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CHEMICAL CHARACTERIZATION OF TOBACCO SOILS IN THE PRILEP REGION: ENVIRONMENTAL AND AGRICULTURAL PERSPECTIVES

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Abstract

The quality of the soil plays a fundamental role in agricultural productivity, particularly in tobacco cultivation, where both high yields and superior leaf quality are essential. The elemental composition of the soil, in particular the balance between essential nutrients and the presence of potentially toxic elements, plays a crucial role in shaping soil quality and influencing overall plant health and development. In this study, the soil quality in the Prilep region in North Macedonia, the main cultivation area for oriental tobacco, is investigated. During the 2021 and 2022 growing seasons, soil samples were collected from selected tobacco fields and analyzed using ICP-MS to determine the concentrations of selected macro and microelements (K, Mg, Fe and Na) as well as potentially toxic elements (As, Cd, Cr, Cu, Ni, Pb and Zn). In addition to elemental analysis, several key agrochemical properties were also assessed: organic matter content (ranging from low to moderate), total nitrogen content (0.03–0.14%), soil pH (mean 6.55, indicating slightly acidic to neutral conditions), the availability of essential nutrients (phosphorus and potassium), the physical structure of the soils (classified as medium loam), and clay content (20.6% to 58.7%). The content of macro- and microelements were closely related to the geological and pedological characteristics of the region. The concentration of potentially toxic elements remained below the internationally accepted thresholds for heavy metals in agricultural soils, indicating a low risk of contamination and confirming the suitability of these soils for sustainable tobacco cultivation.

Key words: *soil, tobacco fields, macroelements, microelements, potentially toxic elements, ICP-MS.*

INTRODUCTION

Agricultural soil is a vital natural resource that underpins plant growth and sustains ecological balance, making it indispensable for current and future agricultural productivity. As a dynamic and heterogeneous matrix, soil comprises varying proportions of inorganic particles — sand, silt, and clay — which determine its texture, structure, and water-holding capacity. In addition to these mineral components, soil contains a variety of organic substances, including humic substances (typically 10–15%), lipids, carbohydrates, lignin, flavonoids, pigments, resins, and fulvic acids. These organic constituents enhance nutrient availability, stimulate microbial activity, and contribute to overall soil health (Pinto et al., 2011). The complex interplay between organic and inorganic matter makes soil quality a cornerstone of sustainable agriculture, as it has a direct impact on soil fertility, crop productivity, and the long-term resilience of farming systems. Furthermore, soil is a fundamental component of the natural ecosystem, the health of which is a reliable indicator of the well-being of the environment. Achieving sustainability in agriculture depends on maintaining soils with a balanced contents of trace elements and essential nutrients (He et al., 2005; Prasad, 2008; Kabata-Pendias, 2011).

Trace element contamination and accumulation in agricultural production systems is a potential threat to food quality, crop growth and has a direct impact on environmental health (McLaughlin at al., 2000; Micó et al., 2006; Peris et al., 2007). Since agricultural soils act as efficient sinks, they accumulate trace elements and pollutants quickly, while their removal occurs slower. Although metals are naturally occurring (Fadigas et al., 2010; Kabata-Pendias, 2011), agricultural soils are affected by anthropogenic influences, especially the application of sewage sludge, manure, pesticides, inorganic fertilizers and wastewater (Drury et al., 2009; Sheppard et al., 2009). The use of phosphate fertilizers is basic factor in pollution, as they contain a high concentration of heavy metals (Golia et al. 2009; Kabata & Pendias, 2011). Phosphate fertilizers, which may contain trace elements such as Cd and Se derived from the rock phosphates used in their manufacture, are a potential source of these elements (Singh, 1994; Prasad, 2008). Numerous of factors contribute to the mobility and availability of these elements, such as the bioecological characteristics of the plant species, the concentration and chemical forms of occurrence of the elements in the soil and, of course, ecopedological conditions (Alloway, 1999; Kabata-Pendias, 2011).

Tobacco growers strive for the highest possible yield and the best possible quality of their production. To achieve these goals, high-quality soil is essential, as it is the basis for optimal plant performance. The protection of tobacco plants involves a variety of measures, with chemical treatments being among the most reliable to ensure successful production. The Republic of North Macedonia is known for its high-quality oriental tobacco and produces 3% of the world's total oriental tobacco (Kabranova & Arsov, 2009). In order to achieve consistent yields, the use of metal-containing substances has increased significantly, with elements such as Cu, Zn, Fe, Mn commonly used in agricultural practices. These elements are frequently incorporated into various mineral fertilizers. in addition, many pesticides, fungicides, and herbicides contain Cu, Zn, Mn, Fe and even As. The group of elements that are considered potential pollutants 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 areas (Alloway, 1999; He et al., 2005).

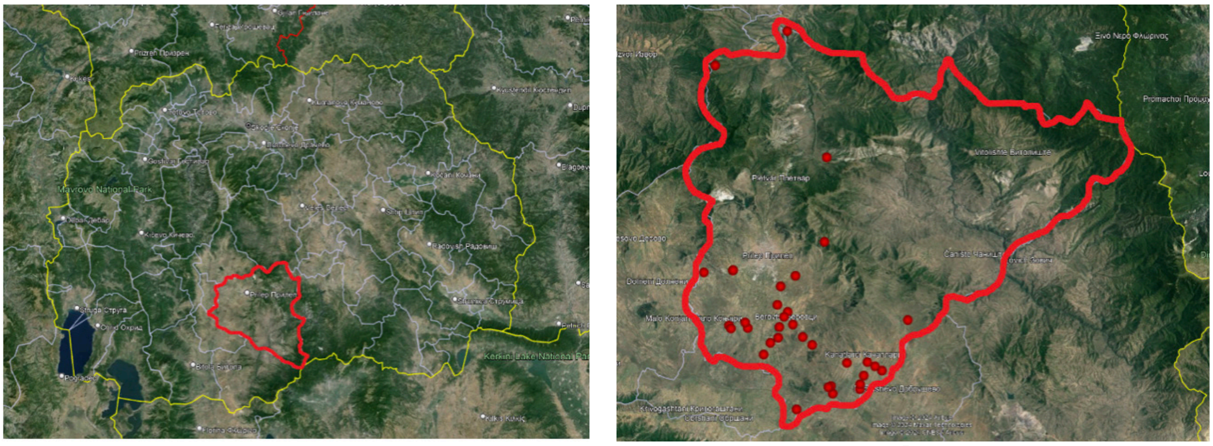
Deficiency and toxicity of trace elements in agricultural soils are closely related to various soil properties, such as organic matter content, type and amount of clay, pH, and cation exchange capacity (CEC), all of which are determined by the parent material of the soil (Fadigas et al., 2010; Kabata-Pendias, 2011). These properties influence the availability, mobilization, and sorption of trace elements and determine their concentration in the soil and their potential impact on plant growth (Chen et al., 1999; Golia, 2001; Kabata-Pendias, 2011). Understanding these interactions is essential for managing soil fertility and addressing both trace element deficiency and toxicity in agricultural systems, as they directly affect plant growth and productivity.

The main aim of our research was to monitor the condition of soils in tobacco cultivation, focusing in particular on the presence and distribution of important macro and microelements, including some potentially toxic element content, to assess their potential impact on plant health and productivity.

MATERIAL AND METHODS

Soil samples from arable land in the Prilep region were collected in 2021 and 2022. The samples were collected from pedological profiles at a standardized depth of 0-30 cm, from 31 locations in the following municipalities: Alinci, Kanatlarci, Topolčani, Berovci, Erekovci, Malo Konjari, Golemo Konjari, Mazučiste, Galičani, Kadino Selo, Varoš). The sampling locations are shown in Figure 1.

The samples were prepared according to the ISO 11464:2006 Soil quality — Pretreatment of samples for physico-chemical analysis. Soil samples were digested using the aqua regia extraction method using HCl and HNO_3 in a 3:1 ratio (U.S. EPA, 2007). A representative sample of up to 0.5 g was digested in a laboratory microwave system (CEM, USA). Two groups of elements were analyzed: macro and microelements (K, P, Mg, Fe, and Na), and potentially toxic elements (As, Cd, Cr, Cu, Ni, Pb, and Zn). The ICP-MS analyses were performed at the Research Institute for Analytical Instrumentation, University of Cluj, Romania. The instrumentation included a SCIEX Perkin Elmer Elan DRC II mass spectrometer (Canada) equipped with an inductively coupled plasma source, a quadrupole and a single detector. The cadmium content was below the detection limit of 1 ppb and is therefore not listed in the following tables. Soil fertility was assessed based on the measured concentrations of organic matter (OM), total nitrogen, available phosphorus and potassium, carbonatesand, clay, and pH (Pelivanoska, 2011).



a b

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

RESULTS AND DISCUSSION

The basic soil properties from the Prilep region, which give an indication of the general soil quality, are presented in Table 1. The average organic matter content in the cultivated soil samples ranges from low to moderate. According to Filipovski (1990), soils with low humus content provide favorable conditions for the cultivation of high-quality oriental aromatic tobacco. The total nitrogen content in the analyzed soils varied between 0.03% and 0.14%. The mean soil pH was 6.55, and most samples were slightly acidic to neutral. All soil samples were non-calcareous and had a wide range of available macronutrients, with phosphorus content ranging from 2.38 to 149.9 mg P_2O_5/100 g and potassium content from 7.69 to 43.11 mg K_2O/100 g. The plant available nitrogen, potassium and phosphorus content of the topsoil varies according to land use and is monitored and corrected annually, which explains the high coefficient of variation for these macronutrients (Tab.1). The soil samples collected were predominantly medium loamy in texture, with clay content ranging from 20.6% to 58.7% (Tab. 1). All analyzed soil parameters exhibited similar values to those previously reported by Jordanoska et al. (2014), indicating consistent soil characteristics typical of tobacco growing soils in the Pelagonian region.

The descriptive statistics of the values of total K_2O, P_2O_5, Mg, Fe, and Na are presented in Table 1. The total potassium content in soils is typically between 0.5% and 3% (as K_2O), depending on soil texture and mineralogy (Lalitha & Dhakshinamoorthy, 2013; Firmano et al., 2020). The potassium content of 0.153% (as K_2O) determined in this study is below the average of 0.3% reported by Jordanoska Shishkoska (2014) for tobacco fields in the Pelagonian region.

The descriptive statistics of the values of total K_2O, P_2O_5, Mg, Fe and Na are presented in Table 1.

Table. 1. Basic soil properties and descriptive statistics of the analyzed parameters.

Parameter	Minimum	Maximum	Mean	Median	SD	CV (%)
Organic matter, %	0.56	2.98	1.62	1.53	0.61	38
Total nitrogen, %	0.03	0.14	0.08	0.07	0.03	38
pH (H_2O)	5.46	8.17	6.55	6.56	0.63	10
pH (KCl)	4.20	7.20	5.33	5.25	0.72	14
$CaCO_3$	0.00	5.09	0.20	0.00	0.93	465
av. P_2O_5, mg/100 g	2.38	149.9	17.62	6.59	34.52	196
av. K2O, mg/100 g	7.69	43.11	17.21	14.61	8.50	49
Clay, %	20.6	58.7	35.33	32.30	9.26	26
P_2O_5, %	0.04	0.848	0.356	0.328	0.163	46
K2O, %	0.040	0.698	0.153	0.115	0.152	99
Mg, %	0.019	0.477	0.091	0.064	0.108	119
Fe, %	0.256	3.152	0.843	0.651	0.600	71
Na, %	0.001	0.034	0.005	0.003	0.006	135

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

The total potassium content in soils is typically between 0.5% and 3% (as K_2O), depending on soil texture and mineralogy (Lalitha & Dhakshinamoorthy, 2013; Firmano et al., 2020). The potassium content of 0.153% (as K_2O) determined in this study is below the average of 0.3% reported by Jordanoska Shishkoska (2014) for tobacco fields in the Pelagonian region. According to the Geochemical Atlas of Macedonia (Stafilov & Šajn, 2016), the average potassium content in soils of the Pelagonian region is 2.3% based on total digestion. Phosphorus is a vital nutrient for plant growth, and its content in agricultural soils, like potassium, varies greatly depending on soil type, mineral composition, and management practices. Phosphate fertilizers used in agriculture to replenish the amounts of this macroelement in the soil can contain significant concentrations of heavy metals, depending on the origin of the phosphorus and the appetites used in their production (Alkorta et al., 2004).

The Fe content in the agricultural soil samples from the Prilep region (Tab. 1) is significantly lower (0.843%) than the mean iron content of 3.1% in the soils of the Pelagonian region and 3.6% in the soils of the entire country according to the Geochemical Atlas of Macedonia (Stafilov & Šajn, 2016). In addition, the mean Fe content is also significantly lower than the European average for agricultural topsoil, which is 2.6% based on total digestion (Salminen et al., 2005; Soriano-Disla et al., 2013). The average sodium content in soils from tobacco fields in the Prilep region is 0.005%, which corresponds closely to the mean value of 0.004% (or 41 mg/kg) reported by Jordanoska Shishkoska (2014) for soils in the Pelagonian region. Table 2 summarizes the descriptive statistics of As, Cr, Cu, Ni, Pb, and Zn in soils of tobacco fields in the Prilep region. Contamination of agricultural soils with arsenic is a pressing global concern due to its toxicity and potential to enter the food chain. Both natural processes and human activities contribute to elevated arsenic levels in soils. Uncontaminated soils usually contain arsenic concentrations around 5 mg/kg (Gonga et al., 2020; Rahman et al., 2023). The average arsenic concentration in agricultural soils used for tobacco cultivation in the Prilep region is 5.45 mg/kg (Tab. 1) and thus corresponds exactly to the average levels found in uncultivated soils in the Pelagonian region (5.6 mg/kg), and the wider average for soils in North Macedonia (9.2 mg/kg) (Stafilov & Šajn, 2016).

Table 2. As, 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	5.45	3.43	4.53	1.30	17.70	83
Cr	10.92	8.60	9.70	3.17	47.21	89
Cu	8.39	6.31	7.29	2.34	34.31	87
Ni	8.67	6.48	6.95	3.06	32.99	80
Pb	10.67	7.87	12.93	3.49	78.13	121
Zn	16.71	12.74	12.50	5.09	63.31	75

SD - standard deviation; CV - coefficient of variation

As shown in Table 2, the chromium content (10.92 mg/kg) in the soils of the Prilep region is significantly lower than the average value of 67 mg/kg reported for non-cultivable soils in the Pelagonian region (Stafilov & Šajn, 2016). Both values 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. Although the concentrations found are not critical, anthropogenic activities such as industrial emissions, improper waste disposal, and the intensive use of agrochemicals are considered to be the main sources of chromium accumulation in soil. Elevated levels of this element are of particular concern for the environment, as chromium is known to be highly toxic. It can impair soil microbial communities, reduce nutrient availability, and negatively impact plant growth and productivity (Zulfiqar et. al., 2023).

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). The mean copper concentration in agricultural soils in the Prilep region (mean value of 8.39 mg/kg), as presented in Table 2, is significantly lower than the mean value of 21 mg/kg reported for non-cultivable soils in the Pelagonian region (Stafilov & Šajn, 2016). This difference can be attributed to lower anthropogenic pressure in the cultivated areas or to differences in the natural soil composition. Copper is an essential micronutrient for plant metabolism, involved in enzymatic activities and photosynthesis; however, both deficiency and excess can have adverse effects on plant health. The lower levels found in the soil from the Prilep region suggest adequate but not excessive availability, which is favorable for the cultivation of sensitive crops such as oriental tobacco.

Nickel concentrations in agricultural soils are typically between 3 and 1000 mg/kg (Kamboj et. al., 2018), depending on soil type, parent material, and anthropogenic influences. According to the data presented in Table 2, the mean nickel content in the soils of the Prilep region (8.67 mg/kg) is significantly lower than the average value of 30 mg/kg reported for non-cultivable soils in the Pelagonian region (Stafilov & Šajn, 2016). This suggests that the tobacco-growing soils in the study area are not impacted by elevated nickel levels and fall within the range considered typical for uncontaminated agricultural soils.

The lead content in soils is usually between 10 and 50 mg/kg in uncontaminated areas, but can exceed 100 mg/kg in regions affected by anthropogenic activities (Kabata-Pendias, 2011). The mobility of lead in plants is highly restricted, as only about 3% of the lead absorbed by the roots being translocated to the aerial parts, such as the stems (Collin et. al., 2022). According to the data presented in Table 2, the mean lead concentration in the tobacco-growing soils of the Prilep region (10.67 mg/kg) is lower than the average value of 70 mg/kg given in the Geochemical Atlas of Macedonia for non-cultivable soils in the Pelagonian region (Stafilov & Šajn, 2016).

The zinc content in soil, which ranges from 10 to 300 mg/kg (Kabata-Pendias, 2011), is also influenced by several factors including soil texture, organic matter content, and pH. Zinc concentrations in the soil of the Prilep region (16.71 mg/kg) were within the expected range for agricultural soils, indicating a balanced availability of this essential micronutrient for plant growth.

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).

CONCLUDING REMARKS

This study provides a comprehensive assessment of the agrochemical and elemental composition of soils used for oriental tobacco cultivation in the Prilep region, a major tobacco-growing area in North Macedonia. The results revealed considerable variability in the concentrations of macro- and microelements (K, Mg, Fe, Na), primarily influenced by differences in land management practices and the natural geological and pedological diversity of the region. Despite long-term and intensive tobacco production, this variability did not indicate contamination.

The evaluated soils were generally classified as having low to moderate organic matter content, low total nitrogen, slightly acidic to neutral pH, and variable phosphorus and potassium availability, reflecting a diversity of fertility levels across the region. The predominant soil texture was medium loam with moderate to high clay content, which is favorable for high-quality oriental tobacco cultivation.

Importantly, the concentrations of potentially toxic elements (As, Cd, Cr, Cu, Ni, Pb, Zn) remained below internationally recognized thresholds for heavy metals in agricultural soils. These findings confirm that the soils in the Prilep region are ecologically safe and agronomically suitable for sustainable oriental tobacco production.

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ХЕМИСКА КАРАКТЕРИЗАЦИЈА НА ТУТУНСКИ ПОЧВИ ВО ПРИЛЕПСКИОТ РЕГИОН: ЕКОЛОШКИ И ЗЕМЈОДЕЛСКИ АСПЕКТИ

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Резиме

Квалитетот на почвата игра суштинска улога во земјоделската продуктивност, особено при одгледување тутун, каде што се неопходни и висок принос и врвен квалитет. Елементниот состав на почвата, особено рамнотежата помеѓу есенцијалните хранливи материи и потенцијално токсичните елементи, значајно влијае врз квалитетот на почвата и развојот на растенијата. Оваа студија го оценува квалитетот на почвите во Прилепскиот Регион во Северна Македонија, главната област за производство на ориентален тутун. Почвените примероци беа земени од избрани тутунски површини за време на вегетациските сезони во 2021 и 2022 година и анализирани со индуктивно спрегната плазма масена спектрометрија (ICP-MS) за определување на концентрациите на избрани макро- и микроелементи (K, Mg, Fe и Na), како и на потенцијално токсични елементи (As, Cd, Cr, Cu, Ni, Pb и Zn). За целосна проценка беа анализирани и неколку агрохемиски параметри, вклучувајќи содржина на органска материја (од ниска до умерена), вкупен азот (0,03–0,14 %), pH на почвата (просек 6,55; слабо кисела до неутрална), достапен фосфор и калиум, физичка структура на почвата (средно глинеста текстура) и содржина на глина (од 20,6 % до 58,7 %). Концентрациите на макро- и микроелементи се тесно поврзани со геолошко-педолошките карактеристики на регионот, додека нивото на потенцијално токсични елементи е под меѓународно прифатените прагови за тешки метали, што укажува на низок ризик од загадување и потврдува погодност на овие почви за одржливо производство на тутун.

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