Predicting of lead distribution and immobilization in soil of the region of lignite mining (Rudovci, Serbia)

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Lead distribution and immobilization in cultivated soils in Rudovci, Serbia was investigated. Sampling was carried out by the method recommended by ICP-Forests Manual, 2006, Part III Sampling and analysis of Soil. The sampling geometry was systematically designed with a random component. The maximum sampling depth was 100 cm and lead distribution was monitored during 425 days. First sample was taken after 50 days and every single next sample was taken after 50 days except for the last sample which was taken after 25 days. Before the profile contamination, physical and chemical soil analysis has been done. The cation exchange capacity of the soil was done because media affect mobility cations (anions) in soil. The effect of immobilizations of Pb is highest in the second horizons where the depth of investigating soil is 25-50 cm.

Key words: *lead, immobilization, soil chemistry, cation exchange capacity, environment*

1. Introduction

Soil contamination is the result of human activity, including its pollution by atmospheric deposition, application of agrochemicals and inflow of domestic waste to the land dumpings. Heavy elements such as Pb, Cu, Cd, Hg and Zn often cause contamination of soil, water and food chains [5]. Pollution of ground and surface water with heavy metals is becoming a major concern since Cd, Cr and Pb are not biodegradable and tend to accumulate in living organisms causing various diseases and disorders. Once enter the soil, the Pb are subjected to weathering processes such as oxidation, hydration and carbonation [1]. In addition, lead in soil may unfavourably affect soil ecology, agricultural production, or product and water quality [3]. Due to higher levels of heavy metals and pesticide residues in plant products from the agricultural area of Belgrade, Serbia, as well as the presence of pesticide residues in some soil samples, their continual monitoring is strongly recommended as a first preventive measure to minimize human health risks [11]. Lead causes encephalopathy, cognitive impairment, behavioural disturbance, kidney damage, anaemia and toxicity to the reproductive system [15].

The interaction of Pb ions with natural soil particles involves multiple mechanisms because various species can exist in solution due to complexes with organic and inorganic ligands. Knowledge on the interactions of Pb with such substances as clay, aluminium oxides, iron oxides and manganese oxides which are present in the soil is necessary to understand the mechanisms of the adsorption of metals [9]. Investigation of sorption and desorption behaviour of lead on Chinese kaolin concluded that kaolin has the high affinity for Pb and suggested that kaolin could strongly retain Pb [23].

The results of both adsorption edge and adsorption isotherm studies showed that Pb adsorption is highly pH dependent. Among all soil properties, the organic matter plays the most important role in controlling lead sorption onto soils [8]. The transfer of lead from soils to plants depends by many types of factors. Between them there is natural radioactivity of soil which was investigated in our previous study [13]. Researches mobility and distribution of Pb in ten industrially polluted Danish surface soils showed that the first factors determining the bonding of Pb in industrially contaminated soils are contamination level and the stability of the originally contaminating Pb-species whereas soil characteristics are of secondary importance [7]. Lead was concentrated in the small (<0.063 mm) grain-fractions of most soils. The study revealed that the adsorption capacity of the soil for Pb and the other heavy metals increases with increasing sorbent concentration [10].

Some studies [15,19] observed that Pb contamination occurs mainly at a depth of 0 -15 cm. Some authors having applied Pb to soil observed that 30 weeks of leaching later, Pb was still concentrated within a few centimetres of surface soil. Generally, the highest Pb is produced in the surface soil horizon of Pb contaminated soil [20]. Investigations also showed that the higher valence of cations the greater its ability to adsorb and penetrate into the compensating layer of negative colloids [6]. The relationship between valence and its ability of

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adsorption can be explained by divalent and trivalent ions build. With anion compounds that dissociated less than monovalent cation reaction between anions and lead cation leads to forming compounds that are less dissociated [2].

The objective of this research, considering of all said above, is to explain the distribution and immobilization of lead in the soils at Rudovci (Serbia) and predict the distribution of lead, when the effect of immobilization as well as physical-chemical properties of soil are known. Rudovci are located around 60 km south of Belgrade and four kilometres from the open cast mines Kolubara basin where lignite (brown coal) is mined. This is the largest open cast mine in this part of Europe. Productive coal seam stretches from east to west. Formations from Palaeozoic form the basis of the field. The land is mainly clayey and sandy-clayey, sand rarely sandy-gravelly.

The mines and power plants are air, water and soil polluters. The region of Rudovci is agricultural and cattle breeding area, supplying with its products Belgrade, the capital of Serbia. Both of these factors affect the significance of this research.

2. Materials and methods

2.1. Study area

The study area (Figure 1) is a small agricultural valley located at Rudovci, near the city of Lazarevac in Serbia (44°22'N, 20°24'E). The average inclination of the slopes is 4-5°.
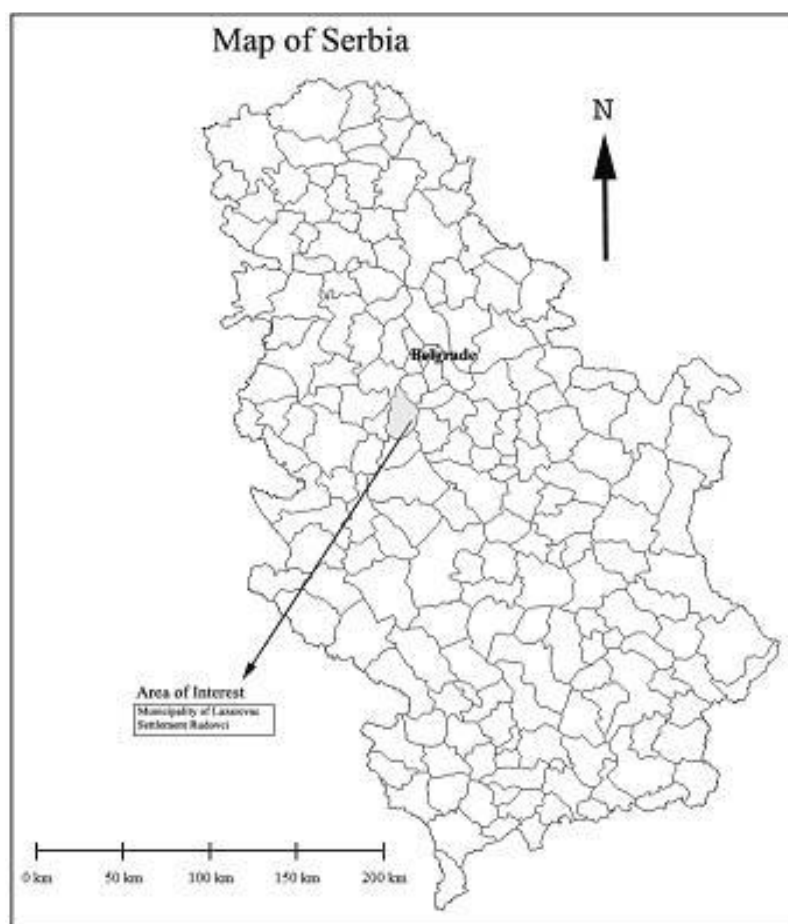

Fig. 1. Location settlements Rudovci as a field of research.

The meteorological parameters (air pressure, air temperature, air humidity, rainfalls, cloudy conditions and wind) have a high influence on the emission of polluting materials. The average annual rainfall was 675 mm (nearest meteorological station is "Rudovci Mines") of period December 10th, 2008 to February 9th, 2010 and average annual air temperature was 11.5 °C (www.hidmet.gov.rs). The climate is moderate continental, with hot summers and long, harsh winters. There is also a considerable effect of southeasterly winds.

2.2. Sampling

There are several major approaches to sampling in the study of contaminated sites (regular networks, statistical approach, the hypothetical-oriented approach etc.) which give a detailed description of the methods ISO 10381-1:2002, ISO 10381- 5:2005 recommended on the basis of proper sampling network (square, less triangles).

The reason for making regular network sampling (Fig. 2) is easiness of setting up a network on the ground, which therefore facilitates the tracking and observations changes in the pollutant spatial distribution.
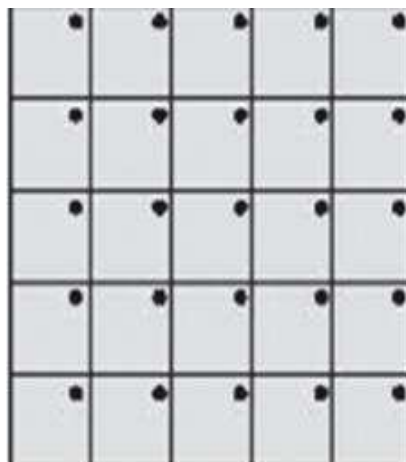

Fig. 2.   A distribution map of sampling profile using the methods of regular network with distance of 20 cm.

2.3. Analysis of soil samples

The samples were taken with special cylinder with the strictly defined volume of 100 cm^3, called Kopecky's cylinder. The cylinders have one sharp side for easier indentation of the ground surface. The samples were taken from the surface to the horizon depth. The ground surface needs to be prepared for taking samples. It means that it needs to be clean and smooth, without roots and grass. The soil specimen was taken from four horizons. The mechanical composition was determined by using the sedimentation method [12]. After drying, samples were sifted through 0.2 mm sift and prepared for characterization. Sand, silt and clay fractions in samples from each horizon of the cultivated soil were determined by the pipette method [12].

The organic matter was determined by the wet digestion method [10], with potassium dichromate as oxidizer. pH was measured with pH-meter (PRO-PH208M) in water. The elements present in soil were determined by XRF analysis (UPA XRF 200, Veeco & UPA). The structure of the soil was examined by X-ray diffractometer Siemens D-500, with Cu K_α radiation. The diffracted X-rays were collected over 2θ range 20-80^0 using a step width of 0.02^0 and measuring for 1s per step.

The water absorption capacity (WCU) was determined according to Rees and Chandrasekhar [16]. The mass loss of sample heated at 300 °C termed "water absorption capacity of dry samples" (WCU) and calculated as:

$$WCU = [(a-b)/b] \times 100 \ (\%) \tag{1}$$

where:

a – mass of sample [1 g],

b – mass of sample after heating.

The cation exchange capacity (CEC) of soils was determined with standard procedure, applicable for calcareous and noncalcareous soils (EPA, 1986.)

The samples were analysed for major and trace anions, i.e. fluoride, chloride, nitrite, bromide, nitrate, orthophosphate and sulphate, by ion chromatography (Metrohm, type 761 Compact IC (Switzerland) with a conductivity detector). The eluent was 3 mM Na_2CO_3 at a flow rate of 1.0 mL min^{-1}. The full-scale range was 50 μS and the injected sample volume was 20 μL for each probe.

In this research cations Na $^+$, K$^+$, Ca $^{2+}$ and Mg $^{2+}$ were determined. The samples were analysed by inductively coupled plasma atomic emission spectrometry (ICP-AES, model Spectro CIROS vision, Kleve, Germany). The spectral range between 125 nm and 770 nm was covered enabling complete scans of the spectrum within 3 s.

2.4. Vertical distribution and immobilization of Lead

For research on vertical distribution of lead in the soil samples were collected from cultivated soil of four horizons at different depths; 0-25 cm (I), 25-50 cm (II), 50-75 cm (III), 79 cm (IV). Surface mines are the biggest soil polluters due to direct particle pollutants, waste waters and harmful gases. It is important to examine the distribution of lead at these depths, because crop plants that are planted in this area (corn and wheat mostly) get water from the soil at a depth from 60-90 cm, and even up to 120 cm depth (Nenadović et al., 2012).

Lead distribution was monitored nine times during 425 days from (December 10th 2008) to (9th February 2010). The period between samplings was 50 days. First sample was taken after 50 days, the next following sample was taken after 50 days and the last sample which which was taken after 25 days.

To investigate immobilization of lead by the soil samples was contaminated at 25°C with a solution of Pb $(NO_3)_2$ (99.99 % Alfa Aesar) of 2.95 mg/l concentration. A sample of 0.2 g of four- horizon soil was placed in the flask and 10 ml of lead nitrate solution was added to it. The flask was closed and shaken for 24 h. To avoid the random error three samples from each sampling depth were analyzed and the average value was calculated. The lead uptake q_e (mg/g) was evaluated using the initial and current, t, lead concentration (c_i cation and c_t cation), in a given solution volume, V, for a given amount of soil as a substrate, m_s (eq. 1):

$$q_e = \frac{(c^i_{cation} - c^t_{cation}) \cdot V}{m_s}$$

(2)

The Pb^{2+} adsorption efficiency, η, on soil was evaluated to optimize the contact time and the amount of soil and was calculated with Equation (3):

$$\eta = \frac{c^i_{cation} - c^e_{cation)} \cdot 100}{c^i_{cation}}$$

(3)

Where c^i_{cation} and c^e_{cation} are the initial and equilibrated cation concentrations (mg/l).

3. Result and discussion

XRD analysis of soils showed the presence of quartz, kaolinite and feldspar as the major phases (Figure 3). Clay assemblage consisted of kaolinite, smectite, interstratified illite - smectite and illite. The latter contains minor titanium and some iron.
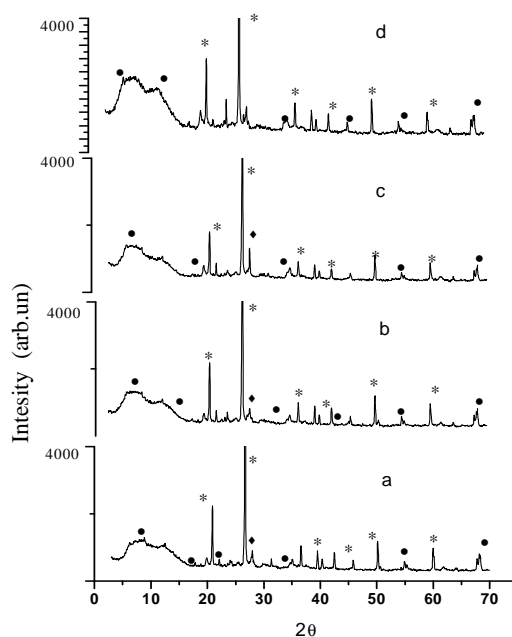

Fig. 3. The comparative X-ray powder diffraction pattern samples from: a) I horizon; b) II horizon; c) III horizon d) IV horizon (●-kaolinite/illite, *- quartz, ◆- feldspar).

In the analysed soils, the clay fraction prevailed in all horizons, making from 55.5 to 65 % (Tab. 1). Percentage of clay increased with the profile depth, the highest was in the horizon IV (65 %). The sizes of clay, silt and sand are < 0.002 mm; 0.002-0.006 mm and 0.06-2 mm, respectively. Sand fraction made from 1.6 to 2.7 % and silt from 0.3 to 0.7 %. The organic matter percentage was the highest (1.6) in the upper horizon (I) and decreased to 0.8 in horizon IV.

pH has a significant effect on the various properties of the soil as well as the mobilization of pollutants through the soil. The pH value of an adsorption edge (i.e., the pH value at which a change occurs in a particular concentration of aqueous ions adsorbed by a solid phase) is dependent on the surface-metal bonding strength and, consequently, on the type of adsorption site involved. pH can significantly affect the solubility of minerals. pH of soil samples was in the range 4.6 - 7.2. On the basis of different pH values on the horizons of the same profile can be seen that the tested soil can be classified from highly acidic to moderately alkaline soil.

In our previous study [12] the values of the Rudovci soil texture were approximately the same for the sand and silt contents, while different clay content was recorded (55.5 - 56.4). Based on the clay content soils may be characterized as light clay (USDA, 1999). In the vicinity of the investigated soil there are clay excavations. Clay deposits have been moved by erosion and water from their primary location. This is a possible reason for a different percentage of clay in the same soil in different periods.

Tab. 1. The soil structure, the water absorption capacity and CEC of the soil.

Samples	Organic matter [%]	Clay [%]	Silt [%]	Sand [%]	WCU [%]	CEC [meq/100g]
I horizon	1.60	55.5	0.60	2.70	3.52	35.9
II horizon	1.20	59.2	0.30	1.60	4.54	59.1
III horizon	1.00	61.1	0.40	2.20	4.47	63.7
IV horizon	0.75	64.9	0.70	2.40	6.68	68.6

The main adsorption mechanism, involved in the cation removal by soil is exchanged with exchangeable cations such as Ca^{2+}, Mg^{2+}, K^+ and Na^+ [17]. The CEC of these materials is a measure of number of negatively charged sites that attract exchangeable cations. The negative charge caused by substitutions within the lattice structure is permanent and pH independent. In the land were the big stones (pebbles), plants and other residues which are removed. This is the reason why percentage sum is not 100 %.

Cation exchange capacity was in the range from 35.9 to 68.6 meq/100 g and increased with the profile depth (Table 1). The rate and stability of adsorption generally increase with organic matter, clay surface area and CEC. In soils low in organic matter or clay content, adsorption may be dominated by cations, while the role of cations in adsorption may be minimal in soils with high organic matter or clay content.

Table 2 shows chemical composition of soil and Si/Al ratio of investigated soil.

Tab. 2. The chemical composition of samples.

Samples	SiO_2	Al_2O_3	CaO	K_2O	Fe_2O_3	ZrO_2	TiO_2	Si/Al
I horizon	74.9	4.89	1.72	3.65	3.27	0.047	1.22	12.3
II horizon	75.0	4.85	1.50	3.68	3.33	0.050	1.15	12.4
III horizon	68.1	5.40	1.49	3.74	4.12	0.038	1.10	10.1
IV horizon	66.2	5.91	1.56	3.56	4.21	0.036	1.04	8.97

Cation adsorption by soil is strongly influenced by the Si/Al surface site ratio, steric effects of interlayers or channels, Lewis acid strength of the metal, and pH of the medium. The ratios Si/Al depends on the proportion of the coarse materials, mainly formed by quartz sands which contains only silicon, and fine clays which contain silicon, aluminium and potassium. Generally Si/Al ratio of all four horizons, in the soil of lignite mining (Rudovci, Serbia) is so high and from this point of view that soil shows great mobility of lead ions through them.

Soil organic matter is an important component of the soil. It has a high surface area, and has functional groups (e.g., carboxyl and phenol groups) that metals can form chemical bonds with. It has been observed that Pb forms strong complexes with organic matter. In our case the percentage of organic matter was low (0.75-1.6 %) and decreased with increasing depth from which soil samples were taken. This low content of organic matter could favourably affect the rate of adsorption of Pb in the investigated soil. Strawn and Sparks, (2000) [18] measured of sorption kinetics using the stirred flow reactor revealed that the extent of the fast and slow reactions occurring in the soil is directly dependent on the amount of soil organic matter present. Specifically, with increasing the soil organic matter content the rate of Pb sorption decreases. [18].

3.1. Vertical distribution of Pb^{2+} in natural soil

Lead in soil is largely insoluble, low in mobility, and seldom leached from the profile. As a result, Pb contamination occurs mainly in the surface soil due to human factors, and the concentrations of soil Pb

drastically decrease with increasing soil depth. Figure 4 shows a vertical distribution of Pb^{2+} of four horizons of soil after 50 days, 100 days, 150 days, 200 days, 250 days, 300 days, 350 days, 400 days and 425 days. Looking at the trend of lead distribution in the soil after a sampling (Figure 4) the movement path of contaminants is obtained. Every point shows mean values of three weighing.

Lead distribution in the first horizon changed in this period from 12.00 mg/kg to 0.60 mg/kg. In the second horizon change of lead concentration ranged from 1.80 mg/kg to 1.20 mg/kg. In the third horizon after the ninth sampling, concentration of lead (2.40 mg/kg) is higher than after the first sampling (1.50 mg/kg). This trend was continued in the fourth horizon. After the first sampling in the fourth horizon concentration was 0.90 mg/kg but after 9th sampling concentration of lead increased to 3.30 mg/kg.
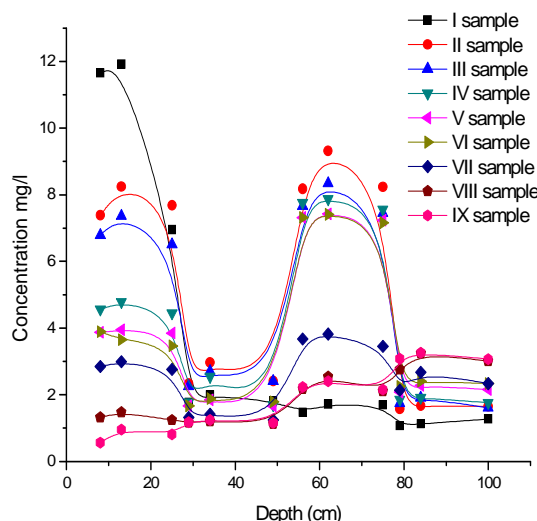

Fig. 4. Vertical distribution of Pb ions in four horizons of soil during different periods
(50 days, 100 days, 150 days, 200 days, 250 days, 300 days, 350 days, 400 days, 425 days).

After the first sampling (after 50 days) the gradual decline in the concentration of lead could be observed. The first horizon was permeable to lead cation. After the second sampling (after 100 days) accumulation of lead in the third horizon was notified. After the third sampling (after 150 days) accumulation of lead in the second horizon occurred. From the fourth to the seventh sampling, lead accumulated in the third horizon. Finally, after the ninth sampling (after 425 days) the maximum concentration of lead was observed in the forth horizon.

3.2. Effects of Lead immobilization

The adsorption of Pb was investigated at different values of pH ranging from 2.05 to 9.2.The adsorption Pb ions in alkaline and acidic media of four horizons are investigated. The adsorption of Pb ions in alkaline media is higher than in acidic media. For this purpose, the effects of lead immobilization in alkaline media of all four horizons were determined.

For calculation of the effect of Pb immobilization in alkaline media (Tab. 3) of four horizons of investigated soil the Equation (3) was used. Table 3 shows the effects of Pb immobilization in the soil

Tab. 3. The effects of Pb immobilization in the soil.

Sample	Depth [cm]	η [%]
I horizon	0-25	65.3 ± 1.06
II horizon	25-50	88.7 ± 0.65
III horizon	50-75	26.3 ± 1.63
IV horizon	75-100	32.7 ± 0.24

The effect of immobilization ranged from 26.3 to 1.63 % to 88.7 to ☐0.65 %. At depths of 25 to 50 cm (the second horizon) the greatest effect of immobilization (88.7 %) is obtained, i.e. the accumulation of lead was the largest (Figure 4). It is obvious that all factors, composition of soil, acidity of soil contribute the best immobilization of lead in the second horizon. The obtained results may used as a guide that in investigated area shouldn't plant the crops at depth 25 to 50 cm.

4. Conclusions

These studies get information about the prediction of vertical distributions and immobilizations of lead in soils in the region of lignite (Rudovci, Serbia) during different time. Physical-chemical properties of soils determine the adsorption of lead and therefore their vertical distribution and immobilizations. The vertical distribution of lead in the period from 100 to 300 days has the same trend. Lead concentration was significantly higher in the first and III horizons in relation to the II and IV horizons. The vertical distribution is approximately equal after 400 and 425 days of all horizons. The adsorption of Pb of the soil is higher in alkaline media and in all of horizons adsorption isoterms have the same trend. The effect of immobilizations of Pb is highest in the second horizons of investigated soil. These results open a new ability to predict the distribution of pollutants, if the effect of immobilization is known as well as physical-chemical properties of soil.

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