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SOIL ELEMENT ASSESSMENT IN DOLNENI'S TOBACCO CULTIVATION AREAS: A STUDY OF ENVIRONMENTAL AND AGRICULTURAL DYNAMICS

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ABSTRACT

Soil quality is a critical factor for achieving high yields and optimal quality in tobacco cultivation. In this study, the soil composition in the Dolneni region of North Macedonia, one of the largest producers of oriental tobacco, is analyzed. Soil samples were collected in 2021 and 2022 and analyzed using inductively coupled plasma – mass spectrometry (ICP-MS) to determine the concentrations of macro and microelements (K, Mg, Fe, and Na) and potentially toxic elements (As, Cd, Cr, Cu, Ni, Pb, Zn). The analysis revealed that the macro and microelement composition of the soil was closely related to the geological and pedological characteristics of the region, while potentially toxic elements were present in concentrations below the established reference values for heavy metals in soils. This study provides valuable insights into the soil quality in the Dolneni region and assesses the potential environmental impact of tobacco cultivation in the area.

Keywords: soil, tobacco fields, macroelements, microelements, potentially toxic elements, ICP-MS

ПРОЦЕНКА НА ПОЧВЕНИТЕ ЕЛЕМЕНТИ ВО ОБЛАСТИТЕ ЗА ОДГЛЕДУВАЊЕ ТУТУН ВО ДОЛНЕНИ: СТУДИЈА ЗА ДИНАМИКАТА НА ЖИВОТНАТА СРЕДИНА И ЗЕМЈОДЕЛСТВОТО

Квалитетот на почвата е критичен фактор за постигнување високи приноси и оптимален квалитет во одгледувањето тутун. Во оваа студија се анализира составот на почвата во регионот Долнени во Северна Македонија, еден од најголемите производители на ориентален тутун. Примероците од почвата беа собрани во 2021 и 2022 година и беа анализирани со помош на индуктивно спрегната плазма-масена спектрометрија (ICP-MS) со цел да се одредат концентрациите на макро и микроелементи (Fe, K, Mg, Na) и потенцијално токсични елементи (As, Cd, Cr, Cu, Ni, Pb, Zn). Анализата откри дека составот на макро и микроелементи во почвата е тесно поврзан со геолошките и педолошките карактеристики на регионот, додека потенцијално токсичните елементи се присутни во концентрации под утврдените референтни вредности за тешки метали во почвите. Оваа студија дава вредни сознанија за квалитетот на почвата во регионот Долнени и дава проценка за потенцијалното влијание од одгледувањето на тутун врз животната средина во оваа област.

Клучни зборови: почва, тутунски полиња, макроелементи, микроелементи, потенцијално токсични елементи, ICP-MS

INTRODUCTION

Soil is a vital resource for agriculture, and its preservation is crucial to maintaining its fertility and ensuring its sustainability for future generations. It is a complex matrix composed of both inorganic elements—such as varying proportions of sand, silt, and clay—and organic matter, including humic substances, lipids, carbohydrates, lignin, flavonoids, pigments, resins, and fulvic acids (Pinto et al., 2011). The quality of soil directly influences agricultural productivity. For tobacco farmers, cultivating healthy crops and maximizing both yield and quality depends on maintaining high soil quality. Only fertile, well-balanced soils can produce high-quality agricultural outputs. Protecting agricultural crops like tobacco requires diverse strategies aimed at minimizing contamination risks. Among these strategies, chemical interventions are foundational to ensuring tobacco plants are shielded from pollutants commonly found in agricultural soils. However, the long-term application of artificial fertilizers and protective agents can disturb the balance of soil's physical and chemical properties.

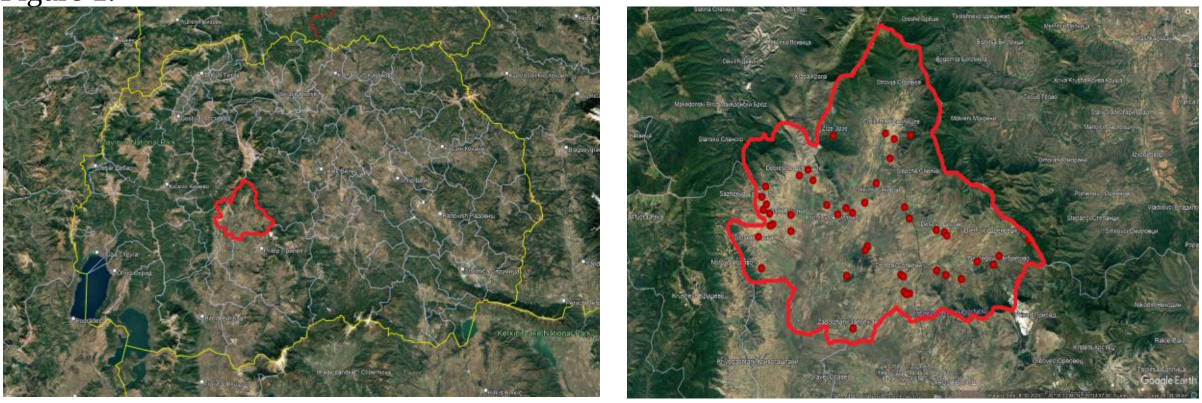
Arable soils often contain a complex mix of metallic, organic, inorganic, and organometallic compounds, serving as a repository for accumulating pollutants, while their degradation occurs slowly. The increasing use of metal-containing fertilizers and agricultural practices involving trace elements like Cu, Zn, Fe, Mn, and B have become standard for maintaining crop yields. Many inorganic fertilizers contain these trace elements, and phosphate fertilizers, which can introduce Cd and Se from the raw materials used in their production, pose an additional source of potential contamination (Singh, 1994; Prasad, 2008). Furthermore, pesticides, fungicides, and herbicides often include metals such as Cu, Zn, Mn, Fe, and even As. These, along with trace elements like copper, selenium, cadmium, and lead added to animal feed, can further accumulate in soil through fertilization (Sheppard et al., 2009). The list of elements that may pose a pollution risk in agriculture includes Ag, As, Ba, Be, Cd, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, Se, Zn, B, Sn, Co, and V (Tóth et al., 2007). Among these, copper, zinc, lead, and cadmium are the most widespread in agricultural soils (Alloway, 1999; He et al., 2005). Soil health is fundamental to the integrity of natural ecosystems. A balanced composition of essential nutrients and trace elements is vital for sustainable agriculture, which depends on maintaining healthy soils (He et al., 2005; Prasad, 2008; Kabata-Pendias,2011).

In this study, inductively coupled plasma mass spectrometry (ICP-MS) was employed to analyze soil samples from the Prilep region. The elemental composition identified includes Ag, Al, As, Au, Ba, Br, Cd, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Hf, Hg, Ho, I, In, Ir, K, La, Lu, Mg, Mn, Mo, Na, Nb, Nd, Ni, Os, Pb, Pd, Pr, Pt, Rb, Re, Rh, Sb, Sc, Se, Sm, Sn, Sr, Ta, Tb, Te, Ti, Tl, Tm, V, W, Y, Yb, Zn, and Zr. This work focuses on key macro and microelements (Fe, K, Mg, Na) and potentially toxic elements (As, Cd, Cr, Cu, Ni, Pb, Zn) to assess their impact on agricultural sustainability and soil health.

MATERIALS AND METHODS

The Dolneni region in North Macedonia is widely recognized for its significant tobacco cultivation. In 2021 and 2022, soil samples were collected from pedological profiles at a consistent depth of 0-30 cm at 40 different locations where oriental tobacco is grown, following the last tobacco harvest. Sampling sites within the Dolneni region are shown in

Figure 1.



a **b**

Figure 1. The location of the Dolneni region on the map of North Macedonia (a) and the location of the soil sampling sites within the region (b)

The collected soil samples were processed according to the ISO 11464:2006 standard. They were air-dried, ground, and passed through a 2 mm sieve. For analysis, the samples underwent digestion using the aqua regia extraction method (ISO 11466), employing a 3:1 mixture of HCl and HNO3. Up to 0.5 g of each sample was digested by heating for 15 minutes with a laboratory microwave system (CEM, USA). ICP-MS analyses were conducted at the Research Institute for Analytical Instrumentation, University of Cluj, Romania, using a SCIEX Perkin Elmer Elan DRC II mass spectrometer (Canada), equipped with an inductively coupled plasma source, a quadrupole, and a single detector. Soil fertility was assessed based on the measured concentrations of organic matter (OM), total nitrogen, available phosphorus and potassium, carbonates and pH (Pelivanoska, 2011).

RESULTS AND DISCUSSION

The basic soil properties of the Dolneni region, which reflect the general soil quality, are presented in Table 1. The average humus content in the cultivated soil samples ranges from low to moderate, indicating relatively modest levels of organic matter. As reported by Filipovski (1990), lower humus levels in soil can be beneficial for cultivating premium oriental aromatic tobacco varieties.The total nitrogen content in the analyzed soils varied between 0.03% and 0.14%, which corresponds to low to moderate nitrogen availability. The mean soil pH was approximately 6.25, indicating that most samples were slightly acidic to neutral, a pH range generally considered suitable for tobacco production.

Table. 1. Basic soil properties and descriptive statistics of the analyzed macro- and microelements

Element	Mean	Median	SD	Minimum	Maximum	CV
Humus, %	1.65	1.65	0.50	0.58	2.92	30
Azot, %	0.08	0.08	0.02	0.03	0.14	30
pH (H2O)	6.25	6.31	0.55	5.05	7.83	9
pH (KCl)	5.07	5.03	0.62	3.93	6.95	12
Total CaCO3, %	0.45	0.00	2.82	0.00	17.81	632
av. P_2O_5, mg/100 g	10.57	6.91	12.97	1.29	78.51	123
av. K_2O, mg/100 g	19.35	18.77	5.94	7.87	34.29	31
Clay, %	36.4	34.1	10.7	20.0	57.4	29
P_2O_5, %	0.293	0.272	0.113	0.160	0.672	39

Element	Mean	Median	SD	Minimum	Maximum	CV
Mg %	0.124	0.107	0.082	0.022	0.401	66
Fe %	1.223	1.137	0.566	0.446	2.332	46
Na %	0.007	0.004	0.006	0.001	0.031	89

av. - available; SD - standard deviation; CV - coefficient of variation

All soil samples from the Dolneni region were non-calcareous and exhibited a wide range of available macronutrient levels. The phosphorus content varied from 1.29 to 78.51 mg P_2O_5/100 g soil, while potassium ranged from 7.87 to 34.29 mg K_2O/100 g. The availability of nitrogen, phosphorus, and potassium in the topsoil is influenced by land use practices and is typically monitored and adjusted annually. This variability is reflected in the high coefficient of variation observed for these macronutrients (Table 1). The soil texture was predominantly medium loamy, with clay content ranging from 20.0% to 57.4%. The analyzed soil parameters closely align with those previously reported by Jordanoska et al. (2014), confirming the consistency of soil characteristics commonly associated with tobacco-growing soils in the Pelagonian region.

The descriptive statistical data for total K_2O, P_2O_5, Mg, Fe, and Na in soil samples from the Dolneni region are summarized in Table 1. The total potassium content, expressed as K_2O, was found to be 2.031%, which is notably higher than the 0.3% average reported by Jordanoska Shishkoska (2014) for tobacco-cultivated soils in the Pelagonian region. This value is approximately equivalent to the elemental potassium (K) content of 2.3% reported in the Geochemical Atlas of Macedonia (Stafilov & Šajn, 2016), which is based on complete digestion methods. Potassium levels in soil are strongly influenced by parent material and soil texture, and typically range between 0.5% and 3% (Lalitha & Dhakshinamoorthy, 2013; Firmano et al., 2020). Phosphorus, another essential macronutrient, also displayed considerable variation across samples, reflecting differences in soil genesis and fertility management practices. Notably, the addition of phosphate fertilizers—commonly used to maintain adequate phosphorus levels—can introduce trace amounts of heavy metals, depending on the raw material source and production process (Alkorta et al., 2004). The phosphorus content, expressed as P_2O_5, was found to be 0.293%, which is significantly higher than the 0.037% reported by Jordanoska Shishkoska (2014), and also exceeds the average value of 0.055% for the Pelagonian region, as indicated in the Geochemical Atlas of Macedonia (Stafilov & Šajn, 2016). The iron (Fe) content in the analyzed soil samples from the Dolneni region (Table 1) averaged 1.223%, which is substantially lower than the mean Fe concentration of 3.1% reported for the Pelagonian region and 3.6% for soils across the Republic of North Macedonia, as indicated in the Geochemical Atlas of Macedonia (Stafilov & Šajn, 2016). Furthermore, this value is also below the European average of 2.6% for agricultural topsoil, based on total digestion methods (Salminen et al., 2005; Soriano-Disla et al., 2013). The average sodium (Na) content in soils from tobacco-growing fields in the Dolneni region was 0.007%, which is higher than the mean value of 0.004% (41 mg/kg) reported by Jordanoska Shishkoska (2014) for soils in the broader Pelagonian region, but substantially lower than the median value of 1.4% reported in the Geochemical Atlas of Macedonia (Stafilov & Šajn, 2016). The mean magnesium (Mg) content of 0.124%, as presented in Table 1, is significantly lower than the value of 0.3% reported by Jordanoska Shishkoska (2014) for soils in the Pelagonian region.

The mean, minimum, and maximum levels of As, Cd, Cr, Cu, Ni, Pb, and Zn in soils from the tobacco fields of Dolneni are given in Table 2. Arsenic contamination in agricultural soils represents a significant environmental issue worldwide, primarily because of its toxicity and ability to accumulate in the food chain. Elevated soil arsenic levels can result from both geogenic sources and anthropogenic influences. Uncontaminated soils usually contain arsenic concentrations around 5 mg/kg (Gonga et. al., 2020; Rahmanet et al., 2023). The mean arsenic concentration in agricultural soils under tobacco cultivation in the Dolneni region was

4.49 mg/kg (Table 2), which is notably lower than the average levels reported for uncultivated soils in the Pelagonian region (5.6 mg/kg), as well as the national mean for soils across North Macedonia (9.2 mg/kg) (Stafilov & Šajn, 2016).

Table 2. As,Cd,Cr, Cu, Ni, Pb and Zn content in tobacco-growing soils in the Prilep region (in mg/kg)

Element	Mean	Median	SD	Minimum	Maximum	CV
As	4.490	3.729	2.983	1.386	17.375	80.003
Cd	0.127	0.124	0.009	0.120	0.139	7.053
Cr	19.519	17.699	10.655	6.272	45.472	54.589
Cu	9.919	9.358	4.017	3.527	19.133	40.494
Ni	13.164	10.579	6.642	4.115	27.017	50.454
Pb	12.641	12.209	3.942	5.599	22.103	31.183
Zn	20.430	19.380	8.225	7.682	40.804	40.258

The average cadmium (Cd) content of 0.127 mg/kg in agricultural soils from tobacco fields in the Dolneni region is comparable to the levels reported for non-arable soils in the Pelagonia region and across North Macedonia, which average 0.17 mg/kg (Stafilov & Šajn, 2016). The chromium content in the soil of tobacco fields in the Dolneni region (19.52 mg/kg) is notably lower than the content for the same element in uncultivated soil of the Pelagonia region 88 mg/kg (Stafilov & Šajn, 2016). The values for As, Cd and Cr are below the target value for chromium in soils established by the Dutch standards (The New Dutch List), indicating that there is no potential contamination. The copper content in agricultural soils typically between 1 and 50 mg/kg, depending on factors such as the parent material, the organic matter content, and the anthropogenic input (Hodges, 1995). As shown in Table 2, the average copper content in agricultural soil from Dolneni 9.92 mg/kg is significantly lower than the 21 mg/kg mean reported for non-cultivable soils in the Pelagonia region, as outlined in the Geochemical Atlas of Macedonia (Stafilov & Šajn, 2016). The atlas also reports average concentrations of nickel (30 mg/kg), lead (32 mg/kg), and zinc (70 mg/kg) in non-cultivable soil from Pelagonia, all of which are substantially higher than the corresponding values in tobacco field soils from the Dolneni region. The mean values of all elements presented in Table 2 are comparable with the average concentrations reported in studies on agricultural soils throughout Europe (Salminen et al., 2005; Soriano-Disla et al., 2013).

CONCLUSIONS

In recent years, agricultural intensification in North Macedonia has led to a greater reliance on fertilizers and pesticides, contributing to elevated concentrations of trace elements and pesticide residues in the soil. This highlights the importance of closely monitoring pollution sources in agricultural soils. The aim of this study was to assess the levels of specific micro and macroelements (Fe, K, Mg, and Na) and potentially toxic elements (As, Cd, Cr, Cu, Ni, Pb, and Zn) in soils used for tobacco cultivation in the Dolneni region, the largest tobacco-growing area. The analysis indicates that macroelement levels are influenced by the region’s geological and pedological characteristics, making these soils suitable for tobacco cultivation from an agrochemical standpoint. Additionally, the concentrations of potentially toxic elements were below the target values for heavy metals. Despite intensive tobacco farming, heavy metal concentrations remain typical of agricultural soils with minimal anthropogenic pollution. Thus, the soils in the Dolneni region, where oriental tobacco is cultivated, are considered environmentally safe concerning the macroelements and potentially toxic elements analyzed.

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