¹ FM. Gained results were evaluated according to hygienic limit for Cd content in fruit given by the Food Codex of the Slovak Republic.

Each analysis was done in 4 repetitions.

Statistical processing of the results was carried out using software Statgraphics Centurion XVI.I. One-way analysis of variance (α = 0.05) was used. Mean comparisons between investigated parameters were done by the LSD test.

RESULTS AND DISCUSSION

Soil samples:

In soil samples the active soil reaction (pH/H₂O) ranged from 3.53 (strong acidity) to 4.56 (extremely strong acidity), data are available in Table 1. Römken et al. (1998) and Barančíková (1998) reported that Cd and Zn solution concentrations were higher in forest soils and were strongly increased below pH 5.5 even despite the low total metal content. Kawabata et al., (2011) presented similar pH values to our results in soil used for blueberry production. On the other hand, Maliníková et al., (2013) presented higher pH values of forest soil (5.28-7.67). The

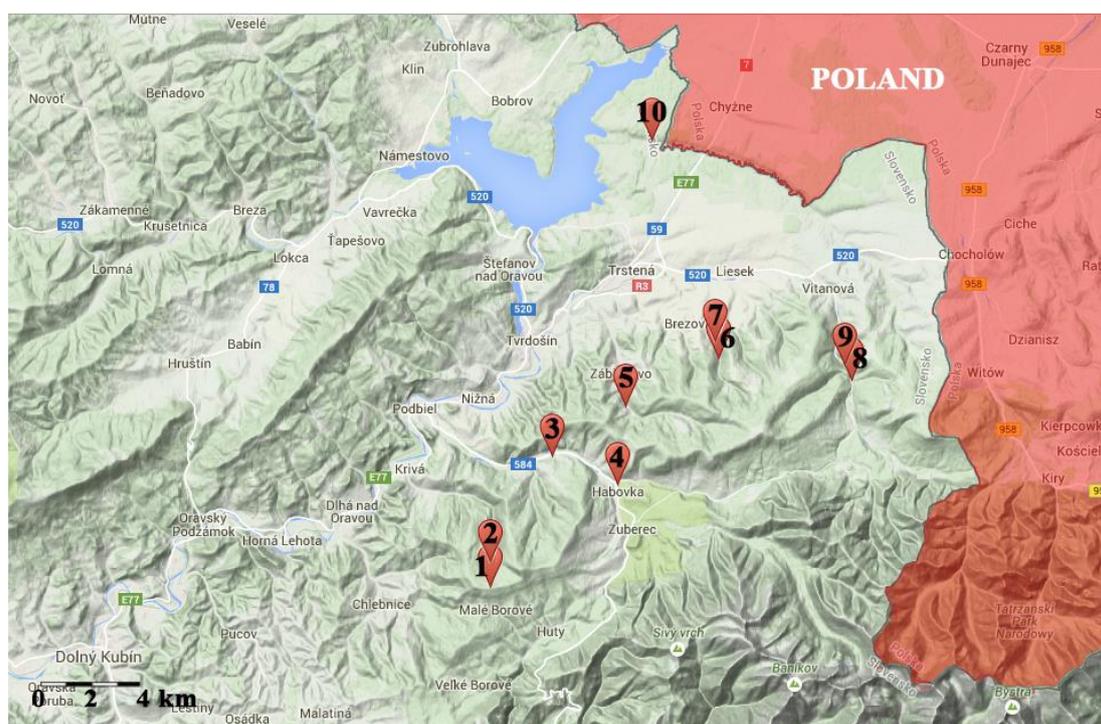


Figure 1 Investigated area and sampling points.

% of humus in soil samples was in range 1.66 (low humic soil) to 4.90 (high humic soil), data are available in Table 1. **Fanrong (2011)** reported a positive correlation between organic matter content and bioavailability of heavy metals in soil. The total cadmium content in soil samples (determined in soil extracts by *aqua regia*) was in the range 0.12 – 1.45 mg.kg⁻¹ (Table 1). In two soil samples (Samples no. 1 and 3) the total content of cadmium exceeded the limit value 0.70 mg.kg⁻¹ given by **European Commission Regulation no. 1881/2006** as well as **Slovak decree no. 220/2004 of coll.** (1.45 and 0.73 mg.kg⁻¹ respectively). Several studies focused on the monitoring of the environmental contamination of the soils in Slovakia were published. **Musilová et al., (2015), Vilček et al., (2012), Tomáš et al., (2009)** determined the total Cd content in soil samples in intervals 0.65 – 6.73 mg.kg⁻¹, 0.36 – 12.26 mg.kg⁻¹, 0.34 – 0.40 mg.kg⁻¹ respectively. **Taraškevičius et al., (2013)** presented values of the Cd contents in soil extracts of *aqua regia* in some European soils in range 0.07 – 12.8 mg.kg⁻¹. In Table 1 also values of content of mobile cadmium forms determined in soil extracts by NH₄NO₃ are presented. The values were compared to the limit value 0.10 mg.kg⁻¹ given by **European Commission Regulation no. 1881/2006** as well as **Slovak decree no. 220/2004 of coll.** In Sample no. 1 (0.15 mg.kg⁻¹), Sample no. 2 (0.10 mg.kg⁻¹) and Sample no. 8 (0.13 mg.kg⁻¹) the determined content of cadmium mobile forms exceeded the limit. **Musilová et al. (2015), Vilček et al. (2012),**

Tomáš et al. (2009) also determined contents of cadmium mobile forms in soils of Slovakia. They determined values in range 0.029 – 0.236 mg.kg⁻¹, 0.02 – 0.78 mg.kg⁻¹, 0.5 – 0.7 mg.kg⁻¹ respectively, similar to our results.

Leaf samples:

The determined Cd content in leaves of investigated forest fruit (Table 2) was compared to hygienic limit 1.00 mg.kg⁻¹ for Cd content in tea mixtures given by the **Food Codex of the Slovak Republic**. Only in Sample no. 6 (2.02 mg.kg⁻¹ DM) the limit was exceeded, all other leaf samples have the determined Cd content under the hygienic limit. Despite that finding our results indicate a significantly higher degree of Cd accumulation in leaves than in fruits.

Fruit samples:

On the other hand even in 8 fruit samples the limit 0.05 mg.kg⁻¹ given for small berries by the **Food Codex of the Slovak Republic** was exceeded (Table 2), whereas in 1 Sample (no. 2) the Cd content was lower than the detection limit. **Von Hoffen et al., (2014)** determined Cd content of blackberries in range 0.004 – 0.18 mg.kg⁻¹ FM. On the other hand, **Reimann et al., (2001)** presented significantly lower values (0.009 mg.kg⁻¹) of Cd content in blueberries compared to our results. According to **Wieczorek et al., (2010)** the concentration of Cd in wild berries, ranged from 6 to 49 µg.kg⁻¹ fresh weight.

Cadmium (Cd) is a toxic heavy metal that can accumulate in the human body and the environment for

Table 1. Analysis of soil samples from Upper Orava region, July 2014.

No.	Sample	GPS coordinates	Active soil reaction [pH H ₂ O]	Cox [%]	% of humus in soil samples [%]	Total Cd content in soil samples [mg.kg ⁻¹ DM]		Content of mobile forms of Cd in soil samples [mg.kg ⁻¹ DM]	
						Average	SD	Average	SD
1	Blueberries	N 49° 14.186'' E 19° 31.819''	3.53	2.84	4.90	1.45	±0.01	0.15	±0.02
2	Blueberries	N 49° 14.791'' E 19° 31.826''	4.11	1.79	3.09	0.37	±0.01	0.10	±0.01
3	Blueberries	N 49° 17.265'' E 19° 33.997''	4.48	1.54	2.66	0.73	±0.01	0.08	±0.00
4	Blueberries	N 49° 16.613'' E 19° 36.374''	4.56	2.53	4.36	0.34	±0.01	0.03	±0.01
5	Blueberries	N 49° 18.442'' E 19° 36.637''	4.23	1.56	2.69	0.27	±0.02	0.05	±0.01
6	Blueberries	N 49° 19.607'' E 19° 40.058''	4.31	1.39	2.39	0.25	±0.01	0.05	±0.01
7	Blueberries	N 49° 19.957'' E 19° 39.881''	4.35	1.47	2.54	0.14	±0.01	0.06	±0.02
8	Blueberries	N 49° 19.053'' E 19° 44.552''	3.89	2.26	3.90	0.57	±0.02	0.13	±0.02
9	Blueberries	N 49° 19.412'' E 19° 44.520''	4.44	0.97	1.66	0.12	±0.01	0.07	±0.01
10	Strawberries	N 49° 24.775'' E 19° 37.613''	4.00	1.61	2.78	0.17	±0.01	0.09	±0.01
Limit value *						0.70		0.10	

NOTE: * limit given by European Commission Regulation no. 1881/2006 as well as Slovak decree no. 220/2004 of coll.

Table 2. Cd contents in samples collected in Upper Orava region (fruits and leaves) and transfer factors (soil-fruit and soil-leaves), July 2014.

No.	Sample	Cd content in fruit samples [mg.kg ⁻¹ FM]		Cd content in leaves samples [mg.kg ⁻¹ DM]		Transfer factors soil - fruit	Transfer factors soil - leaves
		Average	SD	Average	SD		
1	Blueberries	0.09 e	±0.01	0.60 cd	±0.01	1.70	0.25
2	Blueberries	UDL**	-	0.89e	±0.02	-	0.11
3	Blueberries	0.05 c	±0.01	0.57 bc	±0.01	1.73	0.14
4	Blueberries	0.08 e	±0.00	0.92 e	±0.01	0.30	0.03
5	Blueberries	0.05 c	±0.01	0.52 a	±0.01	0.92	0.09
6	Blueberries	0.07 d	±0.01	2.02 f	±0.06	0.78	0.03
7	Blueberries	0.04 b	±0.01	0.61 d	±0.03	1.54	0.09
8	Blueberries	0.05 bc	±0.01	0.56 b	±0.01	2.82	0.23
9	Blueberries	0.05 c	±0.01	0.60 d	±0.02	1.53	0.12
10	Strawberries	0.07	±0.00	0.97	±0.01	1.35	0.09
	P-value	0.0000		0.0000			
	F-ratio	42.19		1413.93			
	Limit value *	0.05		1.00			

NOTE: Average values marked with the same letter are not significantly different ($p < 0.05$)

* limit value given by Food Codex of Slovakia.

** Cd content under detection limit.

lengthy periods (Zhang et al., 2014) and due its exposure the toxic effects in a variety of structures such as kidneys, liver and central nervous system including proteinuria, glucosuria, and aminoaciduria with final renal dysfunction are confirmed (Xu et al., 2013).

The high concentration of heavy metals in soils is usually reflected by higher concentrations of metals in plants, and consequently in animal and human bodies (Buszewski et al., 2000).

The transfer of soil pollutants into the plants causes many physiological disorders. The degree of heavy metal mobility, activity and bioavailability and consequently plant uptake is influenced by many factors such soil reaction, temperature, redox potential, cation exchange capacity of solid phase, competition with other metal ions, ligation by anions, composition and quantity of the soil solution (Wopereis et al., 1988).

To characterize quantitatively the transfer of an element from soil to plant, the soil-plant Partition Coefficient or Transfer Factor (TF) or Concentration Ratio or Biological Accumulation Coefficient (BAC) that expresses the ratio of contaminant concentration in plant parts to concentration in dry soil is used (Chojnacka et al., 2005). In Table 2 the calculated values of TF are presented. Generally, the transfer factors calculated for soil-leaves transfer were low. This may be because only the accumulation of metals in the leaves were studied more metals could have accumulated in the root (Olayinka et al., 2011). The transfer factors calculated for soil-fruit transfer were higher. The higher the value of the TF, the more mobile/available the metal is (Olayinka et al., 2011).

CONCLUSION

In two soil samples from the Upper Orava region the total content of cadmium exceeded limit 0.70 mg.kg⁻¹ and in three soil samples the determined content of cadmium mobile forms exceeded the limit 0.10 mg.kg⁻¹, which can be put into context with extremely strong soil reaction. This factor increases the release of mobile forms in the soil environment. Wild berries such as blueberries (*Vaccinium Myrtillus*) and wild strawberries (*Fragaria Vesca*) have a positive effect to the human health because of their content of bioactive and chemoprotective components as well as an antioxidant activity. On the other hand it is necessary to monitor content of cadmium or other heavy metals in this fruit which is affected by soil-ecological conditions. Heavy metals become toxic for the human organism, when they entering into the food chain. Eating wild berries from the region of Upper Orava may present a potential risk for the human health. Our results indicate a significantly higher degree of Cd accumulation in leaves than in fruits, even though the limit for tea mixture was exceeded only in one sample.

It is necessary to monitor the soil content of hazardous elements in territory of Upper Orava as well as their transfer into plants and the food chain because of food safety.

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