INFLUENCE OF SOIL PARTICLE SIZE ON CONTENT AND AVAILABILITY OF TRACE ELEMENTS IN SOILS UNDER VINEYARDS

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ABSTRACT

In order to test the effect of soil physical and chemical properties on the content and availability of trace elements (TEs), total of 52 soil samples from 26 representative vineyards were analysed (from depths 0-30 cm and 30-60 cm), which belong to Srem, Subotica and South Banat wine sub regions. Collected samples were analysed for pH, CaCO₃ content, organic matter (OM), total organic carbon (TOC), granulometric fractions, pseudo total content TE_T (soil degradation with HNO₃ and H₂O₂): As, Co, Cr, Cu, Fe, Ni, Pb, Zn and available content TE_{EDTA}: Cu, Fe, Mn, Pb (extraction in EDTA). Pollution and potential pollution of the land with copper was determined due to intensive and long-term application of copper-based fungicides. Based on determined statistically significant correlations, if was found that pH increase affects content reduction and availability of TE_T and TE_{EDTA}, except in case of As_T. Content reduction of As_T, Fe_T, Ni_T and Zn_T was determined, with increased OM content in soil layer 0-30 cm. OM content affects the increase of available Pb_{EDTA}. Contents of As_T, Cr_T, Fe_T, Mn_T, Ni_T, Pb_T, Zn_T increase with the increment of clay and silt content, and simultaneously reduce with the increment of fine and coarse sand content, in both soil depths. Available Fe_{EDTA}, Mn_{EDTA} and Pb_{EDTA}, as well as their total content, are affected by the particle size. Availability of Cu_{EDTA} that is of anthropogenic origin, increases with the content of coarse sand in both soil layers, but reduces with the content of fine sand in topsoil layer.

Keywords: soil, soil particle, trace elements, vineyard

INTRODUCTION

Chemical elements (metals, metalloids, non-metals and other elements) in soil are referred to as trace elements (TEs) because of their occurrence at concentrations less than 100 mg kg⁻¹. Each one plays a role in the soil–plant–animal system, either as nutrients or as toxicants.

When assessing the risks of TE pollution of soil, it is important to know its geochemical (mobility, reactivity) and biological properties (availability, toxicity) (Kabata-Pendias, 2004; Menzies et al., 2007). Solubility of trace metals is often shown as a function of pH affected by the amount and type of organic matter. In addition, other soil factors, such as CEC, carbonates, Fe and Mn hydrous oxides, clay minerals, and fine granulometric fractions are known to play significant roles in the behaviour of trace elements (Kabata Pendias and Pendias, 2001).

Trace elements distribution with particle size is primarily a function of mineral composition of soil parent material and amount of adsorption sites in each size fraction (Qian et al., 1996; Acosta et al., 2011). The soil texture is an important factor in trace element retention or release. In general, coarse-grained soils exhibit a lower tendency for TE sorption than fine-grained soils (Hooda, 2010).

Like other agricultural lands, soils under vineyards are potentially affected by pollution of trace elements. It happens due to the agrochemicals use, especially copper accumulation, because of intensive application of copper-based fungicides. Since viticulture is a branch of agriculture with large investments, it is very important that the land under vineyards be kept in constant good condition. It is achieved by applying adequate cultivation practices, while respecting abiotic and biotic factors that prevail in the production area.

The aim of this paper was to study the effect of physical and chemical properties of soil on content and availability of trace elements (As, Co, Cr, Cu, Ni, Pb, Zn), as well as microelements (Fe and Mn) in soils under vineyards.

MATERIALS AND METHODS

Study area and sample collection

Studies were carried out in wine growing region of Vojvodina and occupied three sub regions: Srem, Subotica, and South Banat. Details on the extent of region areas are shown in Table 1 and Figure 1. The most prevalent types of land are listed using geostatistics based on the pedological map (Živković, 1972), by processing the entire surface of the sub region using ESRI ArcGIS Geostatistical Analyst 10 software.

Sub region	Distribution	Sub region area km ²	Wine growing district(s)	The most common types of soil %				
Srem	slopes of Fruška	1,119.42	Fruška Gora	Chernozems 53%, Eutric				
	Gora			Cambisols 23% Rendzinas 15%				
Subotica	Subotica-Horgoš	390.65	Ridjica, Palić,	Chernozems 60%, Arenosols				
	sandy terrain		Horgoš	24%, Regosols 6%				
South Banat	the western slopes of	1,023.53	Vršač, Bela Crkva,	Chernozems 27%, Vertisols				
	Vršac mountains and		Deliblato sands	22%, Regosols 17%				
	the area of Deliblato							
	sands							

Table 1: General characteristics of the observed site	s
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Collective surfaces of individual wine growing districts are much smaller than sub regions surface. On the other side, areas under vineyards are far smaller than the distribution of each wine growing district, as well as potential for wine growing production. These areas have been increasing in recent years, but for illustrative purposes based on agricultural register from 2012 (Ivanišević et al., 2015), areas under vineyards were as follows: Srem 22.15 km², Subotica 3.12 km² and South Banat sub region 17.04 km².

Total of 52 bulked soil samples were taken from 26 separate plots within a 14 representative vineyards (20 samples from both Srem and Subotica and 12 samples from South Banat sub region). Samples were taken from two depths: topsoil 0-30 cm and 30-60 cm soil layer. The second depth, according to the methodology for vineyards soil sampling, was chosen as the most active zone of vine root systems. One composite sample represented 15-25 subsamples from random points within about 5,000 m² plot area. The samples were taken using a soil drill agrochemical probes and stored in polyethylene bags. The initial quantity of samples was approximately 1.5 kg. Soil samples were air-dried at the room temperature, milled and sieved to <2 mm particle size, in accordance with ISO 11464 (2006).





Laboratory analysis

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 SRPS ISO/IEC 17025 (2006). 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 (as CaCO3 content) was determined according to ISO 10693 (1995) volumetric method. The organic matter (e.g. OM) content was measured by oxidation using the sulfochromic oxidation method by ISO 14235 (1998). Total organic carbon (TOC) was determined by elementary analysis using CHNSO VarioEL III Elementar, after dry combustion in accordance with the ISO 10694:1995.

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 μ m), silt (2-20 μ m), fine sand (20-200 μ m) and coarse sand (200-2,000 μ m).

The samples were analysed for "pseudo-total" contents of trace elements i.e. TET (As, Co, Cr, Cu, Fe, Ni, Pb, Zn) after digesting the soil in concentrated HNO3 and H2O2 (5HNO3: 1H2O2, and 1:12 solid : solution ratio) by stepwise heating up to 180°C using a

Milestone ETHOS1 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 $\pm 10\%$ of the certified values. Available TE concentrations e.g. EDTA-extractable TEEDTA (Cu, Fe, Mn, Pb) 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 TE was determined by ICP-OES (Vista Pro-Axial, Varian).

Statistical analysis

In order to confirm the relationship among content of trace elements and different chemical traits, a Pearson's correlation coefficients were calculated. All statistical analyses were performed using the data analysis software system Dell Statistica, version 13 (Dell Inc, 2016).

RESULTS

Physical and chemical soil properties

The obtained results of basic soil properties were within wide interval. Soil pH value ranged from 4.47 (highly acid) to 8.09 (slightly alkaline) (Table 2). Based on the share of individual samples, soil pH was dominantly slightly alkaline, except in South Banat sub region, where soil pH was dominantly acid.

According to the carbonate content, as $CaCO_3$ content, tested samples belong to a category from non-calcareous to highly calcareous soil (>10%) (Table 2). In South Banat sub region, all soil samples belong to the non-calcareous category. Statistically significant positive correlation was determined (p<0.01) between pH value and CaCO₃ content in both depths.

The content of organic matter (OM) ranged from 0.55% very poor in humus to 2.69% humic soil, according to classification for vineyards soils (Ninkov, 2017a). According to the average value of organic matter content, soils belong to the category poor in humus (1-2%) in both depths. OM content significantly decreased with the soil depth. Total organic carbon (TOC) follows the content of OM. Therefore, statistically significant positive correlations between OM and TOC were determined (p<0.01) in both soil depths (Table 2).

Variable	Min.	Max.	Mean	Std. Dev.	As _T	Co _T	Cr _T	$\mathbf{C}\mathbf{u}_{\mathrm{T}}$	Fe _T	Mn _T	Ni _T	Pb _T	Zn _T	Cu _{edta}	Fe _{edta}	Mn _{edta}	Pb _{edta}
0-30 cm																	
pH KCl	4.53	8.09	6.96	1.30	-0.315	-0.756**	-0.621**	-0.690**	-0.619**	-0.682**	-0.425*	-0.733**	-0.528**	-0.495*	-0.864**	-0.541**	-0.462*
CaCO ₃	0.00	17.82	5.65	5.56	-0.068	-0.527**	-0.477*	-0.554**	-0.440*	-0.506**	-0.379	-0.571**	-0.484*	-0.540**	-0.696**	-0.751**	-0.585**
OM	0.57	2.69	1.65	0.59	0.489*	0.352	0.371	-0.040	0.422*	0.426*	0.434*	0.378	0.447*	-0.119	0.010	0.195	0.460*
TOC	0.32	1.73	0.98	0.44	0.455*	0.105	0.260	-0.278	0.276	0.220	0.370	0.183	0.365	-0.286	-0.188	0.028	0.346
Clay	3.08	39.04	21.10	11.24	0.819**	0.959**	0.933**	0.348	0.950**	0.926**	0.858**	0.933**	0.873**	0.108	0.586**	0.687**	0.690**
Silt	1.48	31.04	18.77	10.14	0.926**	0.805**	0.846**	0.087	0.901**	0.863**	0.906**	0.816**	0.897**	-0.087	0.265	0.569**	0.670**
Fine sand	22.67	74.71	49.33	16.07	-0.603**	-0.887**	-0.826**	-0.559**	-0.819**	-0.846**	-0.778**	-0.861**	-0.762**	-0.398*	-0.641**	-0.692**	-0.650**
Coarse sand	0.63	40.16	10.80	12.19	-0.730**	-0.3841	-0.475*	0.343	-0.545**	-0.456*	-0.518**	-0.404*	-0.547**	0.497**	0.084	-0.194	-0.337
								3	0-60 cm								
pH KCl	4.47	8.17	7.02	1.357	-0.230	-0.740**	-0.664**	-0.663**	-0.658**	-0.652**	-0.463*	-0.741**	-0.513**	-0.415*	-0.885**	-0.738**	-0.731**
CaCO ₃	0.00	24.18	7.69	7.265	0.138	-0.472*	-0.459*	-0.620**	-0.386	-0.415*	-0.361	-0.515*	-0.337	-0.554**	-0.661**	-0.722**	-0.654**
OM	0.55	2.36	1.25	0.449	0.294	0.198	0.276	-0.135	0.246	0.275	0.308	0.216	0.301	-0.249	-0.147	-0.117	0.198
TOC	0.32	1.60	0.74	0.312	0.271	0.020	0.116	-0.351	0.103	0.115	0.199	0.034	0.198	-0.385	-0.295	-0.268	0.051
Clay	3.20	41.32	22.48	11.663	0.730**	0.953**	0.931**	0.353	0.952**	0.931**	0.843**	0.939**	0.865**	0.009	0.635**	0.661**	0.740**
Silt	0.88	31.20	18.35	10.148	0.754**	0.781**	0.795**	0.004	0.853**	0.836**	0.852**	0.763**	0.904**	-0.267	0.156	0.275	0.425*
Fine sand	25.02	74.66	48.90	16.052	-0.536**	-0.931**	-0.875**	-0.406*	-0.896**	-0.889**	-0.827**	-0.894**	-0.811**	-0.100	-0.621**	-0.648**	-0.685**
Coarse sand	0.47	33.76	10.28	10.248	-0.738**	-0.400*	-0.476*	0.229	-0.524**	-0.494*	-0.509**	-0.425*	-0.610**	0.410*	0.096	-0.010	-0.190

Table 2. Correlation coefficients between pseudo total and available trace elements (mg kg⁻¹), pH values in 1 M KCl, content of CaCO₃ (%), organic matter contents OM (%), total organic carbon (TOC) and various size soil fractions (%), for observed samples

clay (<2 μm), silt (2-20 μm), fine sand (20-200 μm), coarse sand (200-2,000 μm)

*p=0.95, significantly correlated

**p=0.99, significantly correlated

According to content of individual soil particle sizes, results varied in wide interval with high value of standard deviation (Table 2). Accordingly, various texture classes of land are also present. Clay loam prevails in Srem and South Banat sub regions, while loamy sand soil texture class is the dominant one in Subotica sub region.

According to the criteria for MAC (Maximum Available Concentration) for agricultural land (Official Gazzette RS 23/1994), no agricultural soil samples exceeded MAC, except Cu (Table 2). In South Banat sub region, two vineyards had Cu_T content above MAC.

The effect of observed factors on trace elements content

Statistically significant negative correlation was determined among pH value and all monitored TEs in both tested soil depths, except for the As_T content. Content of $CaCO_3$ was in statistically significant negative correlation in first soil layer (0-30 cm) for all tested TE_T, except for the content of As and Ni. There was no statistically significant correlation in deeper soil layer (30-60 cm) between CaCO₃ content and pseudototal content of As, Fe, Ni and Zn; while other observed TE_T were in statistically significant negative correlation (Table 2).

OM content was in statistically significant positive correlation (p<0.05) only in topsoil layer (0-30 cm) with contents of As_T , Fe_T , Mn_T , Ni_T , Zn_T , while in deeper soil layer there was no statistically significant correlations. TOC was in statistically significant positive correlation (r=0.455*) only with the content of As_T in topsoil layer (Table 2).

According to determined statistically significant correlations among different particle sizes and content of TE_T , except for Co_T and Cu_T , all other observed TE_T (As_T, Cr_T, Fe_T, Mn_T, Ni_T, Pb_T, Zn_T) were in statistically significant positive correlation with the content of clay and silt (p<0.01). Simultaneously, they were in statistically significant negative correlation with the content of fine and coarse sand, in both soil depths. An exception to this rule is only for Co_T in case it is not in statistically significant negative correlation with coarse sand in 0-30 cm layer. Content of Cu_T is in statistically significant negative correlation only with the content of fine sand in both observed depths (Table 2).

The effect of observed factors on trace elements availability

Out of four observed metals Cu, Fe, Mn, and Pb according to available content e.g. TE_{EDTA} . pH value and content of CaCO₃ were in statistically significant negative correlation with all four metals in both soil depths. Organic matter content was in statistically significant positive correlation only with Pb_{EDTA} (r=0.460*) in topsoil layer. There was no statistically significant correlation between TOC and available content TE_{EDTA} (Table 2). According to determined statistically significant correlations among particle sizes and available content of metals, Fe_{EDTA} , Mn_{EDTA} and Pb_{EDTA} are in positive correlation with clay content and negative correlation with fine sand content in both depths (p<0.01). Silt content is in positive correlation with Mn_{EDTA} and Pb_{EDTA} in the first soil layer, while it positively correlated in the second layer only with Pb_{EDTA} content. Content of coarse sand was not statistically correlated with these three metals. Content of Cu_{EDTA} was in negative correlation with fine sand in topsoil layer, while it was in positive correlation with coarse sand in both soil depths (Table 2).

DISCUSSION

Wide variability of pH results, content of CaCO₃, organic matter and physical properties of soil is expected, since observed samples came from different sites, various pedological base (Figure 1), and were subjected to different cultivation practices. These soil traits are primarily the consequence of pedogenic factors. Dominant acid soil reactions in South Banat sub region is the effect of natural characteristic of the Vršac wine growing district parent substrate (Živković et al., 1972). The dominant loamy sand soil texture class was determined as natural characteristic of arenosol soil, which is distributed in Subotica sub region. Beside natural pedogenic factors, application of cultivation practices significantly affects the content of organic matter in the soil. Soil pH value is always correlated with carbonate content in the soil, such as organic matter and total organic carbon content, which was confirmed by this study.

Soils where grapevines are grown are especially threatened by copper accumulation due to long-term and intensive application of copper-based pesticides. In agreement with previous studies, this problem was also present in soils of Serbia (Ninkov et al., 2010; 2012; 2014; 2017a). In this study, average copper content in soil was $46.2\pm39.2 \text{ mg kg}^{-1}$, which is higher than background concentration for Vojvodina soils of 17.1 mg kg⁻¹ (Kastori, 1993). Except for two sites where content of Cu_T was determined to be above MAC of 100 mg kg⁻¹, four more sites had content of Cu_T over 60 mg kg⁻¹, which represents critical concentration, according to literature (Schramel et al., 2000). Available Cu_{EDTA} content was also high at these sites, while it exceeds the value of 50 mg kg⁻¹ at three sites, which is considered potentially phytotoxic (Wasterhoff-u. cit. Ubavić i Bogdanović, 1995). The content of other TE_T was at the level of common values for soils of Vojvodina (Kastori, 1993).

Soil pH is an important parameter that reflects the course of other physical and chemical reactions in the soil. Effects of pH on the trace elements mobility are complex and highly

element specific. The solubility of trace elements that occur of free cations generally increases with decreasing pH (Hooda, 2010). This study confirms that rule for all observed TE_T and TE_{EDTA} , except for the content of As_T . According to the same author, elements that exists as anions such as As, are more mobile in alkaline conditions. According to previous study on large area in Vojvodina Province, As_T was found to be in significant negative correlation with pH value and CaCO₃ (Ninkov et al., 2017b). Since pH and CaCO₃ are in a mutual positive correlation, content of specific TE_T decreases with increment of CaCO₃ content. Free CaCO₃ in soils controls the solubility of trace elements via its influence on pH and formation of metal carbonates (Hooda, 2010). Study on the role of OM in metal mobility presents apparently contradictory results. Soil OM has a high binding capacity for cationic and organic contaminants, which might lead to immobilization of metal ions. In other studies, however, the degradation of OM released low molecular weight of organic acids that bound metals and increased metal solubility. Positive correlations between OM content and specific TE_T were determined only in topsoil layer 0-30 cm, where OM was higher due to favourable conditions for humification process.

The soil texture is an important factor in trace element retention or release. In general, coarsegrained soils exhibit a lower tendency for TE sorption than fine-grained soils (Hooda, 2010). In present study, stated general rule for As_T , Cr_T , Fe_T , Mn_T , Ni_T , Pb_T , Zn_T was confirmed with high significance of determined correlations p<0.01. The content of mentioned metals increase with the increment of smaller soil particles content (clay and silt) and simultaneously decrease with the increment of large elements ratio (fine and coarse sand). The content of Co_T also behaves by stated rule, with an exception that coarse sand content in topsoil layer 0-30 cm does not have confirmed influence. In studies of Acosta et al. (2011), Pb, Cd, Cu, and Zn exhibit preferential partitioning into soil fine particles.

An exception from the common rule of the influence of soil particle size is the content of Cu_T , which decreases only with an increment of fine sand ratio, while ratio of other particles does not affect its content. This is probably the result of anthropogenic influence (pollution of soil with copper), i.e. high variability of obtained results. The effect of soil particle size on the content and availability of copper in vineyard soil was confirmed in previous studies (Ninkov et al., 2013).

Increase of pH value toward alkaline environment decreases the availability of all four observed metals Cu, Fe, Mn and Pb, according to their available content e.g. TE_{EDTA} . Content of organic matter affects the increase of available Pb_{EDTA} . Expected decrease of Pb availability and other TE was not determined with the increase of OM content.

According to the effect of different particle sizes on TE availability, it was not determined that finer particles decrease the availability of TE, i.e. that the share of larger particles increases them. Available Fe_{EDTA}, Mn_{EDTA} and Pb_{EDTA} are under the same influence of particle sizes as well as their total content. This is the result of usual TE values in tested soils, which is the reason their competition for binding to OM and smaller soil particles did not occur. In studies of Qian et al. (1996), greater extractability existed in the fine sand fraction (50-125 µm) for Ni, Cu and Pb, and in the medium sand fraction (125-500 µm) for Co. For Co and Pb, high extractable contents were found in the clay fraction ($<2 \mu m$). According to Kabata Pendias (2004), trace elements whose origin in the soil is natural (lithogenic and pedocenic origin) are found bound to minerals, associated with clay minerals, soil organic matter and oxides, and their bioavailability is very slight to slight. Trace elements of anthropogenic origin in the soil are found associated with SOM and particle surface and their bioavailability is moderate. Availability of Cu_{EDTA}, which is of anthropogenic origin, increases with the content of coarse sand in both soil layers, but decreases with the content of fine sand in topsoil layer. In addition, studies of Ajmone-Marsan et al. (2008) stated that accumulation of TEs in the finer fractions is higher where the overall contamination is lower.

CONCLUSIONS

The pollution and potential pollution of the soil with copper was determined due to intensive and long-term application of copper-based fungicides. Increase of pH value affects the decrease of content and availability of TE_T and TE_{EDTA} , except in case of As_T . The decrease in As_T , Fe_T , Ni_T and Zn_T content was determined with an increase of OM content in topsoil layer (0-30 cm). The content of OM affects the increase of available Pb_{EDTA} content. The content of As_T , Cr_T , Fe_T , Mn_T , Ni_T , Pb_T , Zn_T increases with the increment of clay and silt content and simultaneously decreases with the increment of fine and coarse sand content, in both soil depths. Available Fe_{EDTA} , Mn_{EDTA} and Pb_{EDTA} were under the same influence of particle sizes as well as their total content. Availability of Cu_{EDTA} , which is of anthropogenic origin, increases with the content of coarse sand in both soil layers, but decreases with the content of fine sand in topsoil layer.

The obtained results confirmed that when assessing the risk of soil contamination with TE, it is necessary to analyse the mechanical composition of soil as an important factor in its accessibility.

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IZVOD

UTICAJ MEHANIČKOG SASTAVA NA SADRŽAJ I PRISTUPAČNOST ELEMENATA U TRAGOVIMA U ZEMLJIŠTIMA POD VINOGRADIMA

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U cilju ispitivanja uticaja fizičko hemijskih osobina zemljišta na sadržaj i pristupačnost elemenata u tragovima (TE), analizirano je ukupno 52 uzoraka zemljišta iz 26 reprezentativna vinograda (sa dubine 0-30 i 30-60 cm) koji pripadaju Sremskom, Subotičkom i Južno banatskom vinogradarskom rejonu. U prikupljenim uzorcima određen je pH, sadržaj CaCO₃, organske materije (OM), ukupnog organskog ugljenika (TOC), mehanički sastav zemljišta, pseudo ukupni sadržaj TE_T (razaranje zemljišta sa HNO₃ i H₂O₂): As, Co, Cr, Cu, Fe, Ni, Pb, Zn i pristupačan sadržaj TE_{EDTA}: Cu, Fe, Mn, Pb (ekstrakcija u EDTA). Utvrđeno je zagađenje i potencijalno zagađenje zemljišta bakrom usled intenzivne i dugotrajne primene fungicida na bazi bakra. Na osnovu statistički značajnih utvrđenih korelacija utvrđeno je da povećanje pH vrednosti utiče na smanjenje sadržaja i pristupačnosti TE_T i TE_{EDTA}, osim u slučaju As_T. Utvrđeno je smanjenje sadržaja As_T, Fe_T, Ni_T i Zn_T sa povećanjem sadržaja OM u sloju zemljišta 0-30 cm. Sadržaj OM utiče na povećanje sadržaj pristupačnog Pb_{EDTA}. Sadržaj As_T, Cr_T, Fe_T, Mn_T, Ni_T, Pb_T, Zn_T se povećava sa povećanjem sadržaja gline i praha, a istovremeno smanjuje sa povećanje sadržaja sitnog i krupnog peska, na obe dubine zemljišta. Pristupačni Fe_{EDTA}, Mn_{EDTA} and Pb_{EDTA} je pod istim uticajem veličina čestica kao i njihov ukupni sadržaj. Pristupačnost Cu_{EDTA} koji je antropogenog porekla, se povećava sa sadržajem krupnog peska u oba sloja zemljišta, ali se smanjuje sa sadržajem sitnog peska u površinskom sloju zemljišta.

Ključne reči: zemljište, mehanički sastav, elementi u tragovima, vinograd

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