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## EVALUATION OF TRACE ELEMENTS MPC IN AGRICULTURAL SOIL USING ORGANIC MATTER AND CLAY CONTENT

**ABSTRACT:** The aim of this paper is to investigate the contribution of the influence of organic matter and clay content on the value of maximum permissible concentrations (MPC) of trace elements Pb, Ni, Cr, and Cd. The investigation was conducted on agricultural soil in the territory of Veliko Gradište Municipality. There were analyzed 82 samples of eutric cambisol type soil, 17 samples of chernozem soil, and 32 samples of sandy soil. In the composite soil samples, taken from a depth of 0–30 cm, main parameters of soil fertility (pH, P<sub>2</sub>O<sub>5</sub>, K<sub>2</sub>O, CaCO<sub>3</sub>, soil organic matter – SOM), the content of the clay fraction, and total forms of Pb, Ni, Cr, and Cd were determined. Interpretation of the obtained results was carried out in relation to the MPC of trace elements defined in the Regulations (*Official Gazette*, 88/2018). Based on the ratio of the defined MPC and corrected values whose calculation includes the values of the organic matter and clay content, there was determined the correlation concerning the content of organic matter and the content of clay fractions, respectively, in the tested samples. In addition, the content of Cr and Pb in tested types of soil still did not exceed the adjusted MPC value. As for Cd and Ni, there was no deviation from the established and modified values of MPC. Concludingly, the research should be continued and supplemented by data for other types of soils, which would represent a base for a further assessment of the applicability of the existing regulations taken from Dutch sources and incorporated into Serbian Regulations (*Official Gazette*, 88/2018).

**KEYWORDS:** soil, trace elements, maximum permissible concentration (MPC), soil organic matter (SOM), clay

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## INTRODUCTION

It is generally recognized that the climate, especially air temperature and precipitation, presents the most important factor which regulates the level of organic matter in the soil (Alvarez and Lavado, 1998). The study of Lemenih and Itanna (2004) indicates that the content of soil organic matter increases with the amount of precipitation and decreases with the increases in temperature.

In similar climatic conditions, the organic matter content in the soils of fine texture (clayey soils) is two to four times higher than in the rough texture (sandy) soils (Prasad and Power, 1997). Clayey soils accumulate organic carbon rather quickly, while the sandy soils cannot accumulate it even after 100 years of a high input of organic matter (Freibauer et al., 2004).

Soil organic matter (SOM) is a complex matter system and is of great importance for all processes that occur in the soil, as the nutrients are accumulated within it. It is a source of fertility, contributing to soil aeration in a way that it reduces the soil density, improves the infiltration of the soil, and increases its water and air capacity (Vidojević, 2016). SOM content is of vital importance for the functioning of the ecosystem and has an important effect on the soil structure, the capacity for water retention in the soil, the capacity for the exchange of the cation, and the ability of the soil to form complexes with metal ions, as well as to keep the nutrients (Van Keulen, 2001).

The organic matter in soil affects not only the color of soil, its physical properties, and adsorption capacity of the mineral soils cations, but also the supply of plant accessible nutrients through the expressed exchange of nutrients and release of N, P, and S from organic forms, the extraction of elements from soil minerals, etc. (Miljković, 1996).

Soil can be structural and non-structural. The structural aggregates are formed by joint action of a number of factors which may be classified into five groups: soil, air, biological factors, anthropogenic factors, and time (Dugalić and Gajić, 2012).

In sandy soils or sands, the mechanical elements are generally not interconnected. Loam and clay soils can be structural, non-structural, or slightly structural. In general, at elevated content of the clay particles, the content of organic carbon in the soil tends to be higher. The reason for this is the connection between the surface of the clay particles and organic matter which inhibits the degradation process. Soils with higher clay content have a greater potential for the formation of aggregates.

Sandy soils typically contain less organic matter than the soils with a more delicate texture, loam or clay (Van-Camp et al., 2004). This is because, generally, low moisture content and high aeration in sandy soils result in faster oxidation of the organic matter in comparison with heavier soils.

The level of organic carbon in the soil is closely related to the soil structure and is one of the major factors of aggregation (Bronick and Lal, 2005). Likewise, the content of organic matter plays an important role in soil aggregation. This primarily refers to freshly formed and multivalent cation-saturated humic

and ulmic acids that, after the coagulation, bind particles of the mechanical fractions of clay, silt, and sand in stable structural units. Aggregate stability and preservation of the organic carbon levels in the soil depend greatly on the soil texture. The effect of soil organic carbon on the soil structural stability is enhanced in soils containing a low percentage of the clay fraction (Wuddivira and Camps-Roach, 2007).

The content of organic matter and clay fractions, as well as soil pH, affects the mobility of trace elements. In soils of pH range from 5.50 to 8.00 chromium (Cr) is nearly insoluble, and the solubility of cadmium (Cd) decreases with the increased pH values so that pH values above 7.50 lead to its immobilization (Kabata Pendias, 2011). In soils with pH values from 3.80 to 7.1 Cd is less mobile than nickel (Ni) (Adriano, 2001).

High concentrations of trace elements in the soil can affect the soil's fertility and may represent an ecological and human health risk if they enter the food chain or leach into receiving waters (Daskalopoulou et al., 2014).

Contamination by trace elements is a potential risk to the crops, animals, and humans because of their toxicity, persistence, bioaccumulation and biomagnification in the food chain (Rao et al., 2011). By incorporating the influence of organic matter content and clay fraction into the MPC (maximum permissible concentrations) calculation, the potential danger of determined trace elements content for the environment is more realistically considered. This concept was taken from the Netherlands (MvV, 2000).

The main difference between the two existing regulations of the Republic of Serbia (23/1994 and 88/2018) classification 23/94 and 88/2018 is in the reported values of certain trace elements. Also, for the interpretation of whether a trace element is below or above the MPC at a given location, the modified limit values are used according to Regulation 88/2018, which presents the result of the content of clay fraction and organic matter influence.

Based on the data obtained in a previous study (Institute of Soil Science, 2014), this study aimed to determine the influence of the organic matter and clay content in the soil and evaluate the established maximum permissible concentrations of trace elements (*Official Gazette RS*, 23/1994) following the Regulation (*Official Gazette RS*, 88/2018).

## MATERIALS AND METHODS

The Municipality of Veliko Gradište is located in the northeastern part of Serbia, in the foothills of the Carpathians and Homolje mountains, at the entrance to the Đerdap Gorge (Iron Gate). In the west, it borders with the Municipality of Malo Crniće, in the southeast with the Municipality of Kučevo, and in the east with the Municipality of Golubac (X: 534771, Y: 4948075). In the north, the municipality is bordered by the Danube that separates it from neighboring Romania in a length of 20 km. The municipality belongs to Braničevo district and covers an area of 344 km<sup>2</sup>.

The study territory belongs to the area of moderate continental climate with clearly derived seasons and almost no difference in the characteristics between the lower and higher terrains (Stanojković-Sebić et al., 2015).

In the area of study and according to the sampled soil distribution (Figure 1), the most represented soil type was eutric cambisol – brown forest soil, which is primarily the result of the favorable climate, the substrate on which the soil is formed, and the influence of the relief and vegetation present at its creation. This type is mainly formed on Miocene sediments of the lighter mechanical composition, or loess and old alluvial deposits. After the composition, it is slightly heavier loam, plastic and sticky.

The production value of eutric cambisol is high because this soil is among the very deep soil types of neutral and weakly acidic reaction. It has good physical properties when it was formed on the loess and has medium content of nutrient elements, except the available phosphorus.

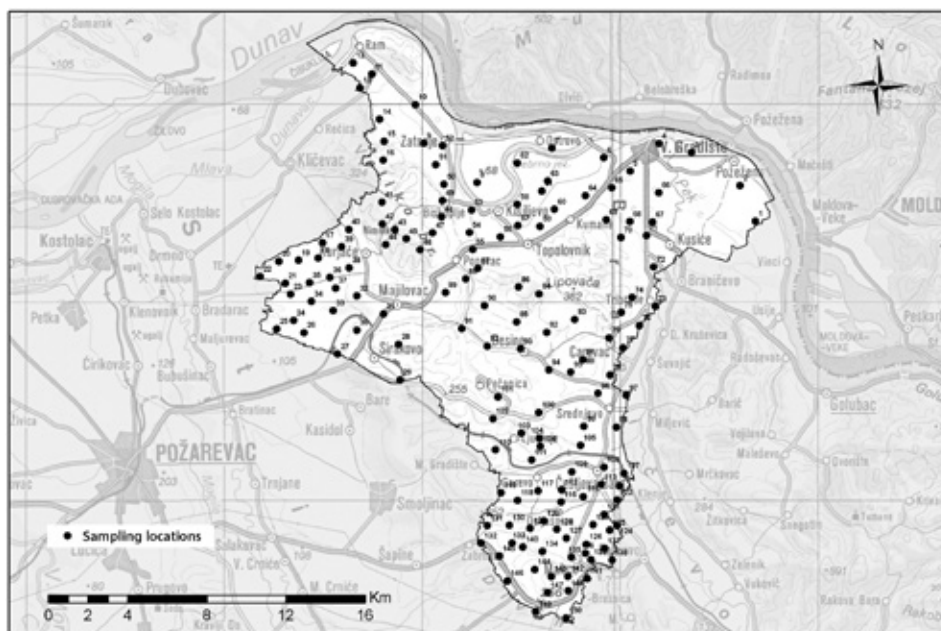


Figure 1. The position of soil samples

In the study area, chernozem soil occurs on smaller areas, on the loess deposits, and the contact zone of loess and sand. Particle size distribution varies from loam to sandy clay loam. The usual chemical analyses show that the chernozem is calcareous throughout the whole depth and that the share of carbonates increases with the depth, although an acid reaction can occur in leached chernozem. The soil reaction is usually slightly alkaline with adsorptive complex highly saturated with cations, mainly calcium. The share of humus is over 3%, while the share of available phosphorus and potassium varies from plot to plot.

A significant area of study is occupied by sandy soils, as follows: Aeolian quicksand, loess sand, and brown sand. Production value of sandy soils is generally low, but it can vary depending on the involvement of pedogenetic processes that go towards the creation of brown steppe soils and shallow chernozem on the sand. Soil profiles on sands are distinguished by the surface parts color and the share of humus in them. Substantially, their soil reaction in surface layers is neutral or slightly alkaline.

Composite random sampling (15–20 single samples) of an agricultural soil was carried out at pre-defined locations (Figure 1), from the depth of 0–30 cm. There were analyzed 82 samples of eutric cambisol soil, 17 samples of chernozem soil, and 32 samples of sandy soil. The soil typology was taken from the pedological map published in a previous study (Institute of Soil Science, 1975; WRB, 2015).

A total number of 131 composite soil samples were prepared for analyses in accordance with SRPS ISO 11464: 2004 – Pretreatment of samples for physical-chemical analyses, and sieved through a sieve of 2 mm in diameter. Soil acidity (pH in 1M KCl, v/v-soil: 1M KCl = 1:5) was analyzed potentiometrically, using glass electrode (SRPS ISO 10390, 2007); available phosphorus ( $P_2O_5$ ) was analyzed spectrophotometrically, and available potassium ( $K_2O$ ) by flame emission photometry, using AL-method according to Egner-Riehm (Riehm, 1958); calcium carbonate ( $CaCO_3$ ) was determined using the volumetric method (SRPS ISO 10693: 2005); SOM (soil organic matter) was calculated using the following formula: SOM content (%) = total organic C (%) x factor 1.724 (Džamić et al., 1996), using the total C content obtained on elemental CNS analyzer Vario EL III (Nelson and Sommers, 1996) and carbonate content.

The granulometric composition was analyzed by determination of particle size distribution in mineral soil material, using the standardized method by sieving and sedimentation (ISO 11277: 2009(E), 2009).

Determination of the total trace elements forms (Pb, Ni, Cr, and Cd) was done by inductively coupled plasma-atomic emission spectrometry – THERMO iCAP 6300 Duo (radial/axial view versions) ICP-OES, after the digestion of the samples with aqua regia (ISO 11466:1995, 1995; ISO 22036:2008, 2008).

Reference soil NCS ZC 73005, Soil Certificate of Certified Reference Materials approved by China National Analysis Center Beijing China, and reagent blanks were used as the quality assurance and quality control (QA/QC) samples during the analysis.

The results of the conducted soil analysis represent the arithmetic means of three replicates of each sampling. Statistical methods that were applied in the data processing were descriptive statistics and correlation, using the statistical program SPSS 18.0.

The interpretation of the content of trace elements in the soil samples was done using the Rule book of permissible concentrations of dangerous and hazardous materials in soil and water for irrigation and methods for analysis, in which MPC for the analyzed trace elements are as follows: Cd = 3 mg kg<sup>-1</sup>, Cr = 100 mg kg<sup>-1</sup>, Ni = 50 mg kg<sup>-1</sup>, Pb = 100 mg kg<sup>-1</sup> (*Official Gazette of RS*, 23/1994).

These values were used in the formula for calculating the limit and remediation values defined in the Regulation on the program of systematic monitoring of soil quality via indicators for the assessment of soil degradation risk and methodology for the creation of remediation programs (*Official Gazette of RS*, 88/2018).

## RESULTS AND DISCUSSION

The results of the present study showed that the soil reaction of chernozem ranged from highly acidic (in one sample of leached chernozem) to alkaline (pH in 1 M KCl was 4.00–7.35), of sands ranged from acidic to alkaline (pH in 1 M KCl was 5.10–7.65), and of eutric cambisol from highly acidic to neutral (pH in 1 M KCl was 3.60–7.05). According to the carbonate content, the chernozem soil samples were in the range of non-calcareous to slightly calcareous (below detection limit of the method, BDLM–2.94%); sands soil samples were in the range of non-calcareous to medium carbonate (BDLM–9.33%), and eutric cambisol soil samples ranged from non-calcareous to slightly calcareous (BDLM–1.91%). The content of organic matter in the chernozem (2.69–4.75%), sands (1.38–4.33%), and eutric cambisol (2.09–5.06%) soil samples were in the range of medium to high.

*Table 1.* Main chemical parameters of the tested soils and the clay fraction content

Soil type	Statistical parameter	pH 1M KCl	CaCO <sub>3</sub>	SOM	P <sub>2</sub> O <sub>5</sub>	K <sub>2</sub> O	Clay fraction (<0.002 mm)
			%	%	mg 100 g <sup>-1</sup>	mg 100 g <sup>-1</sup>	%
Chernozem	Min	4.00	BDLM	2.69	2.14	15.98	23.60
	Max	7.35	2.94	4.75	37.05	38.65	33.20
	Average	5.92	0.60	3.68	8.18	20.81	29.84
	STDEV	1.11	1.05	0.62	7.95	5.45	2.41
	CV	0.64	0.61	1.08	4.59	3.15	4.17
Sand	Min	5.10	BDLM	1.38	2.32	8.94	6.20
	Max	7.65	9.33	4.33	85.89	38.65	46.10
	Average	6.63	1.27	2.75	17.49	17.94	20.12
	STDEV	0.77	2.05	0.65	17.41	7.78	10.13
	CV	0.44	1.18	1.13	10.05	4.49	17.55
Eutric cambisol	Min	3.60	BDLM	2.09	0.55	9.33	14.20
	Max	7.05	1.91	5.06	78.48	38.65	47.60
	Average	4.67	0.03	2.79	7.52	18.86	29.92
	STDEV	0.67	0.22	0.50	12.78	6.16	4.37
	CV	0.39	0.13	0.87	7.38	3.56	29.92

SOM – soil organic matter; BDLM – below the detection limit of the method (0.04%)

The determined content of available phosphorus in all tested soils was in the range from very low to very high, as follows: 2.14–37.05 mg 100 g<sup>-1</sup> in chernozem, 2.32–85.89 mg 100 g<sup>-1</sup> in sands, and 0.55–78.48 mg 100 g<sup>-1</sup> in eutric cambisol. The content of available potassium was in the range of medium to high in chernozem (15.98–38.65 mg 100g<sup>-1</sup>) and sands (8.94–38.65 mg 100 g<sup>-1</sup>), and within the levels of a very low to a high value in eutric cambisol (9.33–38.65 mg 100 g<sup>-1</sup>) (Table 1).

All these obtained data on the main parameters of soil fertility are in accordance with the ones obtained in a previous study (Institute of Soil Science, 1975).

By analyzing the content of total trace elements in soil samples (Figure 2), the content of Pb, Cr, and Cd in all samples of all three types of soil was below the maximum permissible concentrations (*Official Gazette of RS*, 23/1994). Ni content in chernozem and eutric cambisol was also below the MPC, while in the sandy soils the content of this element exceeded the MPC at four localities.

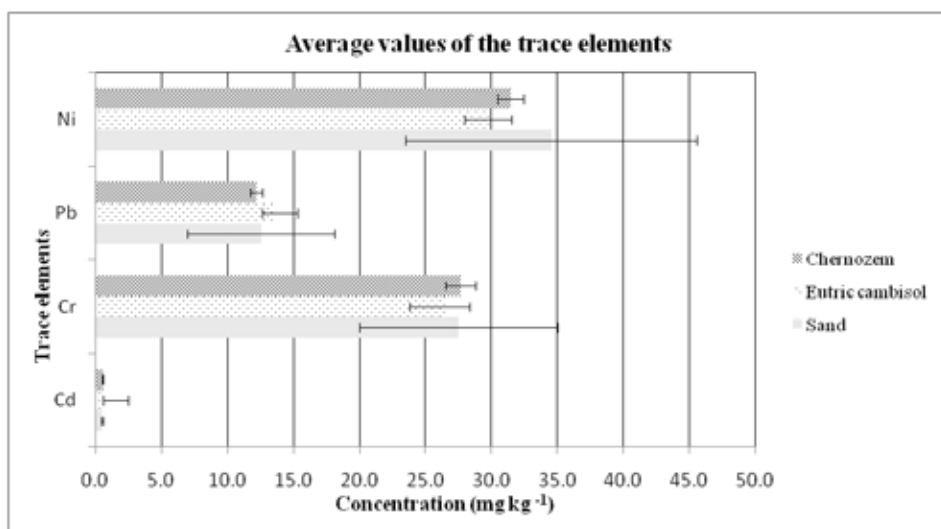


Figure 2. The levels of analyzed total forms of trace elements

For adjusted established MPC (*Official Gazette RS* 23, 1994) it was applied the equation defined by *Official Gazette RS* 88 (2018):

$$(SW, IW)_b = (SW, IW)_{sb} \times \frac{A + (B \times \% \text{ clay}) + (C \times \% \text{ SOM})}{A + (B \times 25) + (C \times 10)}$$

By applying the defined equation below, where  $(SW, IW)_b$  is corrected threshold or remediation value for a particular soil, and  $(SW, IW)_{sb}$  is threshold or remediation value from the table of *Official Gazette of RS* (88/2018), which

determines the corrected MPC value, it was found that in all soil samples of chernozem the Cd content was below the defined limit value (0.8 mg/kg of dry matter); the Cd content in sandy soils was above the limit values in two samples, while in eutric cambisol the content of this element was above the limit values in even 25 samples.

In the equation, A, B, and C represent the constants dependent on the type of trace element.

The Ni content in chernozem and eutric cambisol was below the limit values (35 mg/kg of dry matter), while in sandy soils from five sites the content of this element exceeded the limit value.

By applying the regulative 88/2018 (*Official Gazette of RS*, 88/2018) and the related correction formula, and by replacing the limit values from that regulative with the limit values from regulative 23/1994 (*Official Gazette of RS*, 23/1994) new modified limit values were obtained. By analyzing the content of trace elements from given locations with newly modified values, no values were found that were above the modified limit values.

The number of samples in which the corrected MPC value for Cd was found increased by one in eutric cambisol, and in sandy soils by two. The Ni content above the corrected MPC was higher in sandy soils only at three localities.

The value of the content of organic matter and clay fractions in a ratio of 1:3 affects the mobility of Cd to a negligible extent. The mobility of Cr and Ni was not affected by the content of organic matter in the soil, while the mobility of Pb was equally affected by the content of clay and organic matter (MvV, 2000).

It was also determined the determination coefficient of the relationship between the adjusted MPC value for each element and each soil type and the MPC value defined in Official Gazette of Republic of Serbia (*Official Gazette of RS*, 23/1994) and the content of organic matter and clay fractions, respectively (Tables 2 and 3). The results show a better relationship for clay than SOM. This should be first noted and then discussed.

Table 2. Relationship between the adjusted and unadjusted value of trace elements and organic matter content

Soil type	Average content of trace elements $\pm$ standard deviation (mg kg <sup>-1</sup> ) – R <sup>2</sup> (the correlation coefficient)			
	Ni	Pb	Cr	Cd
Chernozem	31.45 $\pm$ 1.72	12.18 $\pm$ 0.80	27.63 $\pm$ 1.98	0.54 $\pm$ 0.03
	y = 0.0139x + 3.4848 R <sup>2</sup> = 0.0239	y = 0.0788x + 3.815 R <sup>2</sup> = 0.1483	y = 0.0199x + 3.4848 R <sup>2</sup> = 0.0239	y = 0.146x + 5.517 R <sup>2</sup> = 0.4687
Eutric cambisol	29.74 $\pm$ 3.10	13.52 $\pm$ 1.51	27.72 $\pm$ 4.77	0.67 $\pm$ 0.17
	y = 0.0182x + 2.5393 R <sup>2</sup> = 0.2046	y = 0.0497x + 2.9287 R <sup>2</sup> = 0.2881	y = 0.026x + 2.5393 R <sup>2</sup> = 0.2046	y = 0.0719x + 3.8651 R <sup>2</sup> = 0.4443
Sand	34.55 $\pm$ 19.13	12.53 $\pm$ 9.65	27.52 $\pm$ 12.99	0.48 $\pm$ 0.16
	y = 0.0169x + 2.9889 R <sup>2</sup> = 0.5644	y = 0.0404x + 3.33 R <sup>2</sup> = 0.6041	y = 0.0241x + 2.9889 R <sup>2</sup> = 0.5644	y = 0.0512x + 3.9686 R <sup>2</sup> = 0.6715



Table 3. Relationship between the adjusted and unadjusted value of trace elements and the clay fraction content

Soil type	Average content of trace elements $\pm$ standard deviation ( $\text{mg kg}^{-1}$ ) – $R^2$ (the correlation coefficient)			
	Ni	Pb	Cr	Cd
Chernozem	31.45 $\pm$ 1.72	12.18 $\pm$ 0.80	27.63 $\pm$ 1.98	0.54 $\pm$ 0.03
	$y = 0.35x + 25$ $R^2 = 1$	$y = 0.7712x + 31.185$ $R^2 = 0.9434$	$y = 0.5x + 25$ $R^2 = 1$	$y = 0.6834x + 38.449$ $R^2 = 0.6822$
Eutric cambisol	29.74 $\pm$ 3.10	13.52 $\pm$ 1.51	27.72 $\pm$ 4.77	0.67 $\pm$ 0.17
	$y = 0.35x + 25$ $R^2 = 1$	$y = 0.8003x + 32.071$ $R^2 = 0.9906$	$y = 0.5x + 25$ $R^2 = 1$	$y = 0.9058x + 43.405$ $R^2 = 0.9338$
Sand	34.55 $\pm$ 19.13	12.53 $\pm$ 9.65	27.52 $\pm$ 12.99	0.48 $\pm$ 0.16
	$y = 0.35x + 25$ $R^2 = 1$	$y = 0.8096x + 31.67$ $R^2 = 0.9984$	$y = 0.5x + 25$ $R^2 = 1$	$y = 0.9679x + 43.094$ $R^2 = 0.9878$

The obtained results indicate that the C constant, which refers to the organic matter content and is associated with the product used in the calculation of the corresponding element ( $C = 0$  for Ni;  $C = 1$  for Pb;  $C = 0$  for Cr;  $C = 0.021$  for Cd), has a higher dependency in sandy soils, as expected, considering the source of formula which is primarily intended for similar soil types. For the content of Cd in the chernozem soil, it was registered the lowest coefficient of determination for the clay fraction content, which can be explained by the lowest coefficient of variation for the tested parameter which was determined by processing the observed number of samples.

The correlative dependence which refers to the clay fraction content showed a high correlation, which is in correspondence with the assigned B constants for the tested elements ( $B = 1$  for Ni;  $B = 1$  for Pb;  $B = 2$  for Cr;  $B = 0.007$  for Cd).

## CONCLUSION

Using the equation for determination of the adjusted MPC value defined in *Official Gazette of RS* (2018), which in the calculation includes the coefficients of the content of organic matter and clay fractions individually associated with the tested trace element and the limit MPC value, for a determined number of samples with trace elements value above the MPC, defined in *Official Gazette of Republic of Serbia (Official Gazette of RS, 1994)*, it was found that the content of Cr and Pb in tested types of soil still does not exceed the adjusted MPC value. As for Cd and Ni, there was no deviation from the established and modified values of MPC.

Research should be continued and supplemented by data for other types of soils, based on which there would be done a further assessment of the applicability of the existing regulations taken from Dutch sources (MvV, 2000) and incorporated into the *Regulative Official Gazette (88/2018)*.

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ПРОЦЕНА МДК ЕЛЕМЕНАТА У ТРАГОВИМА У  
ПОЉОПРИВРЕДНОМ ЗЕМЉИШТУ ПОМОЋУ  
САДРЖАЈА ОРГАНСКЕ МАТЕРИЈЕ И ГЛИНЕ

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**РЕЗИМЕ:** Циљ овог рада је истраживање доприноса утицаја садржаја органске материје и глине на вредност максимално дозвољених концентрација (МДК) елемената у траговима Pb, Ni, Cr и Cd, које је спроведено на пољопривредном земљишту територије Општине Велико Градиште. Анализирано је 82 узорка на земљишту типа еутрични камбисол, 17 узорака на земљишту типа чернозем и 32 узорка на песковитим земљиштима. У композитним узорцима земљишта, узетих са дубине 0–30 cm, одређени су основни параметри плодности (pH, P<sub>2</sub>O<sub>5</sub>, K<sub>2</sub>O, CaCO<sub>3</sub>, органска материја), садржај фракције глине и садржај укупних форми Pb, Ni, Cr и Cd. Тумачење добијених резултата испитивања спроведено је у односу на МДК испитиваних микроелемената дефинисаних Правилником (*Службени гласник РС*, 88/2018). На основу односа дефинисаних МДК и коригованих вредности које у обрачун узимају и вредности садржаја органске материје и глине, утврђена је корелациона зависност у односу на садржај органске материје, односно садржај фракције глине у испитиваним узорцима. Поред тога, садржај Cr и Pb у испитиваним типовима земљишта и даље не прелази прилагођену вредност МДК. За вредности Cd и Ni, није било одступања од утврђених и модификованих вредности МДК. Закључак је да истраживања треба наставити и допунити подацима и за остале типове земљишта на основу чега би се дала коначна оцена примењивости постојеће регулативе која је преузета из холандских извора а у 2018. години су унета у Правилник (*Службени гласник РС*, 88/2018).

**КЉУЧНЕ РЕЧИ:** земљиште, елементи у траговима, максимално дозвољене концентрације (МДК), органска материја, глина