



Heavy metals, organics and radioactivity in soil of western Serbia

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ARTICLE INFO

Article history:

Received 9 June 2009

Received in revised form 3 December 2009

Accepted 18 December 2009

Available online 4 January 2010

Keywords:

Heavy metals

Organic pollutants

Radioactivity

Soil

Hydrocarbons

Dose rate

Organics

Radioactivity

Absorbed dose rate

ABSTRACT

Western Serbia is a region well-known for potato production. Concentrations of selected metals, polycyclic aromatic hydrocarbons (PAHs) and radioactivity were measured in the soil in order to evaluate the quality and characteristics. The examined soils (Luvisol and Pseudogley) showed unsuitable agrochemical characteristics (acid reaction, low content of organic matter and potassium). Some samples contained Ni, Mn and Cr above the maximal permissible concentration (MPC). The average concentration of total PAHs was 1.92 mg/kg, which is larger than the maximal permissible concentration in Serbia but below the threshold values in the European Union for food production. The average radioactivity of ²³⁸U, ²²⁶Ra, ²³²Th, ⁴⁰K and the fission product ¹³⁷Cs were 60.4 ± 26.2, 33.2 ± 13.4, 49.1 ± 18.5, 379 ± 108 and 36.4 ± 23.3 Bq/kg. Enhanced radioactivity in the soils was found. The total absorbed dose rate in air above the soil at 1 m height calculated for western Serbia was 73.4 nGy/h and the annual effective dose was 90 μSv, which are similar to earlier reports for the study region.

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1. Introduction

Soil is one of the most important environmental backgrounds, as it provides water and nutrients for plant production. In addition, soils are potential scavengers of pollutants and, thus, can inhibit their transfer to ground water. However, soil is subjected to contamination and its quality must be protected, in particular from organic and inorganic pollutants [1,2]. Various natural compounds that are products of chemical and biochemical processes in soil together with anthropogenic organic compounds, like aromatic hydrocarbons, polychlorinated aromatics, organic solvents, aliphatic hydrocarbons, at elevated levels, are known to be harmful to human health [3]. Polycyclic aromatic hydrocarbons (PAHs) represent a group of highly carcinogenic and mutagenic organic contaminants included in the “priority pollutants” listings produced by the United States Environmental Agency [4] and by the European Commission [5].

Natural radioactivity is wide spread in the earth's environment: in soil, plants, water, air, coal and phosphate deposits, among oth-

ers [6–12]. The natural radioactivity in soil is derived mainly from the ²³⁸U and ²³²Th parent series and natural ⁴⁰K. Natural environmental radioactivity and the associated external exposure due to gamma radiation depend primarily on the geological and geographical conditions [13].

The concentrations of pollutants in the soil of western Serbia have not been investigated until recently. Being mainly hilly with an altitude above 400 m, this area is typically used for potato cultivation and pasture. The studied region is shown as the shaded area in the map given in Fig. 1. The objective of the present work was to evaluate the level of pollution of this area in order to conclude whether the soil is suitable for potato production or not. Thus, information on pollution of soil of western Serbia in the context of its quality for crop and potato production is given in this work.

2. Materials and methods

Samples of different types of soil from the upper 30 cm of the ground in western Serbia were collected in 2004 using a spade and knife at the altitude of 400–900 m above sea level. Between 20 and 23 samples per ha were taken for a composite sample.

Concentrations of Cu, Zn, Pb, Ni and Cr in the samples were analyzed after hot plate digestion with a cHNO₃–H₂O₂ mixture under

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Table 1
The basic chemical characteristics of the examined soils.

	pH		CaCO ₃ %	Organic matter %	Total N %	P ₂ O ₅ mg/100 g	K ₂ O mg/100 g
	In H ₂ O	In KCl					
No. of samples	60	60	60	60	60	60	60
Average	4.43	5.63	0.35	2.37	0.18	9.35	20.10
Standard deviation	1.01	0.88	0.30	1.03	0.08	4.12	16.18
Min.	3.31	4.29	0	0.95	0.089	4	8.2
Max.	6.85	7.9	1.25	5.64	0.438	30.5	134

reflux conditions [14]. These elements were determined using an atomic absorption spectrophotometer (AAS), Model Varian SpectrAA 600 with the flame technique. An oxidizing air/acetylene flame was used to determine all elements except Cr, which required reducing flame stoichiometry. Determination of As was performed with an AAS hybrid technique using 3% NaBH₄ in 1% NaOH. The following wavelengths and slit widths were employed: Cu (324.8 nm; 0.7 nm), Zn (213.9 nm; 0.7 nm), Pb (283.3 nm; 0.7 nm), Ni (232.0 nm; 0.2 nm), Cr (357.9 nm; 0.7 nm), As (193.7 nm; 0.7 nm), Cd (228.8 nm; 0.7 nm) and Co (240.7 nm; 0.2 nm).

Polycyclic aromatic hydrocarbons were extracted from soil samples with supercritical CO₂ using a SFE (supercritical fluid extractor) Hewlett Packard 7680A, Palo Alto, USA, device. Soil extracts were analyzed by liquid chromatography (liquid chromatograph HP 1100 Waldbronn, Germany), using acetonitrile/water (35/75) as the mobile phase and a C-18 column of inner radius 2.1 mm and length 200 mm. A UV detector (DAD) was employed to confirm identification. The phosphorus (P₂O₅) and potassium (K₂O) content were measured by the AL method [15], which included extraction with a mixture of 0.1N acetic acid and 0.1N ammonium lactate solutions of pH value 3.7. After 2 h of shaking, the soil suspension was filtered and sulfuric acid and ammonium molybdate were added. A blue color developed after standing for 60 min at 95 °C. Phosphorus was determined using a Cary Varian spectrophotometer, at 830 nm. The potassium content was determined with the flame spectrophotometer Evans Halstead Essex, UK.

The organic matter content was determined by the wet digestion method described by Mebius [16]. The wet oxidation procedure included potassium dichromate with external heat and back titration to measure the amount of unreacted dichromate. Soil pH is a measure of the soil acidity or basicity. Usually the pH value is measured in a soil–water slurry and also in a soil–salt slurry (0.1 M KCl). pH value was measured on a CyberScan 510 pH-meter. The CaCO₃ content (%) was determined with a Scheibler Calcime-

ter spectrophotometer. The total N content was determined using the Kjeldahl method (Tecator Kjeltac Auto 1030, Tecator, Höganäs, Sweden).

Activity concentrations of ²³⁸U, ²³²Th, ⁴⁰K and fission products, such as ¹³⁷Cs, were determined by gamma spectrometry using a high purity Ge-detector (HPGe) ORTEC. Preparation of soil samples for gamma spectrometry was described by Krstić et al. [17].

3. Results and discussion

3.1. Inorganic chemistry

Based on laboratory analyses and in situ survey, the soils from the sampled locations were classified into two orders: automorphic soils, class organic matter-accumulating soils, type Luvisol and Pseudogley [18]. A total of 110 ha of soil area was included in this study.

The chemical characteristics (pH value in H₂O and KCl) and contents of CaCO₃, organic matter content (%), total N, P₂O₅ and K₂O of 60 samples from the Ah layer (0–30 cm depth) are given in Table 1.

The pH values in H₂O ranged between 3.31 and 6.85 (average 4.43); whereas pH in KCl ranged between 4.29 and 7.9 (average 5.63). Thus, the soils are quite acidic. This is reflected in the relatively very low concentrations of CaCO₃ (0–1.25%; average 0.35%, Table 1). The extremely acidic soils in the study area are considered unsuitable for potato cultivation. The optimal values of pH are in the range 5.5–6 [19–21].

The contents of organic matter ranged between 0.95% and 5.64% (average 2.37%). It has already been confirmed [22] that the soil in this region is generally poor in organic matter content. The total N concentration ranged between 0.089% and 0.438% (average 0.18%). The content of P₂O₅ ranged between 4 mg/100 g and 30.5 mg/100 g (average 9.35 mg/100 g). The content of K₂O, ranging between 8.2 mg/100 g and 134 mg/100 g (average 20.1 mg/100 g), represents a regional baseline acceptable for potato farming.

The concentrations of microelements and heavy metals (in mg/kg) in the soils are given in Table 2. The microelement contents are adequate for providing the necessary micronutrients to sustain healthy potato cultivation. However, in some soil samples the concentrations of Ni, Mn and Cr were above maximal permissible levels [23] and their transfer at elevated levels into plants may result in deleterious impacts. Determination of the total fractions of these elements which can readily transfer from soils to plants, followed up by an analysis of plant products in the study area, is therefore suggested. Steps to remedy the problem of soil acidity and high contents of Ni, Mn and Cr are also recommended before any cultivation of potato or other subsistence crops is introduced. Some of the steps suggested include, for example, calcification, phosphorylation and application of ion exchange resins [24,25].

3.2. Hydrocarbons

The soil samples were analyzed for fifteen of the most important polycyclic aromatic hydrocarbons (PAHs). The results are presented in Table 3, which show that benzo-(k) fluoranthene, benzo-(a)



Fig. 1. Map of Serbia. The studied region is represented as a dashed area.

Table 2
Concentration of microelements and heavy metals (mg/kg).

Sample	As	Cd	Co	Cr	Cu	Mn	Ni	Pb	Zn
No. of samples	60	60	60	60	60	60	60	60	60
Average	138.39	0.65	31.34	108.1	22.72	1144.23	229.41	47.41	64.8
Standard deviation	408.88	1.25	24.31	118.1	14.2	471.05	497.65	8.33	8.74
Min.	8.52	0.11	9.73	35.96	10.67	211.87	29.84	27.7	46.4
Max.	2474.69	7.42	155.51	910.53	91.81	2430.97	3356.93	65.81	85.78
MAC ^a		3.00		100.00	100.00		50.00	100.00	300.00
Optimum					10–20		40–100		20–80

^a Maximum allowable concentrations of harmful and hazardous substances in soil and irrigation water (Official Journal of SRJ 23/1994).

Table 3
Concentration of polycyclic aromatic hydrocarbons (PAHs) in soil (mg/kg of fully dry soil).

Sample	I	II	III	IV	V	VI	VII	VIII	IX	X	XI	XII	XIII	Tot PAHs
60	0.01	0	0.03	0.06	0.31	0.37	0.11	0.26	0.11	0	0.04	0	0	1.30
61	0	0	0.07	0.07	0.17	0.11	0.11	0.50	0.15	0.17	0	0	0	1.35
62	0	0.01	0.03	0.05	0.15	0.42	0.12	0.18	0.08	0.14	0	0.03	0	1.19
63	0.05	0.02	0.09	0.71	0.26	0.48	0.17	0.97	0.21	0.25	0.06	0.41	0	3.66
64	0	0	0.04	0.06	0.34	0.46	0.19	0.53	0.07	0.12	0.04	0	0	2.02
65	0	0.0	0	0.03	0.21	0.04	0.13	0.51	0.13	0.14	0.08	0	0.18	1.44
66	0.16	0.05	0.16	0.10	0.39	0.96	0.17	0.30	0.07	0	0.08	0.02	0.21	2.66
67	0	0	0.03	0.05	0.11	0.53	0.06	0.17	0.01	0	0	0	0.16	1.12
68	0	0	0	0.08	0.10	0.07	0.06	0.11	0.02	0	0	0	0.14	0.57
69	0.23	0.06	0.21	0.10	0.42	0.85	0.20	0.25	0.06	0	0	0.03	0.09	2.47
70	0.03	0.03	0.11	0.09	0.42	0.89	0.20	0.48	0.09	0	0.07	0.38	0.11	2.89
71	0.16	0.05	0.16	0.08	0.37	0.67	0.19	0.41	0.07	0	0	0	0.22	2.38
Average	0.11	0.04	0.09	0.12	0.27	0.49	0.14	0.39	0.09	0.16	0.06	0.17	0.16	1.92

(I) Naphthalene; (II) acenaphthene; (III) acenaphthylene; (IV) fluorene; (V) phenanthrene; (VI) anthracene; (VII) fluoranthene; (VIII) pyrene; (IX) benzo(a)anthracene; (X) chrysene; (XI) benzo(b)fluoranthene; (XII) benzo(g,h,i) perylene and (XIII) indeno (1,2,3 cd) pyrene.

pyrene and dibenzo-(a,h) anthracene were not detected among the PAHs.

The average concentration of total PAHs in dry soil was 1.92 mg/kg and this is larger than the mandated value according to regulations in Serbia [22], where the maximal content of PAHs in soil predicted for organic food production is defined as 1 mg/kg. The European Commission [5] determined the threshold values of organic pollutants for soils as 3 mg/kg for the sum of 16 PAHs in soils with organic matter below 8% [23]. If organic matter is above 8%, the limiting value of PAHs is 10 mg/kg.

There are a number of different techniques for soil remediation based on biological, chemical and physical treatments or combinations thereof. Bioremediation and phytoremediation are cost-effective technologies which require long treatment durations compared to all other techniques. The major advantages of these biological treatments are low-costs and the high potential for in situ or onsite treatments and no waste treatment is required [26–28]. No single remediation technology can be the solution for all PAH-contaminated soils. Integrated soil remediation technologies which combine separation and destruction of PAHs appear to be the way

forward in this technical field allowing improved removal efficiencies to be achieved [29,30].

3.3. Radioactivity

The activities (in Bq/kg) of ²³⁸U, ²²⁶Ra, ²³²Th and ⁴⁰K in soils of the selected locations are presented in Table 4. The fission product ¹³⁷Cs was identified in all examined samples. This nuclide originated from atmospheric nuclear weapons tests performed before 1965 and the Chernobyl accident that occurred in 1986. Since the half-life of this nuclide is 30.1 years, ¹³⁷Cs will be present in the soil and ecosystem of Serbia for a long time comparable to two centuries [13,14].

Table 4 shows that the average activities (Bq/kg) of ²³⁸U, ²²⁶Ra, ²³²Th, ⁴⁰K and ¹³⁷Cs match closely those reported in the literature for the region [31,32].

The concentration of all the radionuclides in the study area is within the permissible levels for crop production. ¹³⁷Cs is mostly fixed in soil and its possible transfer into plants is precluded; therefore it should not be regarded as a health hazard [33].

Table 4
Activity concentration of radionuclides in some locations.

Sample	Activity concentration of radionuclides, A [Bq/kg]				
	²³⁸ U	²²⁶ Ra	²³² Th	⁴⁰ K	¹³⁷ Cs
3	90 ± 30	42.2 ± 1.6	58.7 ± 2.4	441 ± 24	31.3 ± 1.3
21	60 ± 14	45.6 ± 2.7	70 ± 3	360 ± 15	33.5 ± 1.7
25	77 ± 28	36.2 ± 1.3	52 ± 2.2	408 ± 21	24.1 ± 1.1
26	62 ± 19	42.9 ± 2.8	63 ± 4	452 ± 25	28.7 ± 2
27	98 ± 35	43.8 ± 2.3	59 ± 4	446 ± 29	27.3 ± 1.9
37	12 ± 9	7.8 ± 0.7	18.5 ± 1.2	121 ± 15	2.6 ± 0.4
38	28 ± 22	9.9 ± 0.8	11.5 ± 0.8	227 ± 15	41.9 ± 1.8
45	80 ± 50	41.5 ± 1.9	60 ± 2.9	406 ± 25	87 ± 4
46	67 ± 22	38.5 ± 2	58 ± 3	410 ± 24	12.1 ± 1.5
51	47 ± 12	28.8 ± 2.7	46.1 ± 2.6	444 ± 19	57 ± 3
52	43 ± 18	27.9 ± 1.4	43.5 ± 2.5	458 ± 27	55.3 ± 2.3
Average ± SD	60.4 ± 26.2	33.2 ± 13.4	49.1 ± 18.5	379 ± 108	36.4 ± 23.3

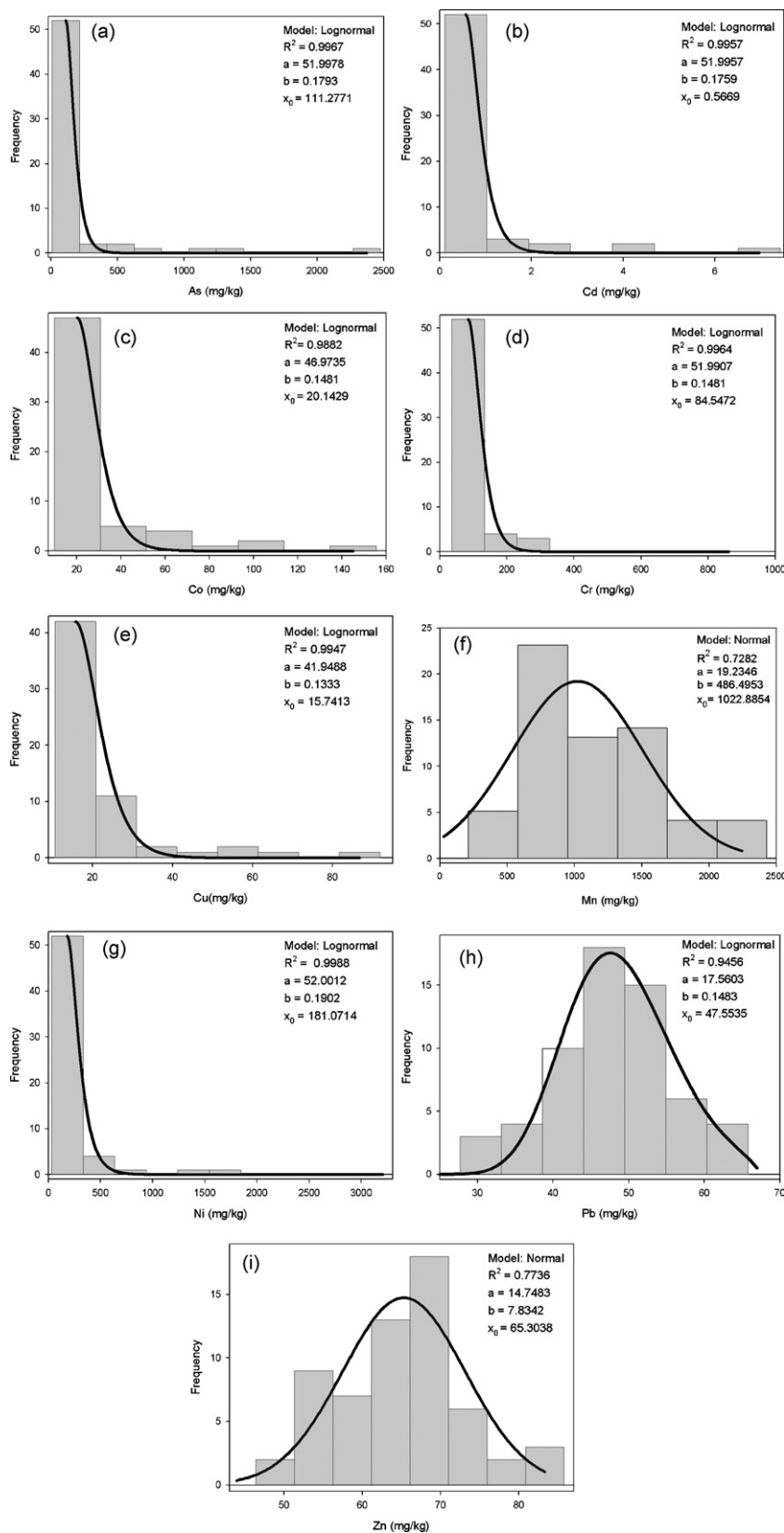


Fig. 2. (a–i) The frequency distribution of heavy metals in soil samples.

3.4. Statistical treatment of data

The frequency distribution of heavy metals in soil samples are shown in Fig. 2a–i. Concentrations of some heavy metals, As, Cd, Co,

Cr, Cu, Ni, and Pb are presented by log normal distributions which is given by formula $y = a \exp(-((\ln(x/x_0))^2/2b^2))$. This function was used to fit the experimental data using the graphical and statistical software called SigmaPlot version 10. Data obtained for Zn and Mn

Table 5
Spearman's correlation coefficients between trace elements (in mg/kg) and radioactive isotopes (in Bq/kg) in soil.

Variable	²³⁸ U	²²⁶ Ra	²³² Th	⁴⁰ K	¹³⁷ Cs	As	Cd	Co	Cr	Cu	Mn	Ni	Pb	Zn
²³⁸ U	1													
²²⁶ Ra	0.709*	1												
²³² Th	0.627*	0.945*	1											
⁴⁰ K	0.327	0.291	0.218	1										
¹³⁷ Cs	-0.054	0.018	0.073	0.182	1									
As	-0.109	-0.282	-0.218	-0.627*	-0.082	1								
Cd	-0.578	-0.797*	-0.770*	-0.246	0.241	0.501	1							
Co	-0.591	-0.309	-0.164	-0.473	0.273	0.627*	0.592	1						
Cr	-0.564	-0.873*	-0.891*	-0.309	-0.436	0.264	0.528	0.054	1					
Cu	-0.782*	-0.718*	-0.773*	-0.045	-0.082	0.036	0.546	0.355	0.682*	1				
Mn	-0.172	0.100	0.191	-0.491	0.354	0.645*	0.301	0.836*	-0.309	-0.054	1			
Ni	-0.700*	-0.709*	-0.764*	0	-0.127	0.245	0.733*	0.427	0.673*	0.836*	-0.18	1		
Pb	0.282	0.364	0.336	-0.136	0.654*	0.409	0.046	0.373	-0.554	-0.300	0.727*	-0.291	1	
Zn	-0.300	-0.491	-0.482	0.236	-0.418	-0.227	0.187	-0.173	0.545	0.591	-0.572	0.491	-0.736*	1

* $p < 0.05$.

Table 6
Average activity concentration of ²³⁸U, ²³²Th, ⁴⁰K in the soil, absorbed dose rate and annual effective dose.

Radionuclide	Average \pm SD (Bq/kg)	Absorbed dose (nGy/h)	Annual effective dose (μ Sv)
²³⁸ U	60.4 \pm 26.2	27.90	90
²³² Th	49.1 \pm 18.5	29.66	
⁴⁰ K	379 \pm 108	15.82	

presented in Fig. 2f and i were fitted according to normal distribution given by formula $y = a \exp(-((x - x_0)^2/2b^2))$. Information about parameters of distributions, and R^2 which show the goodness of fitting, are given in the graphs.

Spearman correlation coefficients between heavy metals and radioactivity were determined. Natural radioactivity in the studied area originated from the volcanic rocks in the basin of the Golija mountain. Spearman correlation coefficients between heavy metals and activity concentrations of natural radioactive isotopes and ⁴⁰K and ¹³⁷Cs are presented in Table 5. Significant negative correlation between radioactivity and Cd, Cr, Cu and Ni is seen (the Spearman correlation coefficient R for all data pairs is between -0.627 and -0.891; the level of significance p is less than 0.05, correlation is significant). Since the studied area is covered with volcanic terrains, the activity concentrations and some heavy metal concentrations are found to be high. It is seen in Table 5 that significant positive correlations occur between Cu and Cr and between Ni and Cd, Cu and Cr. A similar trend was confirmed by Zorer et al. [34].

Correlation between heavy metals and soil properties was also investigated. A significant correlation was determined between the content of heavy metals: Cd, Cu, Ni and Zn and the pH value. The reason is that the concentration of heavy metals, i.e. their solubility increases with increase in soil acidity. The Spearman correlation coefficient for this group of data pairs is between $R=0.345$ and $R=0.719$; the level of significance p is less than 0.05.

A weak correlation with the humus content was noted for Cd, Mn and Pb. The Spearman correlation coefficient for this group of data pairs is between $R=0.374$ and $R=0.450$. A similar correlation was established with the total N content. The weak correlation can be explained by the fact that the total content of these metals is easily accessible to plants entering into complexes with humus. A correlation between the humus content and Cs was confirmed ($R=0.654$) [35].

3.5. Absorbed dose rates in air due to radionuclides in soil and assessment of the annual effective dose

The external terrestrial gamma radiation absorbed dose in air at a height of 1 m above ground level was calculated using conversion

factors: 0.462 (nGy/h)/(Bq/kg) for ²³⁸U, 0.604 (nGy/h)/(Bq/kg) for ²³²Th and 0.0417 (nGy/h)/(Bq/kg) for ⁴⁰K (UNSCEAR, 2000) [13].

The following equation was used [13]:

$$\dot{D} = 0.462C_U + 0.604C_{Th} + 0.042C_K \quad (1)$$

where \dot{D} is the dose rate in nGy/h and C_U , C_{Th} and C_K are the specific activities in Bq/kg.

The gamma dose rate was calculated from the average concentration of ²³⁸U, ²³²Th and ⁴⁰K radionuclides and 27.90, 29.66 and 15.82 nGy/h was obtained, respectively. The value of the total absorbed dose rate is 73.4 nGy/h, which is in accordance with values determined for agricultural soil in Vojvodina, Serbia [36]. Similar values were reported by other authors [31,37,38]. Average dose rates in different countries ranged from 24 to 160 nGy/h; the population-weighted average dose in the region with 3/5 of the world population is 57 nGy/h [13].

The annual outdoors effective dose was calculated utilizing a conversion coefficient of 0.7 Sv/Gy to convert the absorbed dose in air into the effective dose in the human body. This calculation takes into account that people spend about 20% of the time outdoors (outdoor occupancy factor p is 0.2) and 8760 h is the annual exposure time.

The annual effective dose, D_E , due to gamma radiation from soil was calculated as

$$D_E = 0.7\dot{D}tp \quad (2)$$

The absorbed dose rates from outdoors terrestrial gamma radiation and the annual effective dose are given in Table 6. The result obtained for the annual effective dose is approximately 90 μ Sv, and this value is greater than the international average value of 70 μ Sv [13].

4. Conclusions

Considering the high acidity and low contents of organic matter and phosphate it can be concluded that the agrochemical characteristics of western Serbian soils are unsuitable for crop production, especially for potato. Improvement of these soil performances is possible by undertaking remediation steps, such as intense fertilization and calcification. In some locations the contents of Mn, Ni and Cr are above the maximal permissible levels; a situation probably resulting from acid leaching of the substrate.

Values of PAHs are somewhat larger than the limiting value defined by our law, but they are smaller than EU regulation values. Limiting values for PAHs are lower according to our regulations than those in the European Community, but we believe that this discrepancy will be resolved in the close future.

Statistical analysis of data was performed including correlations between heavy metals and radioactivity. In addition, data were fitted according to lognormal distribution and results are given in Fig. 2 and Table 5.

Radioactivity of the soils was not above the level that may hinder crop production. The external terrestrial gamma radiation absorbed dose in air at a height 1 m above ground level was calculated to be 73.4 nGy/h, which is typical for the region.

Acknowledgment

The authors would like to thank the Serbian Ministry of Science and Environment Protection for supporting this work through project No. 141023.

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