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Evaluation of urban contamination with Co, Ni and Pb in three urban parks in Serbia using pine (*Pinus nigra* Arnold) needles and urban topsoil

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Abstract

Increasing industrialization and human activities intensify the emission of various pollutants, including metals and other harmful substances into the environment. Concentration of Co, Ni and Pb were determined in the surface layer of soil (0-20 cm) and in 1- and 2-year old needles of Pinus nigra in urban parks in three cities in Serbia exposed to different pollution sources. Contamination factor was used to assess potential ecological risk in urban topsoil. Analysis showed that Ni concentrations in soil surpass the target value of 35 mg kg^{-1} but are below background values for selected sampling sites, which indicates its geological origin. However, Pb content in all examined soils surpassed the background values, and in Smederevo the target value of 85 mg kg^{-1}. This was confirmed by contamination factor that indicates moderate contamination and anthropogenic influence. Element content in both 1- and 2-year old needles was within normal values for plant tissues. Although results obtained in this study suggest low contamination in needles, it should be noted that anthropogenic and industrial pollution causes alkalinization of soils in examined parks, which consequently leads to immobilization of elements in soils.

Key words: *urban soils, heavy metals, contamination factor, Pinus nigra*

Introduction

Rapid urbanization and industrialization, as well as high population density has led to an increasing level of pollution in urban environment. Urban areas are exposed to anthropogenic contaminants released from both stationary (power plants, various industries and waste disposal) and mobile (traffic) sources [1]. Due to the toxic, persistant and non biodegradable properties, heavy metals represent aggressive pollutants that are most frequently emitted and transported in the form of particles [2, 3, 4].

Soils are more sensitive to pollution in relation to other components of the natural environment because they do not undergo dilution or dispersion like water and air [5]. For this reason metals in soil can persist long time after their introduction. Assessment of soil contamination is mostly based on the degree of contamination using various pollution indices, which is why identification of hazardous elements is essential in determining the pollution status of urban soils [6, 7].

Long term soil contamination may be assessed by using plants as biomonitors [8]. The main advantages of using plants are the greater availability of biological material, the simplicity of species identification, sampling and ubiquity of some genera, which make it possible to cover large areas [9]. Austrian pine (*Pinus nigra* Arnold) is considered as a good environmental biomonitor [10] given the fact that it is resistant to drought and wind, and it tolerates urban conditions well [11]. Its needles have a thick epicuticular was layer and are particularly sensitive to environmental pollution [10, 12].

The objectives of this study were: determination of the total concentrations of cobalt (Co), nickel (Ni) and lead (Pb) in urban soils of three parks in three Serbian cities; determination of contamination factor (Cf) and assessment of the extent of the anthropogenic influence; to estimate total content of Co, Ni and Pb in 1- and 2-year old needles and difference in terms of their accumulation ability.

Materials and methods

Sampling sites

The research sites were municipal parks in three cities in Serbia that are exposed to airborne and heavy metal pollution from various industrial activities and heavy traffic. In Smederevo the dominant pollution source is the Smederevo Steelworks (Železara Smederevo); in Obrenovac the major sources of pollution are two thermoelectric power plants—'Nikola Tesla A' and 'Nikola Tesla B', as well as two ash disposal sites; in Belgrade the dominant source of pollution is heavy traffic. Sampling was performed in municipal parks in each city: in Smederevo from the Park 'Tri heroja', which is located 7 km southeast from the Smederevo Steelworks; in Obrenovac from the Park 'Trg Dr Zoran Đinđić' that is about 4 km southeast from the power plant 'Nikola Tesla A' and about 15 km from the 'Nikola Tesla B'; in Belgrade from the Park 'Pionirski Park' which is within 500 m away from several major traffic roads.
The research was carried out in June 2012.

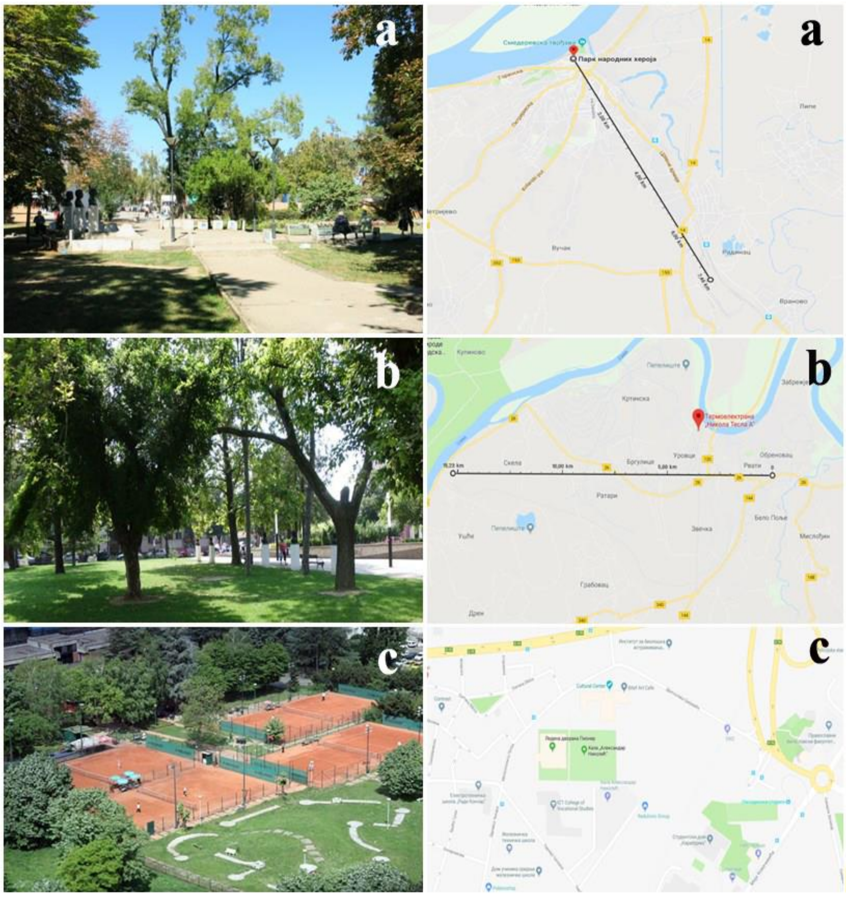

Fig 1: Selected sampling sites and their distance from the main source of pollution

Sampling and element concentration analysis

Soil samples

Composite topsoil samples at a depth of 0–20 cm were collected around each sampled *Pinus nigra* tree at five random locations. Stones and foreign objects were removed by hand, while soil samples were stored in clean polyethylene bags. Soil samples were first air dried for 10 days, then in a dryer (Binder, Tuttlingen, Germany) at a temperature of 105° C, and afterwards sieved through a stainless steel sieve with a mesh diameter of 2 mm.

Soil pH in an aqueous solution was measured using a glass electrode (1:2.5 soil–water ratio), after agitating the samples for approximately 30 min [13]. In order to determine total element concentrations, soil samples (0.3 g) were digested in a microwave (CEM, 39 MDS-2000), using the USEPA 3051A method (USEPA 1996). Five replicates were performed for each sample, while total element concentrations were determined with inductively coupled plasma optical emission spectrometry (ICP-OES) (SpectroGenesis Genesis Fee, Spectro-Analytical Instruments GmbH, Kleve, Germany). Quality control for soil was carried out using standard soil reference material—ERMCC141 loam soil obtained from the IRMM (Institute for Reference Materials and Measurements, Geel, Belgium) and certified by the EC-JRC (European Commission-Joint Research Centre). Concentrations of all the measured elements are expressed in milligrams per kilogram of dry weight (mg kg^{-1} d.w.). The limits of detection were: Co-0.000653, Ni-0.00117, Pb-0.016 mg kg^{-1}.

For assessment of contamination level of urban soil samples, contamination factor (Cf) was used. It is determined by following equation:

$$Cf = {}^{Cm}/_{Bm}$$

where Cf represents the contamination factor of the element of interest, Cm is the concentration of the element in tested sample, while Bm depicts background concentration in soil. Contamination factor that is less than 1 refers to low contamination, 1<Cf <3 represents moderate contamination, 3<Cf <6 means considerable contamination, while Cf>6 represents very high contamination [14].

Plant samples

Element content was also determined in the needles of *P. nigra* collected from five randomly chosen trees about the same age at each sampling site. Needle samples were taken uniformly from each tree from different quarters of the tree crown in all directions around the tree with stainless steel scissors. The samples from each tree in every location were mixed into a composite sample (10–20 g), taking into consideration the age of the needles (separate 1- and 2-year-old samples), resulting in one composite needle sample per sampling site. All plant samples were air dried for 10 days at room temperature and then dried to a constant weight at 65° C (Binder, Tuttlingen, Germany). For the element concentration analysis, plant samples (0.3 g) were digested in a microwave (CEM, 39 MDS-2000), using the USEPA 3052 method (USEPA 1996). Five replicates were performed for each sample. Quality control for leaves was performed using standard leaf reference material—BCR-100 beech leaves obtained from the IRMM (Institute for Reference Materials and Measurements, Geel, Belgium) and certified by the ECJRC (European Commission-Joint Research Centre). All the limits of detection for plant samples were the same as for soil samples. Element concentrations were expressed in milligrams per kilogram of dry leaf weight (mg kg^{-1} d.w.).

Figure 2: Pinus nigra

Results and discussion

Total element, background concentration, contamination factor and pH values at selected sampling sites are given in Table 1, and content of Co, Ni and Pb in Pinus nigra needles are shown in Table 2. Concentrations of Co in selected soil samples varied in narrow range between 5.70 mg kg^{-1} measured in Belgrade to 7.96 mg kg^{-1} in Obrenovac, and were around mean proposed values for worldwide sandy soils of 5.5 mg kg^{-1} [15], below background concentrations for examined sites (Table 1) and below target values of 9 mg kg^{-1} established by National legislation [16].
Geochemical cycle of Co closely resembles cycling of Al, Fe and Mn which is why Co easily interacts with all metals associated with Fe [15]. Cobalt origin in soils is mainly derived from parent materials, while contamination of soils is mainly caused by mining and smelting activity, fertilizer use and sewage sludge spreading [17, 18]. Cobalt (II) is dominant form of Co appearance in nature and it is least mobile under neutral conditions, but as the environment become acidic it becomes available [19, 20]. In alkaline conditions which were measured at all sampling sites it is expected that Co is immobilized. The bioavailability and toxicity of Co is affected by physicochemical properties of the soil like pH, organic matter and soil texture [18, 21]. The reduction of soil Eh and a decrease in soil pH can result can result in the solubilization of precipitated or adsorbed Co [15]. However, adequate amounts of Co in soil are essential to its biological activity since Co represents a component of vitamin B12 [22], especially to grazing animals (ruminants). The results obtained in this study for the total element concentration, as well as contamination factor (<1) suggest that there is no evident contamination of soil originating from Co.
Uptake of Co in plants is highly controlled by both soil factors and ability of plants to absorb it. Plants can accumulate small amounts of Co from the soil, however there is limited literature base relating to risk or toxicity of Co to higher plants [23]. It is though that Co has low mobility in plants, which can

restrict its transport from roots to shoots [23, 24]. Cobalt content in both needle types at all sampling sites varied in narrow range from 0.92 mg kg^{-1} in Smederevo up to 1.35 mg kg^{-1} in Belgrade. The current research showed that Co content did not depend on age of needles given the fact that both needle years contained equal amount of Co. Sufficient or normal concentrations in leaves are considered to range from 0.02-1 mg kg^{-1} according to Kabata-Pendias and Pendias [22], which coincides with our results. This study confirmed that Co has limited mobility and poor transfer both from soil to shoots, as well as within plant parts.

Nickel content in selected soil samples varied in wide range from 35.19 mg kg^{-1} in Belgrade to 118.69 mg kg^{-1} in Smederevo. Soils throughout the world contain Ni within the broad range from 0.2-452 mg kg^{-1}, while the highest Ni content is usually reported for clay and loamy soils [22]. Soil samples from Smederevo surpassed background concentrations, while the samples from Belgrade were the only ones that were within set target values of 35 mg kg^{-1} (Table 1) established by National legislation [16].

Nickel is ubiquitous element in the Earth's crust that has similar distribution trends like Fe, but is also closely related to Co in both chemical and biochemical properties. It is stable in aqueous solution which is why it can be easily transferred to greater distances [5, 22]. The Ni content in soil is highly dependent on its content in parent material, however in surface soils it also reflects anthropogenic pollution given the fact that it represents one of the most serious environmental contaminants that is released in processes of metal, alloy, Ni-Cd batteries and electronic component production. Although literature confirms that soils in Serbia are characterized by high Ni content that is of geological origin [25], Ni concentration measured in samples from Smederevo, which were above the background and target values, are quite likely the consequence of industrial emissions, presumably Smederevo smelter. This was furthermore confirmed by contamination factor (1.15), which indicates moderate contamination. Results of previous researches that were conducted in the wider area of Belgrade showed high variation in Ni content (54-112 mg kg^{-1}), which greatly depended on the pollution source and its proximity [25, 26, 27, 28].

Literature data on the Ni significance and its essence in living beings are contradictory. While Adriano [5] considers it necessary due to the fact it represents component of the enzyme, Kabata-Pendias and Pendias [22] argue that there is no clear proof of its essentiality. Its accumulation in plants depends on both pedological and plant characteristics, but most pronounced factor is the influence of the soil pH [22]. Nickel content in needle samples varied in narrow range between sampling sites and was slightly higher in one year old needles in relation to two years old. Despite high concentrations that were measured in soil samples, results obtained in both year needles of Pinus nigra were within normal values for plant tissues which are considered to be 3.7 mg kg^{-1}, which is quite likely the consequence of alkaline conditions.

Lead content in selected soil samples varied in wide range from 56.39 mg kg^{-1} in Obrenovac to 118.63 mg kg^{-1} in Smederevo. The mean average Pb value for worldwide soils is 32 mg kg^{-1}, while mean values for various soil types range from 10 mg kg^{-1} to 67 mg kg^{-1} [22]. All the results obtained were above the background concentrations for examined sites (Table 1), while the soil sample from Smederevo was above target value of 85 mg kg^{-1} established by National legislation [16].

The natural content of Pb in soils is mostly inherited from parent material. However due to widespread Pb pollution most soils are likely to be enriched especially in the top horizon [5, 22]. Lead is considered to be the least mobile among other heavy metals. Alkaline conditions cause precipitation of Pb in the form of hydroxide, phosphate or carbonate which are stable complexes [22], while increasing acidity causes increase in Pb solubility. High content of Pb measured in all tested soil samples is almost exclusively related to the residue of the leaded gasoline [27] that was officially banned in the Republic of Serbia only in 2010. Results of previous researches of urban soils in Belgrade confirm high content of Pb in topsoil - Gržetić and Ghariani [27] obtained average value of 230.83 mg kg^{-1}, while Marjanović et al. [29] even greater 298.6 mg kg^{-1}. Researches conducted after the ban on leaded gasoline in 2010 show its gradual decline in the wider area of Belgrade 80.36-137 mg kg^{-1} [28] and in composite soil samples in Novi Sad 82.3 mg kg^{-1} [30]. Despite this fact, aforementioned decline is slow, which is supported by contamination factor that indicates moderate contamination for Pb at all sampling sites, with the highest calculated in Smederevo 2.21.

Lead is not readily available metal to plants. Although it naturally occurs in all plants, its essentiality has not yet been proved [22]. Its accumulation in plants can be either from soil solution or by foliar application [5]. General opinion is that Pb primarily accumulates in the roots and that is poorly translocated to other plant parts, which is of great importance for the environment. Along with poor transfer and the existing alkaline conditions, Pb is immobilized which is the reason why its content in needles was within normal values for plant tissues (0.1-10 mg kg^{-1}) (Table 2), in spite of its high concentrations in soil. Concentration of Pb in two year old needles was slightly higher in relation to one year old needles.

Table 1: Total element, background concentration, contamination factor and pH values at selected sampling sites

Site	Depth (cm)	Total element concentration (mg kg^{-1})			Background concentration (mg kg^{-1})			Contamination factor (Cf)			pH
		Co	Ni	Pb	Co	Ni	Pb	Co	Ni	Pb	
Smederevo	0-20	5.74±0.15	118.69±10.9	118.63±4.68	6.97	103.0	53.5	0.82	**1.15**	**2.21**	8.56
Obrenovac	0-20	7.96±0.44	63.78±1.11	56.39±1.69	8.76	131.0	35.1	0.90	0.48	**1.60**	8.68
Belgrade	0-20	5.70±0.56	35.19±0.83	77.96±0.83	6.73	72.0	37.0	0.84	0.48	**2.10**	8.78
Target value RS		9	35	85							

Values for total element concentration and pH are mean ± sd, n = 5

Table 2: Content of Co, Ni and Pb in Pinus nigra needles (mg kg^{-1} d.w.)

Site	Pinus nigra					
	1-year-old needles			2-year-old needles		
	Co	Ni	Pb	Co	Ni	Pb
Smederevo	0.92±0.01	2.08±0.04	0.78±0.12	0.92±0.04	1.47±0.05	1.02±0.10
Obrenovac	1.14±0.01	2.52±0.95	0.95±0.18	1.17±0.01	1.72±0.03	1.07±0.12
Belgrade	1.30±0.01	1.94±0.14	1.12±0.17	1.35±0.03	1.78±0.05	1.27±0.23

Values are mean ± sd, n = 5

Conclusion

An analysis of the topsoil from three urban parks under different sources of pollution showed that there is no significant contamination by Co and Ni which was also confirmed by contamination factor. Although results for Ni in soil surpass the target values of 35 mg kg^{-1} set by National legislation, most of them are below the background values indicating its geological origin. However all results obtained for Pb were above the background values and in Smederevo above the target value of 85 mg kg^{-1}. Contamination factor for Pb at all sampling sites confirmed moderate contamination that can be related to anthropogenic influence.

Element content in plant material, both 1-year and 2-year old needles indicates low pollution, and was within normal values for plant tissues. Although results obtained in this study suggest low contamination in needles, it should be noted that anthropogenic and industrial pollution causes alkalinization of soils in examined parks, which consequently leads to immobilization of elements in soils. Under existing environmental conditions these metals are unavailable for plants at pH>8. Any change in the environment can lead to accessibility of these elements, which can be potential hazard to both human health and environment.

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