RADIONUCLIDES AND HEAVY METALS IN SOIL, VEGETABLES, AND MEDICINAL PLANTS IN SUBURBAN AREAS OF THE CITIES OF BELGRADE AND PANČEVO, SERBIA

by

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The content of radionuclides (^{40}K, ^{238}U, ^{226}Ra, ^{232}Th, and ^{137}Cs) and heavy metals (As, Cd, Cu, and Pb) was determined in samples of soil, vegetables and medicinal plants collected in the period 2007-2017, from two suburban areas of Belgrade – the municipalities of Palilula and Surčin, and Pančevo – the 'Dr Josif Pančić' Institute for the Study of Medicinal Herbs. During the research period, activity concentration of ^{137}Cs in soil decreased from 16 Bqkg^{-1} to 3.9 Bqkg^{-1} (Palilula, Belgrade) and from 18 Bqkg^{-1} to 12 Bqkg^{-1} (Surčin, Belgrade). Mean activity concentrations of natural radionuclides in the soil were higher than the global average. Trend for heavy metal levels, according to the average concentrations found in the soil, were as follows: Cu >Pb >As >Cd for Palilula, Pb >Cu >As >Cd for Surčin and Dr Josif Pančić' Institute, Pančevo. The obtained results indicate that the industrial pollution has no impact on food production in the study area and that the main anthropogenic source of radionuclides and heavy metals in soil are mineral phosphorous fertilizers, often used in agricultural fields.

Key words: radionuclide, heavy metal, pollution, agricultural

INTRODUCTION

Radionuclides and heavy metals are present in the environment through naturally occurring geochemical processes, or as products of industrial or agricultural emissions, deposition and disturbances. Main anthropogenic activities which can enhance concentrations of naturally occurring radionuclides and heavy metals in the environment and foodstuffs are: uranium mining, phosphate ore processing, coal ash, water treatment, metal mining and processing, geothermal energy production wastes, and the petroleum industry, mining and smelting operations, industrial discharge, vehicular emissions, atmospheric deposition of particles, biosolids and manures, and the application of soil fertilizers [1-5].

Naturally occurring radioactive elements in the environment date back to the period of the Earth's formation. The main radionuclides present in the Earth's crust are ^{40}K, ^{238}U, and ^{232}Th and their radioactive decay products [6]. Natural radionuclides are not distributed uniformly in soil and their concentration depends on geological and geographical conditions.

Artificial radionuclides, such as ^{137}Cs with a half-life of 30 years, are present in the environment as a result of nuclear testing and nuclear disasters. Environmental contamination with ^{137}Cs, in Serbia and the region, is a consequence of the Chernobyl accident (1986) [7] and even today it can be found in environment [8-10].

The accumulation of heavy metals in soil can lead to reduction of fertility in arable soil, by inhibiting microbial populations [11], earthworms and other macro fauna populations [12]. Due to their accumulation and mobility, heavy metals can enter the food chain and, depending on their content and toxicity, have harmful effects on humans and animals.

In this regard, the aim of this study is to assess the impact of industrial and other pollutants on arable fields in municipal areas of Belgrade and Pančevo cities, Serbia. For this purpose, content of natural (^{40}K, ^{238}U, ^{226}Ra, and ^{232}Th) and artificial (^{137}Cs) radionuclides, as well as heavy metals (As, Cd, Cu, and Pb), was determined in soil, vegetables and medicinal plants. The term 'heavy metals' is used in its broadest sense in this paper and includes the non-metallic heavy element arsenic.

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MATERIAL AND METHODS

First sampling site was the municipality of Palilula in Belgrade (45°06' N, 20°23' E), located on the left bank of Danube river. Today it is one of the most fertile areas in Serbia with alluvial Fluvisol soil [13]. Around 15 km away is the city of Pančevo, where the largest petrochemical producers in Serbia – 'Pančevo'oil refinery, with total installed processing capacity of about 4.8 million tons a year, and 'HIP-Petrohemija' – are located, making it the main industrial hot spots in Serbia.

Second sampling site was in the production fields of the 'Dr Josif Pančić' Institute for the Study of Medicinal Herbs in Pančevo (44°53' N, 20°40' E) located 4.5 km away from Pančevo city center and 19.6 km away from Belgrade city center, which is characterized by Chernozem soil [13]. Municipality of Sur?in in Belgrade (44°47' N, 20°16' E), was used as a control area, because there are no industrial sources of environmental pollution. This sampling site is also characterized by Chernozem soil [13].

Between the years 2007 and 2017, samples of cultivated soil and vegetables were collected from Palilula (sampling points: Ovča, Borča, and Dunavac) and Surčin (sampling points: Jakovo and Bojčin forest), both areas with intensive agricultural production, and from 'Dr Josif Pančić' Institute in Pančevo, fig. 1.

For radionuclides (^{40}K, ^{238}U, ^{226}Ra, ^{232}Th, and ^{137}Cs) determination in soil and medicinal plants, we used gamma ray spectrometry on a High Purity Germanium detector (ORTEC) with a relative efficiency of 30% and energy resolution of 1.85 keV (at 1332.5 keV, from ^{60}Co). Samples of soil (cultivated) with a mass of 3-5 kg were collected from a depth of 0-20 cm, homogenized, dried at 105 °C, and stored in 1l Marinelli beakers. Samples of medicinal plants were dried at 105 °C, homogenized and measured into 1l Marinelli beakers. Samples of medicinal plants were dried at 105 °C, homogenized and measured into 1l Marinelli beakers. Homogenized samples were sealed tightly and kept for 40 days to ensure that equilibrium between ^{226}Ra and its short-lived decay products was reached. We used commercially available standards with mixed radionuclides for calibration:

- ^{241}Am and ^{152}Eu, dispersed in silicone resin in a Marinelli beaker, density (0.182 0.05) gcm^{-3}, volume 1l and
- ^{241}Am, ^{109}Cd, ^{139}Ce, ^{57}Co, ^{60}Co, ^{137}Cs, ^{113}Sn, ^{85}Sr, and ^{88}Y, dispersed in silicone resin in a Marinelli beaker, density (1.22 0.01) gcm^{-3}, volume 1l.

The first calibration standard was used for plant measurements, and the second for soil measurements. The counting time for the samples, as well as for the background, was 60000 s.

For heavy metal analysis, soil samples were air-dried and passed through a 0.2 mm stainless steel sieve, including blank sieving prior to chemical analysis. Plant samples were dried to a constant weight at 65 ºC and ground in a laboratory mill (Polymix, Kinematica AG, screen size 2.0 mm). Sample mineralization (soil 0.5 g; plant 0.4 g) was conducted through wet digestion in a microwave (CEM, Microwave 39 MDS- 2000), in advanced composite vessels, by using a 3:1 mixture of nitric acid (HNO$_3$, 65 %) and hydrogen peroxide (H$_2$O$_2$, 30 %). Total concentrations of As, Cd, Cu, Ni, Pb, and Zn were determined through inductively coupled plasma spectrometry (ICP-OES, Spectro Genesis). In order to check the accuracy and determine the uncertainty of the measured concentrations of heavy metals, standard reference samples (standard reference soil-ERM-CC141 (loam soil) and plant material-BCR-100 (beech leaves) obtained from the IRMM (Institute for Reference Materials and Measurements, Geel, Belgium) and certified by the EC-JRC (European Commission – Joint Research Centre), were used. Measurements were done in 5 replicates.

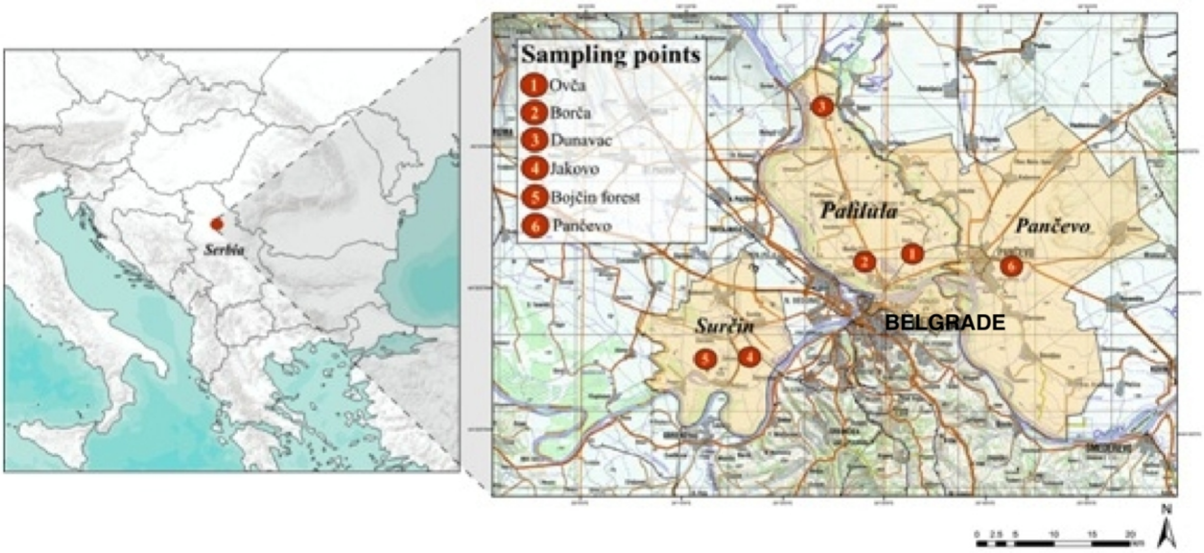

Figure 1. Study areas with the designated sampling points

Radiation hazard parameters

In order to assess the radiation hazard, the following parameters were calculated: absorbed dose rate $\dot{D}$ (nGyh^{-1}), annual effective dose *EDE* (mSva^{-1}) and external hazard index H_{ex}.

Calculation of the absorbed dose rate

The mean activity concentrations of ^{236}Ra, ^{232}Th, and ^{40}K were converted into doses using conversion factors. The calculations were performed according to the following eq. [14]

$$\dot{D}(\mathrm{nGyh}^{\ 1}) \quad 0.462C_{\mathrm{Ra}} \quad 0.604C_{\mathrm{Th}} \quad 0.042C_{\mathrm{K}} \tag{1}$$

In the eq. (1), it is assumed that all the decay products of ^{226}Ra and ^{232}Th are in equilibrium with their precursors.

Calculation of the annual effective dose

In order to estimate the annual effective dose, eq. (2), the conversion coefficient from the absorbed dose in the air to the effective dose must be considered. Thus, from the dose rate data obtained from the concentration values of natural radionuclides in the soil and by adopting a conversion factor of 0.7 SvGy^{-1} [14] and assuming that the people in Serbia spend an average of 20 % of their time outdoors, the annual effective dose can be calculated as

$$AEDE(\mathrm{mSv}) \quad \dot{D}(\mathrm{nGyh}^{\ 1}) \quad 24(\mathrm{hd}^{-1}) \\ 365(\mathrm{d}) \quad 0.7(\mathrm{SvGy}^{\ 1}) \quad 0.2 \tag{2}$$

Calculation of the external hazard index

The external hazard index, H_{ex}, is defined by the following equation [15]

$$H_{\mathrm{ex}} = C_{\mathrm{Ra}}/370 + C_{\mathrm{Th}}/259 + C_{\mathrm{K}}/4810 \tag{3}$$

If H_{ex} is less than one, the radiation hazard is insignificant. H_{ex} value equal to one corresponds to the upper limit of radium equivalent activity (370 Bqkg^{-1}).

RESULTS AND

The global average activity concentrations of natural radionuclides present in soil [14] are 400 Bqkg^{-1} for ^{40}K, 35 Bqkg^{-1} for ^{238}U and ^{226}Ra, and 30 Bqkg^{-1}for ^{232}Th. In the studied soils from municipalities Palilula and Surčin, mean activity concentrations of ^{40}K (675 Bqkg^{-1}), ^{238}U (39 Bqkg^{-1}), ^{226}Ra (38 Bqkg^{-1}) and ^{232}Th (43 Bqkg^{-1}) were above the world average, as a consequence of geological characteristics, tab. 1. Higher activity concentration of ^{40}K was detected in soil from the 'Dr Josif Pančić' Institute, Pančevo, tab. 1, probably due to the use of potassium mineral fertilizers [16]. Mineral fertilizers may contain high activity concentrations of natural radionuclides like ^{238}U, ^{232}Th, and ^{40}K and heavy metals like Pb, Cd, and Cu [17] and can be an anthropogenic source of these elements for the environment. Research conducted on arable and non-arable soils in suburban regions of Belgrade showed that mineral fertilizers did not cause an increase in natural radioactivity in the studied soils [18]. In contrast to these findings, in Switzerland, due to the use of mineral fertilizers since 1985, uranium concentrations in arable soils have increased significantly [17]. According to Serbian legislation (Official Gazette RS 97/13) [19], the permissible levels of ^{238}U and ^{226}Ra in

Table 1. Activity concentrations of radionuclides (^{40}K, ^{238}U, ^{226}Ra, ^{232}Th, ^{137}Cs) and radiation hazard parameters in arable soil collected in Belgrade municipalities and Pančevo (the 'Dr Josif Pančić' Institute)

Sample	^{40}K	^{238}U	^{226}Ra	^{232}Th	^{137}Cs	Absorbed dose rate	Annual effective dose	H_{ex}
	[Bqkg^{-1}]					[nGyh^{-1}]	[mSv]	
Palilula, Belgrade, 2007-2008								
Arable soil	608 18[1]	48 9	43 4	40 4	16 1	70	0.09	0.40
Palilula, Belgrade, 2016-2017								
Arable soil	619 19	34 4	35 4	37 3	3.9 0.2	65	0.08	0.37
Surčin, Belgrade, 2007-2008								
Arable soil	642 17	40 9	44 4	41 4	18 1	72	0.09	0.41
Surčin, Belgrade, 2013-2014								
Garden soil	531 23	35 5	33 3	35 1	15 1	59	0.07	0.33
Arable soil	623 15	51 10	39 3	42 1	16 1	70	0.09	0.40
Surčin, Belgrade, 2016-2017								
Arable soil	564 25	33 8	35 3	37 3	12 1	62	0.08	0.35
"Dr Josif Pančić" Institute, Pančevo, 2016-2017								
Soil (under Common fennel)	839 39	39 9	41 4	52 4	12 1	86	0.10	0.49
Soil (under Horseradish)	822 35	30 8	34 3	50 4	14 1	80	0.10	0.46
Soil under (Lemon balm)	801 35	40 9	38 3	47 4	15 2	80	0.10	0.45

[1] Mean standard deviation

mineral phosphorus fertilizers are 1600 Bqkg^{-1} (3200 Bqkg^{-1} for raw materials) and 1000 Bqkg^{-1}, respectively, which reduce input of radionuclides into the environment.

Radiocesium-137 was detected in soil, but during the research period, its activity concentration decreased from 16 Bqkg^{-1} to 3.9 Bqkg^{-1} (Palilula) and from 18 Bqkg^{-1} to 12 Bqkg^{-1} (Surčin), as a consequence of its half-life.

All the locations exhibited an absorbed dose rate higher than the world average of 58 nGyh^{-1}[6], as a result of the particularly high activity concentrations of natural radionuclides in the soil, which, in turn, increased the terrestrial gamma dose rate, tab. 1. The mean value of the absorbed dose rate was 71 nGyh^{-1}. Similar results were obtained in Bulgaria and Italy, where the mean values of the absorbed dose rate were 70 nGyh^{-1} and 74 nGyh^{-1}, respectively [6]. Obtained external hazard index values were mostly less than one, tab. 1, which is the limit recommended by ICRP [20], meaning that the research area is safe for humans to carry out their activities.

Absorption of radionuclides via roots and their accumulation in plants depends on many abiotic and biotic factors, such as soil type, climatic conditions, plant species, moisture regime, stand composition and age. In medicinal plants from "Dr Josif Pančić" Institute, Pančevo, both natural (^{226}Ra and ^{232}Th) and artificial (^{137}Cs) radionuclides were detected in medicinal plants, tab. 2. The average activity concentration of ^{137}Cs was 1.7 Bqkg^{-1}, with the highest content detected in horseradish (3.9 Bqkg^{-1}), tab. 2.

Highly reactive and often toxic at low concentrations, heavy metals may enter soils and groundwater, bioaccumulate in food webs, and adversely affect biota including humans. Vegetable crop plants have a high ability to accumulate metals from soil, which may pose as a risks to human health when they are grown on or near contaminated soils and consumed; thus, the accumulation of heavy metals in the edible parts of vegetables represents a direct pathway for their incorporation into the human food chain [21]. Average concentrations of As, Cd, Cu, and Pb (mgkg^{-1}) in soil and vegetables, from the sampling sites, are shown in tab. 3 (municipalities of Palilula and Surčin, Belgrade) and tab. 4 ('Dr Josif Pančić' Institute, Pančevo). Trend of heavy metal levels, according to the average concentrations found in the studied soil, was as follows: Cu > Pb > As > Cd for Palilula, Pb > Cu > As > Cd for Surčin and 'Dr Josif Pančić' Institute, Pančevo.

Table 2. Specific activity concentrations of ^{40}K, ^{226}Ra, ^{232}Th, and ^{137}Cs in medicinal plant samples collected at the 'Dr Josif Pančić' Institute, Pančevo [Bqkg^{-1}]

Sample/radionuclide	^{40}K	^{226}Ra	^{232}Th	^{137}Cs
Common fennel	969 39[1]	<MDA[2]	<MDA	0.5 0.1
Horseradish	866 26	<MDA	11 1	3.9 0.3
Lemon balm	449 22	4.4 0.3	3.9 0.5	0.6 0.1

[1] Mean standard deviation [mgkg^{-1}]; [2] MDA: minimum detectible activity

Table 3. Metal content (As, Cd, Cu, and Pb) in soil and vegetables collected from the municipalities of Palilula and Surčin, Belgrade

Sample/heavy metal	As	Cd	Cu	Pb
Palilula, Belgrade, 2016-2017				
Cultivated soil – arable soil	3.38 0.94[1]	1.95 0.16	37.0 0.3	34.6 6.1
Carrots	<MDA	<MDA	0.58 0.03	<MDA
Potatoes	<MDA	<MDA	0.41 0.03	<MDA
Onions	<MDA	<MDA	0.22 0.05	<MDA
Surčin, Belgrade 2013-2014				
Cultivated soil – garden soil	5.55 0.78	<MDA	30.6 0.6	43.8 5.9
Cultivated soil – arable soil	7.09 1.14	<MDA	34.1 0.5	59.7 2.6
Carrots	0.25 0.08	0.02 0.02	3.41 0.67	0.33 0.03
Potatoes	0.74 0.14	0.02 0.01	3.19 0.26	0.49 0.11
Onions	0.50 0.19	<MDA	1.53 0.19	0.21 0.04
Cabbage	0.84 0.18	<MDA	1.14 0.03	<MDA
2016-2017				
Cultivated soil – arable soil	6.61 2.79	1.63 0.42	32.0 3.4	36.7 11.7
Carrots	<MDA	<MDA	0.58 0.03	0.35 0.31
Potatoes	<MDA	<MDA	0.17 0.01	<MDA
Onions	<MDA	<MDA	0.44 0.06	0.39 0.0
Cabbage	<MDA	<MDA	0.21 0.04	<MDA

[1] Mean standard deviation [mgkg^{-1}]

Table 4. As, Cd, Cu, and Pb concentrations in soil and medicinal plants, and the plant concentration factor (PCF), at the "Dr Josif Pančić" Institute, Pančevo

Sample/heavy metal	As [mgkg^{-1}]	Cd [mgkg^{-1}]	Cu [mgkg^{-1}]	Pb [mgkg^{-1}]
Soil	8.41 0.21[1]	2.75 0.77	29.0 0.1	32.7 0.9
Plant (common fennel)	<MDA	<MDA	8.66 0.33	<MDA
PCF	/	/	0.3	/
Soil	10.66 0.18	3.50 0.56	29.5 0.1	42.8 0.8
Plant root (Horseradish)	1.95 0.58	<MDA	1.89 0.47	<MDA
PCF root	0.18	/	0.06	/
Plant leaves (Horseradish)	1.66 0.29	<MDA	2.94 0.17	<MDA
PCF leaves	0.16	/	0.1	/
Soil	10.76 1.35	3.75 0.47	28.0 0.5	46.9 4.8
Plant (Lemon balm)	<MDA	<MDA	11.59 0.39	<MDA
PCF	/	/	0.41	/

[1] Mean standard deviation [mgkg^{-1}]

Table 5. Reference values for concentrations of heavy metals in soil [mgkg^{-1}]

Element	Off. Gaz. RS 23/94) [22]	EEC/86/278) [27]
As	25	–
Cd	3	1-3
Cu	100	50-140
Pb	100	50-300

Mean concentrations of heavy metals in soil from Palilula, did not exceed maximum permissible levels prescribed by Serbian regulations [22] and Council Directive [23]. However, content of Cd in soil from 'Dr Josif Pančić' Institute, Pančevo, exceeded the reference values, tab. 5.

Arsenic was detected in vegetables collected from Surčin in 2013-2014, with the average concentration being 0.58 mgkg^{-1} (0.25-0.84 mgkg^{-1}), tab. 3, pointing to the effects of intensive agricultural production, *i. e.* the use of herbicides and insecticides [24], as well as the use of phosphate fertilizers containing As [25]. However, research conducted in 2016-2017 showed that arsenic was not present in the vegetables at this location.

Anthropogenic source of Cd is industrial discharge from the metallurgical and chemical industries, combined with developed agricultural production, including the use of phosphate fertilizers, which are one of the most ubiquitous sources of Cd contamination in agricultural soils throughout the world [26]. Normal Cd content in soils is between 0.2-1.1 mgkg^{-1} [26]. Cadmium can enter the food chain via plants, with vegetables being the main source of Cd for humans [26]. Phosphate fertilizers and sewage sludge are the main source of Cd input in soils [17]. In Switzerland, mean Cd concentrations were 58 % higher in arable top soil than in arable subsoil, which points to the significance of Cd input in arable soils [17]. The presence of Cd was noted only in carrot and potato samples collected from Surčin in 2013-2014, tab. 3.

Copper was found in all investigated soil, vegetables and medicinal samples, tabs. 3 and 4. In soil, copper content is closely associated with soil texture and several other soil parameters, in particular soil pH and SOM, which control its distribution and behavior [26]. Cu content in plant tissue depends on its levels in the soil, but also on the characteristics of the plants themselves [26]. In the studied vegetables, tab. 3, Cu content fell within an appropriate range for food consumption in the USA, which amounts to 0.1-3.2 mgkg^{-1} for vegetables, as well as within a normal range for the optimal functioning of plants [28]. National regulations do not prescribe a norm for levels of this element in foodstuffs of plant and animal origin.

Lead is a metal found in the earth's crust and has been mobilized in the environment by recent anthropological activities. In our study, lead concentrations ranged from 32.70-59.70 mgkg^{-1} with a maximum level found in Surčin, tabs. 3 and 4. However, in vegetables, tab. 3, Pb levels were within the range of reference values (3 mgkg^{-1}), while in medicinal plants, tab. 4, Pb was not detected.

Plant concentration factor (PCF), tab. 4, varied with heavy metal type and plant species, and depended on the metal concentrations in soil and each plant's heavy metal uptake capacity. The PCF for Cd and Pb was not established.

CONCLUSION

Research in Belgrade and Pančevo suburban areas was conducted with the aim of determining the content of radionuclides and heavy metals in the environment. Sampling sites were selected on the grounds that they have significant areas of agricultural land for production of food for residents of Belgrade and are located in the vicinity of a large industrial area dominated by oil refinery and petrochemical industry in the city of Pančevo. Results obtained in this long-term study indicate that industrial pollution has no impact on food production in the study area and that the main anthropogenic source of radionuclides and heavy metals in soil are probably mineral phosphorous fertilizers, often used in agricultural fields. It was also found that all of the studied samples of vegetables are safe for both human and animal consumption. These results emphasize the importance of environmental monitoring, especially in those localities where there is a potential risk of anthropogenic contamination with different pollutants.

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AUTHORS' CONTRIBUTIONS

B. M. Mitrović, B. R. Vranješ, O. A. Kostić and M. M. Mitrović collected samples. B. M. Mitrović, B. R. Vranješ, V. S. Perović, and O. A. Kostić analyzed the samples. B. M. Mitrović, P. Ž. Pavlović, O. A. Kostić, and M. M. Mitrović interpreted the results and discussed them in reference to the literature data. All the authors contributed to the manuscript preparation and approved its final version.

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РАДИОНУКЛИДИ И ТЕШКИ МЕТАЛИ У ЗЕМЉИШТУ, ПОВРЋУ И ЛЕКОВИТОМ БИЉУ ПОРЕКЛОМ ИЗ ПРИГРАДСКИХ ОПШТИНА БЕОГРАДА И ПАНЧЕВА, СРБИЈА

У земљишту, поврћу и лековитом биљу сакупљаним током 2007-2017 године на подручју приградских насеља Београда (општине Палилула и Сурчин) и Панчева (Институт за проучавање лековитог биља "Др Јосиф Панчић"), одређиван је садржај радионуклида (^{40}K, ^{238}U, ^{226}Ra, ^{232}Th,^{137}Cs) и тешких метала (As, Cd, Cu, и Pb). Резултати су показали да се током периода истраживања специфична активност ^{137}Cs у земљишту смањила са 16 Bqkg^{-1} на 3.9 Bqkg^{-1} (Палилула, Београд) и са 18 Bqkg^{-1} на 12 Bqkg^{-1} (Сурчин, Београд).

На подручју општине Палилула у земљишту је било највише Cu, а затим Pb > As > Cd, док је на испитиваним локалитетима општине Сурчин и Института за проучавање лековитог биља "Др Јосиф Панчић" концентрација Pb била највећа, а затим Cu > As > Cd. Добијени резултати су показали да близина индустријских постројења нема негативан утицај на пољопривредну производњу на испитиваним локалитетима и да су главни изовр контаминације земљишта и биљака радионуклидима и тешким металима фосфатна минерална ђубрива.

Кључне речи: радионуклид, тешки метал, загађење, пољопривредна производња