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Arsenic Content and Distribution in Agricultural Soils of Vojvodina Province, Serbia

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ABSTRACT
Arsenic (As) is metalloid designated as a pollutant in the environment due to its harmful effects on biota. Sources of arsenic soil contamination originate from both indigenous and anthropogenic inputs, including atmospheric deposition (mining, industry, dumpsites). In addition, As has been used in agriculture as a component of different agrochemicals. Contamination of groundwater with arsenic is a global issue. Arsenic in drinking water can affect human health; it is considered as one of the most prominent environmental causes of cancer mortality in the world. The content and retention of As in soils, as well as the other heavy metals, is highly dependent on the physicochemical properties of the soil. Vojvodina Province in the northern part of Serbia represents its most important agricultural area. The aim of this study was to determine the content and distribution of As in agricultural soils, its spatial distribution in different geomorphological units and soil types of Vojvodina Province, and to establish permanent monitoring. A grid superimposed on Vojvodina soil by means of a GIS tool has divided study area into 4 × 4 km units, each representing an area of 16 km^2. Total number of 1,370 bulked soil samples (0-30 cm depth) were taken from agricultural land. The samples were analysed for pseudototal content of As_T (after MW digesting the soil in $_{cc}HNO_3$ and H_2O_2) and available contents of As_{EDTA} (EDTA extraction). The concentrations of As were determined by ICP-OES. Basic soil properties were determined according to standard methods at the Laboratory for Soil and Agroecology of the Institute of Field and Vegetable Crops, Novi Sad, accredited according to the standard ISO/IEC 17025 (2005). The software used for the mapping and spatial analysis was ESRI ArcGIS Geostatistical Analyst 10. All statistical analyses were performed using the data analysis software system Statistica for Windows, version 13.2 (Dell 2015). The obtained results of As_T were within interval 1.0–31.4 mg kg^{-1}. The average concentration of As was 8.4 and 0.4, with median 8.3 and 0.3 mg kg^{-1} for pseudototal and available content, respectively. Only four samples were above 25 mg kg-1 for AsT, which is the national legislative threshold. These samples belong to the District of Srem, South Banat and two samples are from the South Bačka District. It was found that As_T content is negatively correlated with pH value and $CaCO_3$ and positively correlated with organic matter contents in soil. The effect of different soil particle size on availability of As was proven; availability decreases with higher proportion of clay and slit. As spatial distribution indicated, the majority of Vojvodina Province area has geochemical origin of As. Based on As concertation along geomorphological units, As_T is higher on mountains (10.4) and lower on the sandy area (5.6 mg kg^{-1}) with statistical difference among the other units. Oppositely, As_{EDTA} is higher on sandy area (0.6) and lower on the mountains (0.3 mg kg^{-1}), which proves effect of organic matter and clay on As availability. Average values of As_T concentrations according to the soil types coincides with main geomorphological units where soils have been formatted. As_T is higher in cambisol (10.9) and lower in arenosol (4.8 mg kg^{-1}). Average values of As in chernozem, most frequent soil type in Vojvodina, was 8.6 and 0.4 mg kg^{-1} for pseudototal and available content, respectively. The obtained results show that measured levels of As in the soil are not limiting factors for safe food production in Vojvodina. The results emphasize the importance of knowing the spatial determinants of geomorphological units and soil types in establishing a permanent monitoring.
KEY WORDS: soil, arsenic, heavy metals, geomorphological units

INTRODUCTION
Arsenic (As) is a metalloid, designated as a pollutant in the environment, due to its harmful effects on biota. Arsenic is an element and is a naturally occurring mineral found widely in the environment. Arsenic enters the environment naturally through ground water, mineral ore and geothermal processes. Sources of arsenic soil contamination originate from both indigenous and anthropogenic

inputs, including atmospheric deposition (mining, industry, dumpsites). In addition, As has been used in agriculture as a component of different agrochemicals (ATSDR, 2009).

The toxicity of arsenic is partly related to its form, valence state, solubility, rate of absorption and elimination from the body. Inorganic arsenic is generally more toxic than the organic one. Arsenic is non-soluble in water but its compounds arsenite and arsenate are highly soluble.

Among all the possibilities of arsenic entering the food chain, arsenic contamination of rice (*Oryza sativa* L.) endangers the global food security and human health (Meharg et al., 2009; Seyfferth et al., 2014, Neumann et al., 2017). In flooded paddy soil, arsenic is mobilized through reductive dissolution of arsenic bearing iron(III) oxides. Climate warming could alter availability and plant uptake of arsenic, because soil warming increases arsenic availability in the Rice rhizosphere (Neumann et al., 2017).

Contamination of groundwater with arsenic is another global issue. The natural occurrence of arsenic in groundwater constitutes a major setback in the provision of safe drinking water to millions of people in Asia and worldwide (Amini et al., 2008; Thakur et al., 2011). Arsenic in drinking water can affect human health; it is considered to be one of the most prominent environmental causes of cancer mortality in the world (Shrivastava et al., 2014).

In Vojvodina, 800,000 people drink water with arsenic (mostly in Central Banat), in Hungary 1,800,000 people (Dalmacija et. al, 2010).

The content and retention of As in soils, as well as the other heavy metals, is highly dependent on the physicochemical properties of the soil (Adriano, 2001).

Vojvodina Province in the northern part of Serbia represents its most important agricultural area, therefore, soil protection is of crucial importance for economic development of the Vojvodina Province.

The aim of this study was to determine the content and distribution of As in agricultural soils, its spatial distribution in different geomorphological units and soil types of Vojvodina Province, and to establish permanent monitoring.

MATERIAL and METHOD

Study area

Vojvodina is the autonomous province in Republic of Serbia and occupies the area between 44^o 38’ and 46^o 10’ northern latitude and 18^o 10’ and 21^o 15’ eastern longitude. Vojvodina is situated in the south-eastern part of the Pannonian (Carpathian) Basin, the plain that remained when the Pliocene Pannonian Sea dried out. Consequently, Vojvodina is rich in fertile loamy loess soil. Regarding the distribution of soil types, as much as 60% of the Vojvodina Province soil is chernozem, which is considered ideal for crop production due to physical and chemical properties. Other fertile soil types with considerable areas include hydromorphic black soils (16%), and the alluvial soils (9%) (Skoric et al., 1985). Out of the total area of the Vojvodina Province (21,506 km^2), the agricultural land takes 16,940 km^2 or 80%. Vojvodina is a typical rural region in which ploughed land and gardens cover 90% of the agricultural land (STAT. YEARB. SERB., 2015). More than 60% of this lowland area is covered by loess and loess-like sediments at terraces and loess plateaus (Markovic et al., 2008). Beside four lowland geomorphological units (sandy area, loess plateaus, upper Pleistocene, alluvial terraces and alluvial plains), the most distinctive landforms of the Vojvodina region are two mountains: Fruška Gora Mountain, which is situated between the Danube and Sava rivers, and Vršac Mountains, which are located in the south-eastern part of the region close to the border with Romania (Fig. 3).

The climate of Vojvodina is moderate continental, with cold winters and hot and humid summers, wide range of extreme temperatures and unequal distribution of rainfall per months, which leads to different aridity types. The mean annual air temperature is 11.1°C and the annual amount of precipitation is about 600 mm (Hrnjak et al., 2013; Tosic et al., 2014). Potential vegetation is mostly replaced by crops at lowland zone. Forest vegetation is limited to narrow belts close to the rivers, Deliblato sandy area and two mountains. Vojvodina has a population of about 2 million inhabitants (about 27% of Serbia's total).

Sample collection and processing

A grid superimposed on Vojvodina land by means of a GIS tool GIS ArcView 10 has divided the land of the Vojvodina into 4 × 4 km units, each representing an area of 16 km^2 (Fig. 1). Samples were taken from the defined spots of agricultural land, or the centre of each quadrant using GPS manual receivers (GPS receivers Trimble GPS GeoXH 3000, Trimble GPS Juno SC, Terrasync Professional software). In case a sample could not be taken from the centre of a quadrant for any reason, the correction sampling was performed at the nearest corresponding location, up to 500 m away from the defined spot, rarely up to 1,000 m away (Fig. 1). If the defined spots were in the urban area, the samples were taken from urban gardens and garden plots.

Total of 1,370 bulked soil samples were taken during the period 2011-2013. The topsoil samples were taken from 0-30 cm soil layer. This depth was chosen as the most active zone of field crops root systems. The samples were taken using a soil drill agrochemical probes and stored in polyethylene bags. Samples were stored in cold (+4°C) during transport. One composite sample represented 10-15 subsamples from random points within about 300 m^2 grid in each sampling site. The initial quantity of samples was approximately 1.5 kg. The soil samples were air-dried at the room temperature, milled and sieved to <2 mm particle size, in accordance with ISO 11464 (2006).

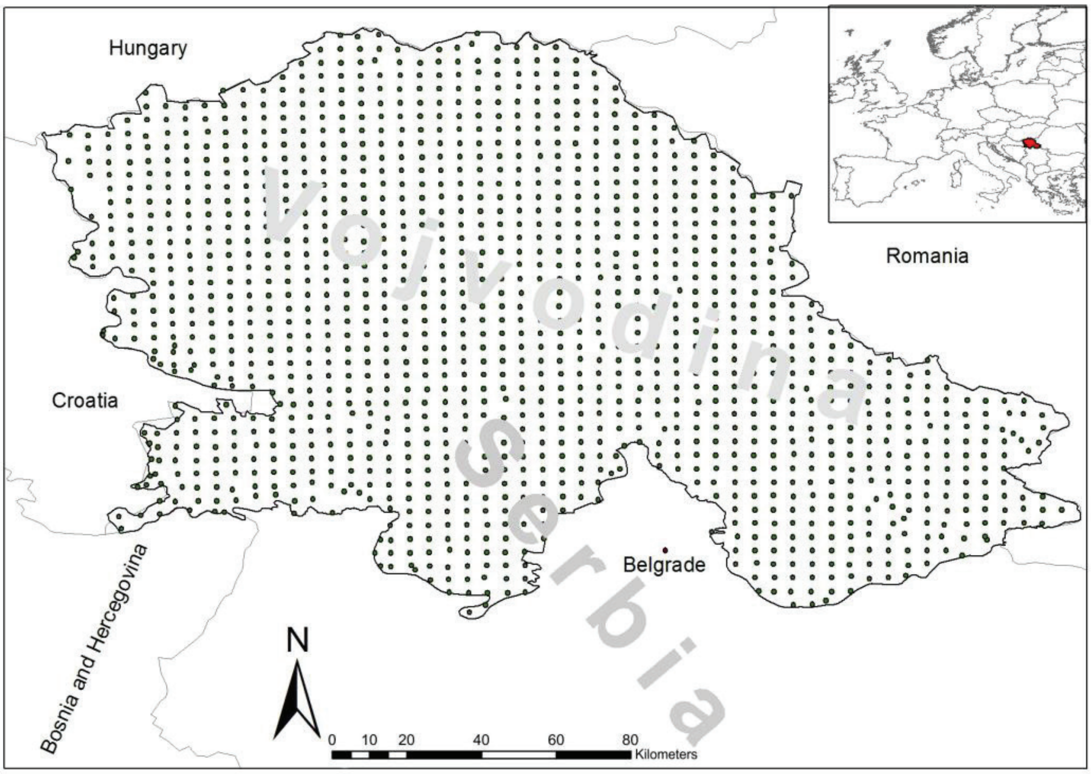

Figure 1. Layout of 1.370 taken soil samples

Laboratory analyses

All laboratory analyses were performed at the Laboratory for Soil and Agroecology of the Institute of Field and Vegetable Crops, Novi Sad, accredited according to the standard ISO/IEC 17025 (2005).

The pH value in 1:5 (V/V) suspension of soil in 1 mol/L KCl was determined using glass electrode according to ISO 10390 (2010). The carbonate content ($CaCO_3$ content) was determined according to ISO 10693 (1995) by volumetric method. The organic matter content was measured by oxidation using the sulphochromic oxidation method by ISO 14235 (1998). Readily available phosphorus (P_2O_5) and available potassium (K_2O) were extracted by ammonium lactate extraction (AL method by Egner and Riehm, 1960), and measured by the means of spectrophotometry and flame photometry, respectively. Particle size distribution was determined in the <2 mm fraction by the pipette method (Van Reeuwijk, 2002). The size fractions were defined as clay (<2 µm), silt (2-20 µm), fine sand (20-200 µm) and coarse sand (200-2,000 µm).

The samples were analysed for "pseudo-total" contents of As (e.g. As_T) after digesting the soil in concentrated HNO_3 and H_2O_2 ($5HNO_3$: $1H_2O_2$, and 1:12 solid:solution ratio) by stepwise heating up to 180°C using a Milestone Vario EL III for 55 min. The concentration of elements was determined by ICP-OES (Vista Pro-Axial, Varian) in accordance with US EPA method 200.7:2001. Quality control was periodically carried out with reference materials ERM CC 141 and deviations were within ±10% of the certified values. Available As concentrations (e.g. EDTA-extractable As_{EDTA}) were determined by the EDTA extraction protocols for IRMM BCR reference materials CRM 484: 5 g soil/50 ml EDTA concentration 0.05 mol/L pH=7.00.The concentration of available As was determined by ICP-OES (Vista Pro-Axial, Varian).

Geochemical mapping

The GIS mapping technique was used to produce the spatial distribution maps of arsenic concentrations in Vojvodina Province soil. The software used for the mapping and spatial analysis was ESRI ArcGIS Geostatistical Analyst 10. The arsenic concentration was interpolated with the geostatistical analyst using a spatial interpolation method of inverse distance weighting (IDW) from ArcGIS 10. Grid used 4 × 4 km units with available input points.

Statistical analysis

Parameters of descriptive statistics (minimum, maximum and mean value, median value, standard deviation, coefficient of variation and percentiles) were shown for arsenic content (mg kg^{-1}) in agricultural soils of Vojvodina Province. All statistical parameters were shown in tables and box-plots graphs. The significance of the differences in the arsenic content between the geomorphological units and soil types were determined using the Duncan multiple range test ($p \leq 0.05$). In order to confirm the relationship among content of arsenic and different chemical traits, a Pearson's correlation analysis was applied to dataset. All statistical analyses were performed using the data analysis software system Statistica for Windows, version 12 (Dell 2015).

RESULTS and DISCUSSION

Arsenic concentration in soil

The obtained results of As_T were within interval 1.0-31.4 mg kg^{-1}. The average concentration of As was 8.4 and 0.4, with median 8.3 and 0.3 mg kg^{-1} for pseudototal and available content, respectively. (Fig. 2). Variation coefficient value CV (31.2%) for As_T points out medium heterogeneity of tested soil samples (Fig. 2). The obtained average concentrations of As suited to concentration for non-contaminated soils which ranged from 0.1 to 10 mg kg^{-1} (Kabata-Pendias and Pendias, 2001).

Based on the GEMAS project, median As concentrations in an aqua regia extraction determined by ICP-MS were 5.7 mg/kg for the agricultural land (Ap horizon, 0–20 cm) (Tarvainen et al., 2013). In present study, median value was higher than EU median value and suited with median value in the agricultural soils of southern Europe, which amounted to 8 mg kg^{-1}. The dominant feature is the southern margin of the former glacial cover seen in the form of a sharp boundary between northern and southern European As concentrations. Based on the GEMAS, the As content in grazing land (0–10 cm) from Vojvodina Province area was within 5.75 and 9.68 mg kg^{-1} (Tarvainen et al., 2013).

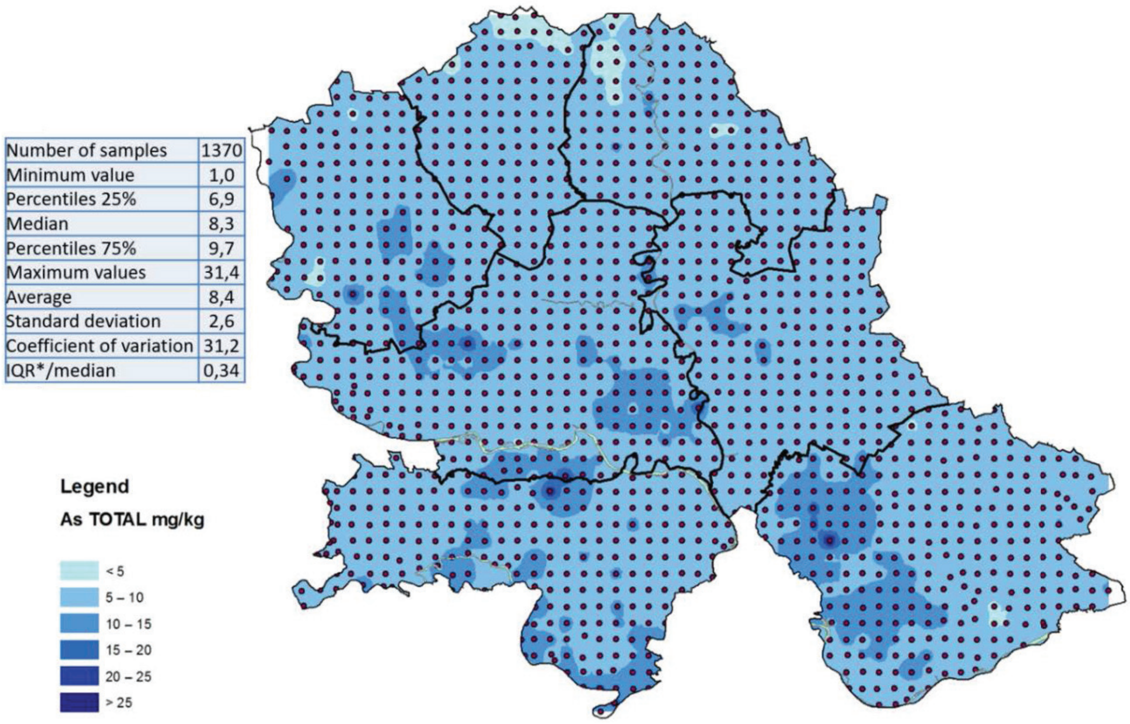

Figure 2. Spatial distribution of arsenic with statistical summary of total content (mg kg^{-1}) in agricultural soils of Vojvodina Province

Table 1
Correlation coefficients between total, available content of arsenic and basic physicochemical soil properties, for observed samples of agricultural soils in Vojvodina

	As_T [mg kg^{-1}]	As_{EDTA} [mg kg^{-1}]
pH KCl	-0.089**	-0.030
pH H_2O	-0.058	-0.025
$CaCO_3$[%]	-0.112**	-0.119**
OM [%]	0.117**	0.046
N_T[%]	0.097**	0.018
AL-P_2O_5[mg/100g]	-0.031	0.209**
AL-K_2O [mg/100g]	0.037	0.144**
Clay [%]	0.176**	0.013
Slit [%]	0.398**	-0.075*
Fine sand [%]	-0.340**	0.015
Coarse sand [%]	-0.024	0.063*
As_T[mg/kg]	1.000	0.053
As_{EDTA}[mg/kg]		1.000

* p<0.05; ** p<0.01

According to the established correlations shown in Table 1, As_T was found to be significantly negatively correlated with pH value (in KCl) and $CaCO_3$ content and significantly positively correlated with organic matter contents and total nitrogen content in soil. Available content of As_{EDTA} was significantly negatively correlated with $CaCO_3$ content.

Available content of As_{EDTA} was significantly positively correlated with nutrients (easily accessible phosphorus and potassium), which indicates that this element can be introduced in the soil using mineral fertilizers.

Clay fraction was in significant positive correlation (r=0.176) with As_T content, while was not in significant correlation with As_{EDTA} content. Silt fraction content was in significant positive correlation (r=0.398) with As_T content, while silt fraction content was in significant negative correlation (r=-0.075) with available As_{EDTA} content (Tab. 1). Fine sand fraction was in significant negative correlation (r=-0.340) with As_T content, while coarse sand fraction was in significant positive correlation (r=0.063) with As_{EDTA} content (Tab. 1). Based on established correlations, the effect of different soil particle sizes on availability of As was proven. Availability decreases with higher proportion of clay and slit and opposite, availability increases with higher proportion of sand. In general, clay particulates present negative charge, tends to undertake sorption of cations, and they are associated with As retention in soil (Hooda, 2010).

Only four samples were above 25 mg kg^{-1} for As_T, which is national maximum allowable concentration (MAC) for agricultural soils as prescribed by the laws of the Republic of Serbia (OG 23/94, 1994). These samples belong to the District of Srem, South Banat and two samples are from the South Bačka District.

Arsenic concentration in soil along geomorphological units

Based on As concentration along geomorphological units (Bukurov, 1972), As_T is higher on mountains (10.4 mg kg^{-1}) and lower on the sandy area (5.6 mg kg^{-1}) with statistical difference among the other units (Fig. 3, 4). Oppositely, As_{EDTA} is higher on sandy area (0.6 mg kg^{-1}) and lower on the mountains (0.3 mg kg^{-1}), which proves effect of organic matter and clay on As availability.

As spatial distribution indicated, the majority of Vojvodina Province area has geochemical origin of As. Regarding the pedogenesis of Vojvodina soils, the relief is the dominant factor compared to the other pedogenetic factors. Alluvial plains and mountains have higher geological and pedological diversity, unlike sandy areas, loess plateaus, Upper Pleistocene and alluvial terraces (Zivkovic et al.,1972).

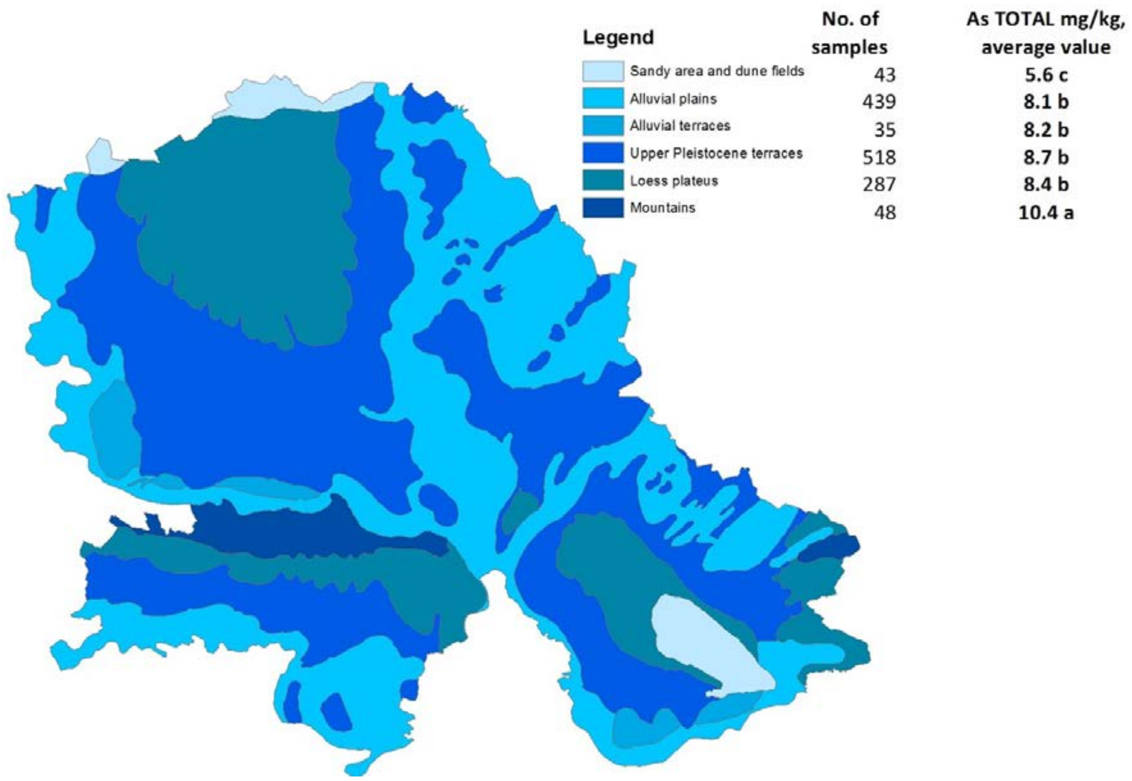

Figure 3. Spatial distribution of arsenic along geomorphological units with statistical summary of pseudototal content (mg kg^{-1}) in agricultural soils of Vojvodina Province, values are marked with same letter do not differ statistical significance for the level of importance α=5% (according to the Duncan multiple range test)

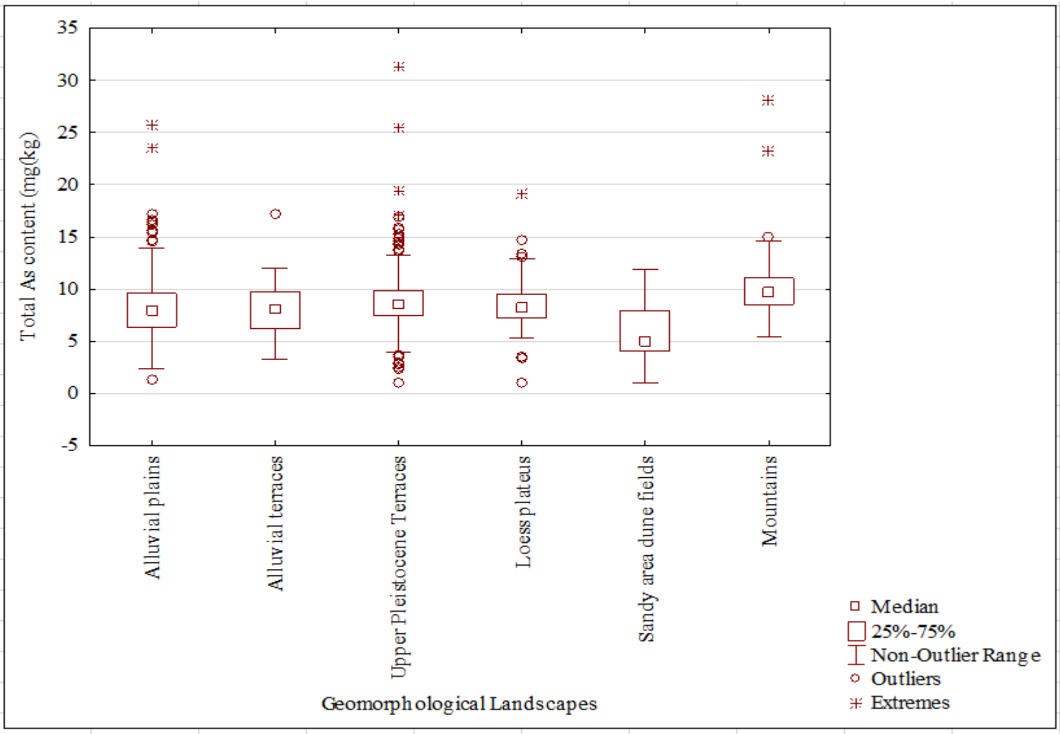

Figure 4. Box-plot graphical display of pseudototal arsenic content (mg kg^{-1}) along geomorphological units of Vojvodina Province

Arsenic concentration in soil along different soil types

The distribution of As in soils varies with soil type, depending on the nature of the parent material. Average values of As_T concentrations according to the soil types coincide with main geomorphological units where soils had been formatted.

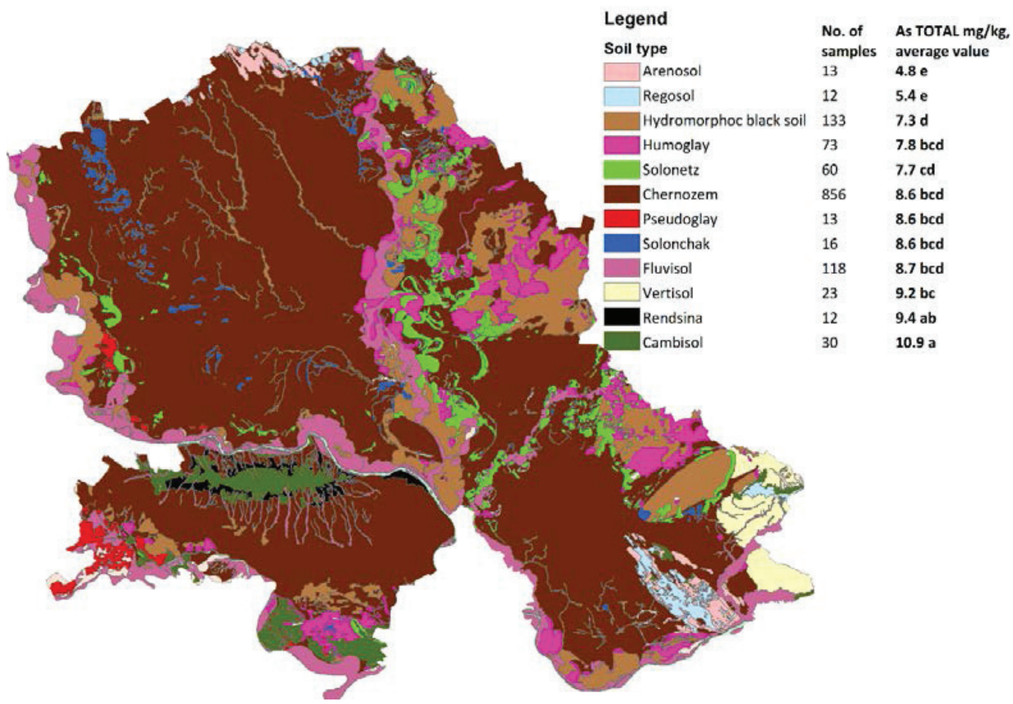

Figure 5. Spatial distribution of arsenic along different soil types with statistical summary of pseudototal content (mg kg^{-1}) in agricultural soils of Vojvodina Province

As_T is higher in cambisol (10.9 mg kg^{-1}) which is typical soil type on mountains and lower in arenosol (4.8 mg kg^{-1}) which is typical soil type on sandy area (Fig. 5,6).

According to established statistical difference between soil types, weakly developed soils arenosol and regosol are separated into one group (Fig. 5).

Generally, comparing the survey of As content in different soil types in soils of central Serbia (Mrvić et al., 2013), distribution of As content along soil types is similar, but in present study higher concentration was established.

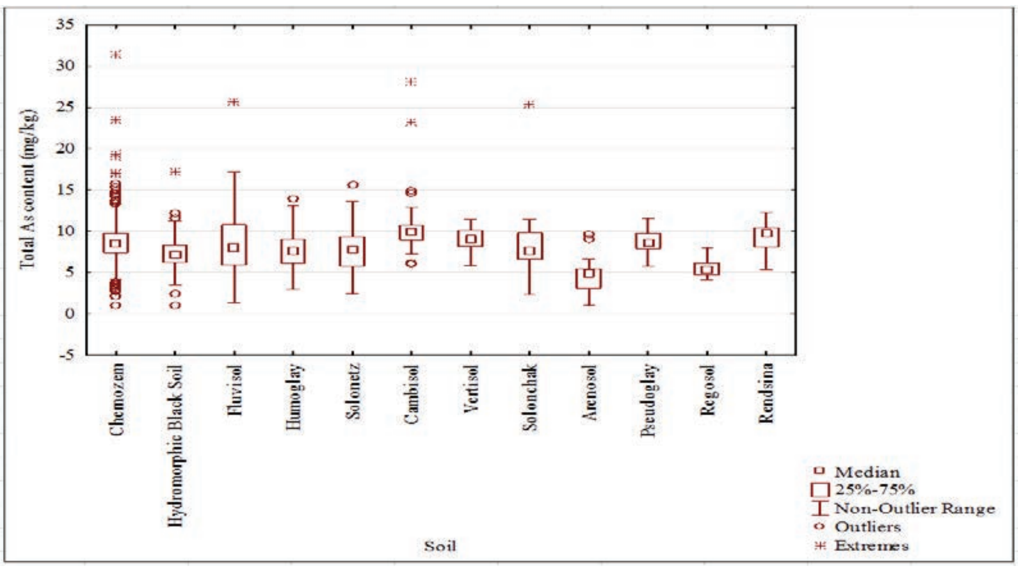

Figure 6. Box-plot graphical display of pseudototal arsenic content (mg kg^{-1}) along soil types of Vojvodina Province

Average values of As in chernozem, most frequent soil type in Vojvodina, was 8.6 and 0.4 mg kg^{-1} for pseudototal and available content, respectively. Median value was 8.5 mg kg^{-1} for As_T pseudototal content, which is much higher than reported median value for central Serbia area of 4.7 mg kg^{-1} (Mrvić et al., 2013).

CONCLUSIONS

The average concentration of As in agricultural soils of Vojvodina Province was 8.4 mg kg^{-1} for pseudototal content which suited to concentration for non-contaminated soils.

Based on established correlations, the effect of different soil particle sizes on availability of As was proven. Availability decreases with higher proportion of clay and slit.

As spatial distribution proved, the majority of Vojvodina Province area has geochemical origin of As.

The obtained results show that measured levels of As in the soil are not limiting factors for safe food production in Vojvodina. The results emphasize the importance of knowing the spatial determinants of geomorphological units and soil types in establishing a permanent monitoring.

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