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IOSUD - „DUNĂREA DE JOS” UNIVERSITY OF GALAȚI

Doctoral School of Mechanical and Industrial Engineering

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DOCTORAL THESIS

ABSTRACT

Research on iron and steel works industry impact on soil edaphic and vegetal potential in the adjacent areas

PhD Student,
Sorina-Simona ARBANAȘ (MORARU)

Scientific Coordinator,
Prof. univ. dr. eng. habil. Antoaneta ENE

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GALAȚI 2022

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President: Prof. univ. dr. eng. Cătălin FETECĂU
President of the Senate - „Dunărea de Jos" University of Galați

Scientific Coordinator: Prof. univ. dr. eng. habil. Antoaneta ENE
„Dunărea de Jos" University of Galați

Scientific Referents: Prof. univ. dr. eng. Cristian - Vasile DOICIN
POLITEHNICA University of Bucharest

Prof. univ. dr. Octavian G. DULIU
University of Bucharest

Prof. univ. dr. eng. Luminița MORARU
Director of the Doctoral School of Mechanical and Industrial Engineering - „Dunărea de Jos" University of Galați

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FOREWORD

The greatest gift a man can receive is to be healthy and to have people by his side, to support him unconditionally, bring him balance and inner peace in moments of hesitation, and encourage him when he is about to give up. First of all, I would like to thank *my family*, who, throughout my doctoral studies, supported me with patience and comprehension, because they instilled in me a tireless desire to know and surpass myself.

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I express my gratitude to *Assoc. prof. dr. Steluța Gosav*, a Faculty of Sciences and Environment teacher and *Lecturer dr. eng. Dana Iulia MORARU*, teacher of the Faculty of Food Science and Engineering, for the scientific support, the constructive advice I received and the didactic guidance. I would also like to thank *RS dr. Ana PANTELICĂ* and the team of physicists from "Horia Hulubei" National Institute of Physics and Nuclear Engineering of Magurele, to *Dr. Magdalena AFLORI* from "Petru Poni" Institute of Macromolecular Chemistry of Iasi, to *Lecturer dr. eng. Alina Mihaela CEOROMILA*, teacher of the Cross-Border Faculty, "Dunarea de Jos" University of Galati, and *Assoc. prof. dr. Claudia STIHI*, a teacher at the "Valahia" University of Targoviste, for supporting me in applying some modern methods of environmental samples advanced analysis.

I thank the INPOLDE Research Center of the "Dunarea de Jos" University of Galati and the following projects coordinated by *Prof. dr. eng. habil. Antoaneta ENE*, in which the results presented in this doctoral thesis were acquired: Black Sea Basin Operational Program 2014-2020, projects code BSB27-MONITOX and code BSB165-HydroEcoNex, Projects JINR-Romania no. 111/2018, 112/2018, 64/2019, 63/2020, 68/2020 and 71/2020, as well as the JINR-Romania Research Grants no. 21/2018, 26/2019 and 32/2020.

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At the same time, I would like to thank my colleagues from the County Soil Survey Office of Galati, *Director dr. Alina BADILA* and *Ec. Camelia Beatrice COMAN*, who supported me morally and helped me in the documentation process, and *Jr. Gabriel Marian PISICA*, director of the County Soil Survey Office of Brăila, for the support provided for documentation.

Last but not least, I would like to address my doctoral colleagues, my friends and all those that directly or indirectly participated with advice, positive thinking and support in achieving this goal.

Galati, 2022 Sorina-Simona Arbanas (Moraru)

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INTRODUCTION

Soil, an essential natural resource for maintaining life on Earth, is located at the intersection of geospheres and has a permanent relationship of matter and energy exchange with them. The ecological importance of soil has already been put into evidence by many studies [Verhoef, H.A., 1996], [Blum, W.E.H., 2005], [Trap, J. et al., 2016], edaphic conditions influencing the normal development of plants. Soils located near industrial areas, heavily trafficked roads, mines, and landfills receive significant amounts of toxic chemical elements and compounds (As, Cr, Zn, Cu, Ni, Pb, Cd, polychlorinated biphenyls, hydrocarbons, etc.) [Ene, A. et al., 2011a], [Popescu, I.V. et al., 2014], [Kumar, A. et al., 2020], [Danielovič, I. et al., 2014]. It causes disturbances in the natural development of biological processes as they may be transferred into the tissues of plants and introduced into the food chain. Harmful elements are introduced into the soil and through products used in agriculture for fertilization and fungal treatment of cultivated plants.

Research topic motivation

In recent decades, internationally and nationally, many studies have been conducted on the impact of industrial activities on various components of the environment, the transfer of contaminants in the relationship between air-soil-water-vegetation-humans/ animals and the effect of stress caused by pollutants on living organisms. In the Galati-Braila region, there are fewer data regarding the contamination of the soil and sediments with heavy metals induced by the steel or other anthropogenic activities. With regard to the contamination of crops, vegetables and fruits, fodder plants with toxic elements from industrial activity, mismanagement of fertilizer doses, amendments, pesticides and/or road traffic, for the area under investigation in this paper, there are no data to reflect the real situation.

Under these conditions, the doctoral thesis ***Research on iron and steel works industry impact on soil edaphic and vegetal potential in the adjacent areas*** has as its main objective the study of the level of contamination with major and minor elements of the upper layer of agricultural soils in the vicinity of Galati Iron and Steel, as well as the concentration of these elements in the vegetable sections of wheat, maize and sunflower, grown on these soils.

The research topic addressed in this paper will contribute to the local and national database with results for the level of contamination with heavy metals and other toxic elements of soils and crop plants, as well as information on the mineralogical footprint specific to Tulucesti and Sendreni territories (Galati county) and Vadeni (Braila county). This paper may be an outset for new directions of agropedological research and plant physiology for the southeastern region of Romania.

The doctoral thesis ***Research on iron and steel works industry impact on soil edaphic and vegetal potential in the adjacent areas***, developed under the scientific coordination of Prof. PhD. Eng. Habil. Antoaneta Ene (scientific coordinator), Prof. PhD. Eng. Luminița MORARU, Prof. PhD. Eng. Gabriela Elena BAHRIM and Prof. PhD. Habil. Cătălina ITICESCU is structured in 9 chapters, 312 pages and includes 70 tables, 87 figures and 53 annexes.

Chapter 1 presents aspects regarding the characterization of the pedogenetic factors involved in the formation and evolution of the soils in the studied area. At the same time, it gives information about the natural agricultural potential of the soil in the territories of Tulucesti and Sendreni, Galati county and Vadeni, Braila county, to the ecological factors.

Chapter 2 describes the methodology of environmental sampling and the analytical methods used to determine their physical properties and chemical composition. At the same time, it presents the methods for determining hydrophysical indices, evaluating the impact of pollutants on soil quality through specific pollution indices, and the bioaccumulation of toxic elements in crop plants are specified.

Chapter 3 describes the physical and hydrophysical properties of the researched agricultural soils.

Chapter 4 consists of the agrochemical parameters of the soil involved in the development of plants, in general, and of crops, in particular.

Chapter 5 highlights the concentrations of micro and macroelements in the soil and plant tissues of wheat, sunflower and maize and related connections to the legislation in force and the literature.

Chapter 6 contains ATR-FTIR and SEM-EDX microstructural and mineralogical composition results of agricultural soils and tissues of wheat, maize and sunflower.

Chapter 7 presents the soil pollution levels assessment by simple and complex pollution indices.

Chapter 8 highlights the bioaccumulation of heavy metals levels in wheat, maize and sunflower, assessed by the bioaccumulation factor.

Chapter 9 contains general conclusions of experimental activity, the original contributions and future research directions.

Original contributions and novelty issues

The doctoral thesis ***Research on iron and steel works industry impact on soil edaphic and vegetal potential in the adjacent areas*** is the first scientific contribution to complex soil investigation to the Galati-Braila region, including mineralogical composition, physical, hydrophysical and chemical properties, aspects of soil and crop plants pollution with heavy metals and trace elements. Comparative analysis of toxic elements bioaccumulation in the plant tissues of three crops (wheat, maize and sunflower) having the largest share among agricultural plants grown in the counties of Galati and Braila and source of nutrition for the population, no more has been addressed by other previous studies. Another novelty of this paper is the varied multi-elemental analysis of soil and plants in agroecosystems located in the vicinity of the Galati Iron and Steel Works, using complementary instrumental advanced techniques of investigation, and high sensitivity (spectrometric, atomic and nuclear), which allowed the identification the lowest concentrations of elements.

The research results were presented at national and international conferences, mentioned in the final part of the thesis under the heading *Dissemination of research results*, Ten scientific papers were published, of which 1 article in an ISI indexed journal (IF 2020 = 2,576) and 9 articles in BDI indexed journals. It is to mention that some national and international projects and grants, in which the author was a member of the implementation team or volunteer, supported the research activity during the doctoral studies.

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I. *THE RESEARCH STATE OF THE ART*

1. DOCUMENTARY STUDY

This chapter presents aspects regarding the characterization of the pedogenetic factors involved in soil formation and evolution (characterization of relief, geology, hydrography, climatic conditions, vegetation, anthropogenic influence), description of the soil cover and its agricultural potential.

1.1. The studied area physico-geographical framework

1.1.1. *Study area geographical position*

From the geographical position point of view, the studied territories are in the east of Romania, south of Moldova (Sendreni and Tulucesti communes), and northeast of Muntenia (Vadeni commune) - Figure 1.1.

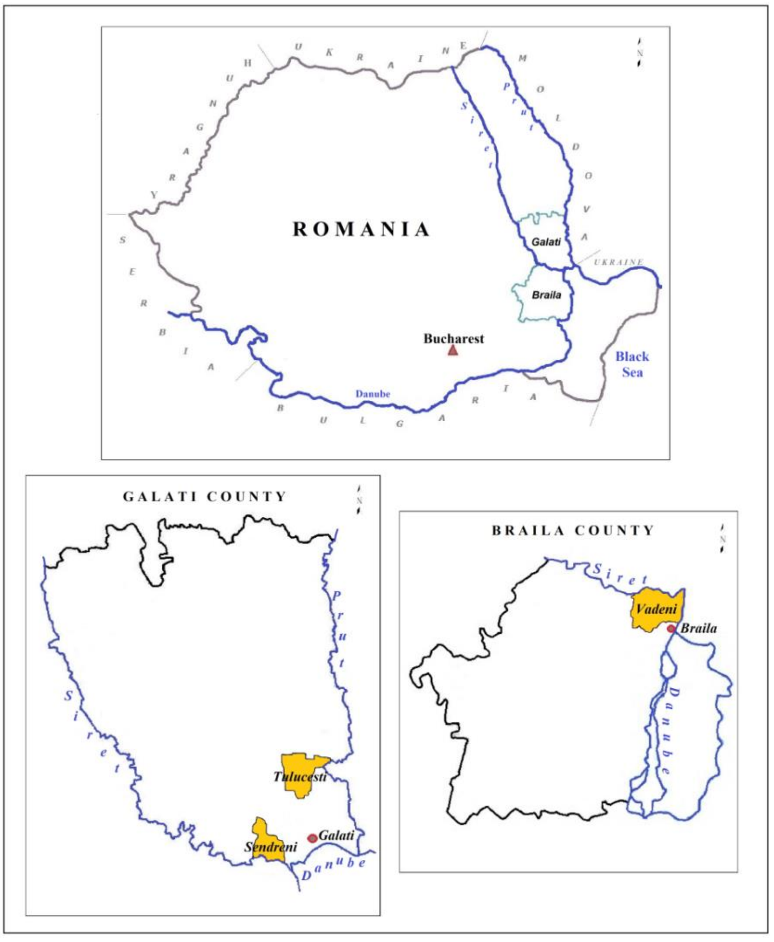


Figure 1.1 Location of Tulucesti, Sendreni, Galati county and Vadeni, Braila county (maps outline processed after [ANCPI, https://geoportal.ancpi.ro/portal.home/])

Tulucesti administrative territory is in the south-eastern part of Galati county, being delimited by the following: Frumusita, to the north, Republic of Moldova, to the north-east (natural border Prut river), Vanatori, to the south, Galati city, to the east and Smardan, to the west. It consists of the villages of Tulucesti (the residence of the commune), Tatarca and Sivita. Compared to Galati Steel Plant (LIBERTY Galati) it has a NE position at a distance of about 18 km.

Sendreni administrative territory is in the southern part of Galati county and is bordered by: Smardan commune in the north, Vadeni commune (Braila county) in the south, the territory of Galati municipality in the east and north-east, Branistea and Schela communes in the west. Sendreni commune includes the villages of Sendreni (commune residence), Movileni and Serbestii Vechi. The entire eastern side of this territory borders the Galati Iron and Steel Works.

Vadeni administrative territory is in the northeast of Braila county and in the south of Galati county. Vadeni commune is bordered by Sendreni and Galati, to the north (natural limit of the Siret river), Cazasu and Braila to the south, Smardan and I.C. Bratianu (Tulcea county) to the east (natural limit of the Danube river) and Silistea to the west. It consists of Vadeni (commune residence), Baldovinesti and Pietroiu villages. Vadeni commune has a southern position at about 20 km from the Galati Iron and Steel Works.

1.1.2. *Aspects regarding the relief of Tulucesti and Sendreni (Galati county) and Vadeni (Braila county) territories*

The studied areas belong to the following geomorphological units: the Covurlui Plateau, in the Covurlui Plain compartment, consisting of two subunits - the “Baleni-Tulucesti plateau microdistrict” (Tulucesti territory) and the “terraced interfluves microdistrict of Pechea-Galați ” (Sendreni territory) [Sficlea, V., 1980] and the Romanian Plain, in the Lower Siret Plain subunit, which cover the extreme south of Sendreni and Vadeni areas) [Coteț, P.V., 1976]. The relief of the studied region is closely related to the petrographic composition and the orientation of the geological strata. Thus, the Tulucesti and Sendreni communes relief resemble a low fragmented and NNW-SSE sloped plateau. The altitude decrease constantly from north to south, from 150 m in the north of Tulucesti commune to the Siret corridor (in the south of Sendreni commune), where at the contact with its meadow it has the lowest values (5-6 m). Considering the subsidence movement in the Lower Siret Plain, on the territory of Vadeni commune, the altitude decreases from 23 m in the south, at the contact with north of Baragan, to 2-6 m towards Siret meadow, located in the north of the territory.

1.1.3. *Aspects regarding the geology of Tulucesti and Sendreni (Galati county) and Vadeni (Braila county) territories*

The formation of the relief and the distribution of soil types require knowing about the evolution and the geological composition of the interest area study.

The territories of Tulucesti and Sendreni overlap with the Covurlui Platform. The defining characteristic of this structural unit is the tectonic sinking due to the southern part of the Moldavian Platform fall, with which it borders to the north, and of the North Dobrogea Promontory, located at the south of the depression. The administrative-territorial unit of Vadeni is situated on the Moesic Platform, the Valah compartment, having lithological facies different from its other structural sections.

The basement of the Covurlui Platform consists of crystalline schists of Precambrian age (gneisses, amphibolites, eruptive rocks) and Paleozoic, Triassic and Liasic sediments (sandy conglomerates, limestones, dolomites, sandstones, et cetera) [Băcăuanu, V. et al., 1980], [Sficlea, V., 1980], covered by Jurassic, Cretaceous, Neozoic and Quaternary formations. Many excavations have shown that the basement of the Wallachian Platform consists of crystalline schists with magmatic intrusions of the Late Proterozoic-Lower Paleozoic age.

The sedimentary cover consists of a loess cover, which in the south of the Covurlui Plateau is tens of meters thick. Along with the Siret Valley and within the other valleys, the Quaternary deposits are of fluvial origin.

1.1.4. *Aspects regarding the climatic conditions in the Galati-Braila area*

Due to the geographical position and the complexity of the factors, the studied region is part of a temperate-continental climate, characterized by hot summers, long periods of drought and harsh, windy and frosty winters. According to the Kőppen classification, Bsax code [Kőppen, W. and Geiger, R., 1936], [Kottek, M. et al., 2006], the study area is in the steppe climate region with severe summer drought, characterized by an average temperature of the warmest month of ≥22°C, with a maximum of precipitation achieved in summer and a minimum towards the end of the year.

Thermal regime. From a thermal point of view, wide variations during the year are noticed. *The annual mean temperature* is very high (10.7°C (Galati) and 11.0°C (Braila)). *The annual mean thermal amplitude* is high, with a value of 25.4° in Galati and 33.3° in Braila, which marks the climatic continentalism [Apostol L., 2000], as Romania is in an area with thermal amplitudes higher than 20°C.

Precipitation regime. After [OSPA Galați, 2014 and 2020] and [OSPA Brăila, 2018], the precipitations record high oscillations from one season to another. The annual mean precipitation is low. This variability in precipitation emphasizes the continental character of the climate. *The multiannual mean precipitation* amounts are 485.7 mm at Galati and 450 mm at Braila. Most of it falls in May-September, associated with heavy rain or hail.

1.1.5. *Aspects regarding the hydrography of Tulucesti and Sendreni (Galati county) and Vadeni (Braila county) territories*

The hydrographic network of the studied area belongs to the Siret and Prut river basins of the eastern group of Romania.

In the studied territories, the level of pedophreatic water is at more than 10 m depth on interfluves and 0.51-5.00 m in the meadow areas.

1.1.6. *Aspects regarding the vegetation and fauna of Tulucesti and Sendreni (Galati county) and Vadeni (Braila county) territories*

Considering the position within the Pontic Floristic Province, the researched region has a definite ecological potential both of the variety of species and their distribution in space. Humans' historical activities have generated changes in the existing plant species. At the same time, the accentuated continentalism led to the retreat to the north of the forest steppe and the installation of the steppe vegetation, an extension of the Ukrainian steppe in the Covurlui High Plain and the Lower Siret Plain. (Ponto-Sarmatic steppe [Băcăuanu, V. et al., 1980]). The Sarmatian-Pontic fauna is connected with the flora and has rarer specimens of Eastern Mediterranean origin [Băcăuanu, V. et al., 1980].

1.1.7. *Aspects regarding the soils of Tulucesti and Sendreni (Galati county) and Vadeni (Braila county) territories*

According to the pedogeoclimatic microzones of Romania [Florea, N. et al., 1999], the studied territories fall into the following pedogeoclimatic areas: Tulucesti and Sendreni territories: IL-SA 30/7a - alluvial soil microzone, with hot-dry climate, in meadow relief; IC-CZ

11/1a - chernozem microzone, with hot-dry climate, in regions with low rugged relief. Vadeni territory: IL-SA 30/8 - alluvial soils microzone, with hot-dry climate, in meadow relief; IO-PS 28/2 - microzone of sandy soils (psamosols), with hot-dry climate, in regions with wavy relief; IS-CZ 4/3 - microzone of calcaric chernozems, with hot-dry climate, in lowland regions; IL-LC 24/1 - gleysols microzone, with hot-dry climate, in meadow regions.

The taxonomic classification of soils

After [OSPA Galați, 2014 and 2020] and [OSPA Brăila, 2018], the soils present in the researched area belong to the classes Protisols (Regosol, Psamosol and Fluvisol types), Cernisols (Chernozem and Phaeozem types), Hydrisols (Gleysol type), Salsodisols (Solonchak type) and Anthrisols (Anthrosol type).

1.1.8. *Aspects regarding the anthropic influence on Tulucesti and Sendreni (Galati county) and Vadeni (Braila county) territories*

Since ancient times, the anthropogenic factor has influenced the process of pedogenesis in various forms. Increasing anthropogenic pressure on ecosystems in general and on agroecosystems, in particular, can cause some of the most severe, hard-to-fix degradation.

From the land use viewpoint, Galati and Braila counties are known for their great agricultural lands. The arable lands on Tulucesti and Sendreni territories represent 85.20 % and 86.62 %. In the Vadeni commune, 94.79 % of the agricultural area is arable land. Wheat, maize and sunflower are the most cultivated plants. It is important to note that, over time, the agricultural land borders have fluctuated depending on the works to expand the built-up areas of the localities and the construction of various objectives of local interest (construction of wind and photovoltaic parks, expansion of the road network, et cetera).

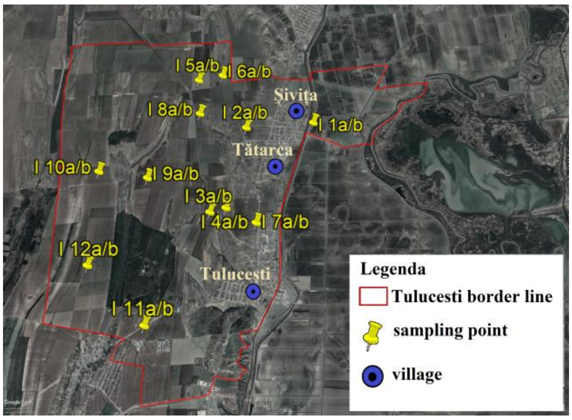
II. *PERSONAL CONTRIBUTIONS*

2. MATERIALS AND METHODS

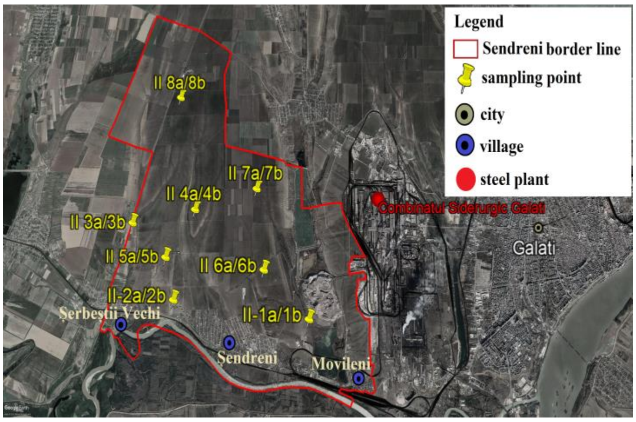
2.1. Land pedo-agrochemical mapping. Soil and crops sampling

2.1.1. *Soil and plant sampling*

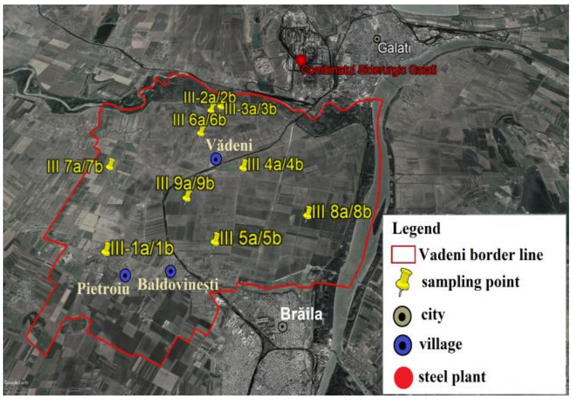
The samples were collected from arable lands cultivated with wheat, maize and sunflower near the Galati Iron and Steel Works (Liberty Galati). The field trip took place in July, August, September and October 2018 and 2019, depending on the stage of physiological maturity of crops. Figure 2.1. show the position of the sampling points.



(a)



(b)

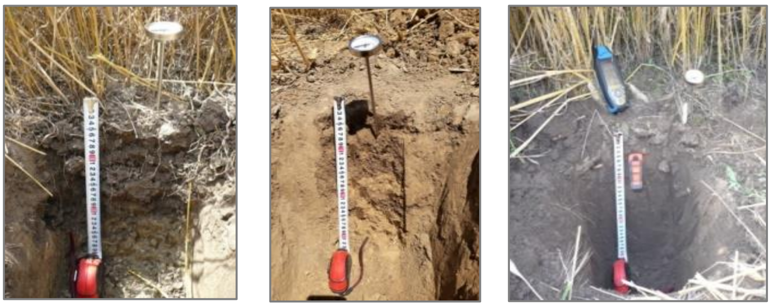


(c)

Figure 2.1 Soil sampling points location: (a) Tulucesti, (b) Sendreni, (c) Vadeni (source: processed after Google Earth Pro)

Soil sampling

Soil samples were taken from pits with a size of 30x30x30 cm by 0-5 cm and 5-30 cm depth (Figure 2.2) according to [Order no. 184/1997], to determine the concentrations of heavy metals and trace elements, and [STAS 7184/1-84] for the level of soil nutrients and physical properties. Fifty-eight soil samples were collected, 29 for each depth.



(a) (b) (c)

Figure 2.2 Delimitation of soil depth sampling: (a) Tulucesti (sample I-1 a/b), (b) Sendreni (sample II-4a/b), (c) Vadeni (sample III-1 a/b) (source: Moraru S.S.)

After delimiting the sampling depths and refreshing the excavation, the soil samples were collected from the bottom up to avoid contamination with the material in the upper layer. Natural and disturbed state soil samples were taken (Figure 2.3). The soil in the natural state was collected in 200 cm^3 cylinders, provided with a plastic cover according to [Florea, N. et al., 1987c].

(a) (b) (c)

Figure 2.3 Soil sampling: (a) disturbed sample, (b) și (c) undisturbed sample (source: Moraru S.S.)

Plant sampling

Crop plants samples (wheat - Triticum Vulgare Vill., Corn - Zea mays L. and sunflower - Helianthus annuus L.) consisted of healthy, mature individuals, harvested from the lots in which the soil samples were taken according to [Răuță, C. et al., 1981], [Kalra, Y.P., 1998]. During the sampling procedures, ends of the lots and visibly affected plants were avoided. Appendices 2.2a, 2.2b and 2.2c show sampling lots and their positions in the territory.

Preparation of samples for analysis

Preparation of soil samples

Soil sample preparation for the principal physico-chemical parameters analysis was performed according to [Stoica, E. et al., 1986] and after [SR ISO 11466: 1999] for heavy metals concentration determination.

Preparation of plant samples

The plant preparation procedure for analysis was performed after [Kalra, Y.P., 1998]. Figure 2.5 shows the studied plant species and their sections.

(a) (b) (c)

Figure 2.5 Samples of plants in their natural state and after washing and air drying: (a) wheat (*Triticum vulgare* Vill.), (b) maize (*Zea mays* L.), (c) sunflower (*Helianthus annuus* L.) (source: Moraru S.S.)

2.2. Soil and plant methods of analysis

Soil and plant samples, prepared according to subchapter 2.1.1., were subjected to the following types of analyzes:

- *Analysis of soil physical properties:* granulometric composition (according to [STAS 7184/10-79]), soil volumetric weight and humidity (according to [SR EN ISO 11272: 2014] and [SR ISO 11465: 1998]) within OSPA Galati;

- *Analysis of soil chemical properties:* determination of pH (according to [SR 7184/13: 2001]), total nitrogen concentration (Kjeldahl method), mobile phosphorus (extraction according to Egner-Riehm-Domingo method and dosing by UV-VIS spectrophotometry), mobile potassium (extraction according to the Egner-Riehm-Domingo method and dosing by flame atomic emission spectrometry), humus content, organic carbon (according to [STAS 7184/21-82]), $CaCO_3$ content (volumetric method according to [SR EN ISO 10693: 2014]), the level of the sum of the exchange bases, the hydrolytic acidity and the degree of base saturation (after [Borlan, Z. and Răuță, C., 1981]), the electrical conductivity and the total content of soluble salts (after [SR ISO 11265 + A1: 1998] and [STAS 7184 / 7-87]) within OSPA Galați. The interpretation of the results was according to the classifications presented by [Florea, N. et al., 1987a, c] and [Florea, N. et al., 2012].

- *Analyzes by advanced complementary methods for soil and plant study - spectroscopic methods:* mineralogical and elemental analysis by SEM-EDX analysis technique in the Electron Microscopy Laboratory of the Faculty of Sciences and Environment - UDJG and ICMPP Iasi and ATR-FTIR technique in the INPOLDE Research Center of UDJG; elementary analysis by the HR-CS AAS method in the INPOLDE Research Center of UDJG and ICP-MS within the Multidisciplinary Scientific and Technological Research Institute of Targoviste (ICSTM-UVT); *nuclear methods:* elemental analysis by PIXE, PIGE and RBS techniques within IFIN-HH Magurele.

The methods of analysis are described in the extenso doctoral thesis, subchapters 2.2.1 - Methods of physical analysis of soil, 2.2.2 - Methods of chemical analysis of soil and 2.2.3. - Complementary advanced spectroscopic methods.

2.3. Calculation methods for the determination of soil hydrophysical indices, assessment of pollution by soil-specific pollution indices and plant bioaccumulation factors

2.3.1. *Determination of soil physical and hydrophysical indices*

The soil's physical and hydrophysical indices were calculated based on the colloidal clay content, dust, and volumetric weight according to the formulas given by [Florea, N. et al., 1987c], [Dumitru, E. et al., 2009] and [Dumitru, M. and Manea, A., 2011]. The interpretation of the results was according to the appreciation classes of the same authors (extensively in the thesis).

2.3.2. *Determination of bioaccumulation factors of metals in plants*

The bioaccumulation factor, also called the transfer coefficient [Ishii, K. et al., 2015], was calculated according to the formula:

$$BF = \frac{C_i plant}{C_i soil}$$

where: BF - bioaccumulation factor; Ci plant - the concentration of metal *i* in plant sections (mg/kg d.w.); Ci soil - the concentration of the metal *i* in soil (mg/kg d.w.). [Olowoyo, J.O. et al., 2010] and [Rădulescu, C. et al., 2013] appreciate that at values of BF>1 the plant is a bioaccumulator, if FB = 1 the metals do not influence the physiology of the plant, this can be appreciated as an indicator, and when FB <1, the plant excludes that element in the nutrition process.

2.3.3. *Determination of soil pollution indices*

In recent decades the use of pollution indices in assessing the level of soil and sediment contamination has proven to be particularly useful. It provides essential information on the source of contamination, whether natural, anthropogenic or due to the combined action of the two factors, and the risk on the environment associated with it [Müller, G., 1969], [Gong, Q. et al., 2008], [Håkanson, L., 1980], [Cabrera, F. et al., 1999], [Reimann, C. and de Caritat, P., 2005], [Kalavrouziotis, I.K. et al., 2012], [Nikolaidis, C. et al., 2010].

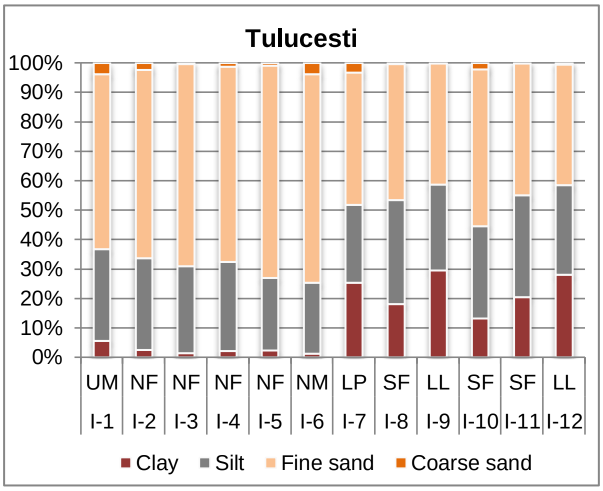
The following pollution indices were calculated to assess the level of heavy metal soil pollution in the territories in the vicinity of the Galati Steel Plant: *simple pollution indices* (Geoaccumulation index - Igeo, Enrichment Factor - EF, Pollution Index - PI) and *complex pollution indices* (Sum of Pollution Index - PIsum, Nemerow Pollution Index - $PI_{Nemerow}$, Contamination Severity Index - CIS, The Mean Effect Range Median Quotient - MERMQ, Pollution Load Index - PLI, The New Pollution Index - PIN, Potential Ecological Risk Index - PERI). The extenso version of the doctoral thesis presents the calculating formulas and the classes of values appreciation.

3. RESEARCH ON THE CHARACTERIZATION OF THE PHYSICAL AND HYDROPHYSICAL PROPERTIES OF AGRICULTURAL SOILS IN THE AREAS ADJACENT TO THE GALATI STEEL PLANT

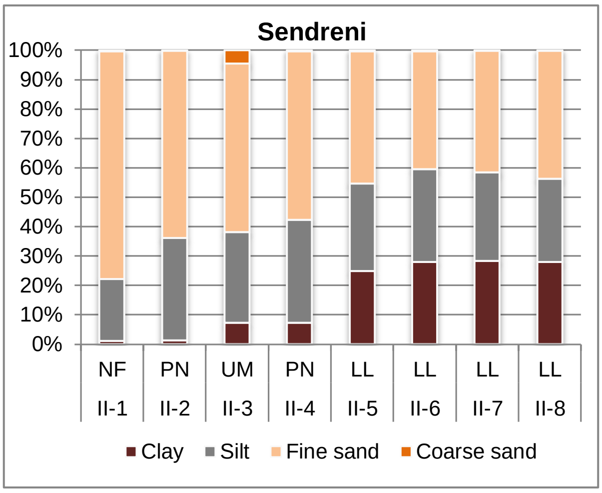
3.1. Physical properties of agricultural soils study

3.1.1. *Soil texture*

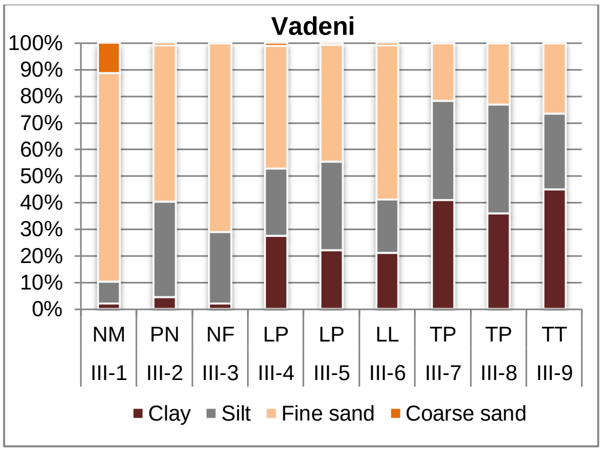
The influence of the soil granulometric composition and the other physical properties (volumetric weight, compaction degree, total porosity, aeration porosity) on the hydro-physical indices and the absorption and circulation of nutrients and toxic elements in the soil-plant system was presented at the international conference [Arbanas (Moraru), S.-S. et al., 2019c] and published in [Moraru, S.-S. et al., 2020]. The investigated soils fall into the coarse, medium and medium-fine texture classes. Given the same geological foundation of the Tulucesti, Sendreni, and Vadeni territories, the particle size composition of the soils is similar. Figure 3.1 show the percentage distribution of analytical values.



(a)



(b)



(c)

Figure 3.1 Percentage distribution of particle size fractions in investigated soils by texture subclasses: (a) Tulucesti, (b) Sendreni, (c) Vadeni (processed after [Moraru S.-S. et al., 2020])

For the studied area [OSPA Galați, 2014, 2020], [OSPA Brăila, 2018], [Arbanas (Moraru), S.-S. and Ene, A., 2018c] and [Moraru, S.-S. et al., 2018] reported the same textural classes and subclasses.

3.1.2. *Soil bulk density*

The soil's volumetric weight, also called bulk density (BD), is an important physical parameter for other physical parameters estimation (total porosity, aeration porosity, soil compaction), organic matter and nutrient reserves, and the hydro-physical indices of the soil.

For the studied territories, the volumetric weight of the soil falls within the extremely low-medium limits due to ploughing of the 0-30 cm sampling layer [Moraru, S.S. et al., 2020]. In each territory, the bulk density correlated with the type of texture falls into the following classes: extremely low (0.96-1.21 g/cm^3) - samples I-1, I-2, I-3, I -4, I-5, I-6, I-8, I-11, I-12, characterizing the soils with coarse texture, and very low (1.16-1.25 g/cm^3) - samples I-7, I-9, I-10, for soils with clayey texture, on Tulucesti territory; extremely low (0.82-1.07 g/cm^3) - samples II-2, II-3, II-5, II-6, II-7, II-8, with general-clay texture, and very low (1.21-1.26 g/cm^3) - samples II-1, II-4, with coarse and silty texture, on Sendreni territory; extremely low (0.91-1.34 g/cm^3) - samples III-3, III-4, III-6, III-7, III-8, on coarse to clayey textures, very low (1.16-1.34 g/cm^3) - samples III-1, III-2, III-9, having a coarse, silty and clayey texture, and medium (1.40 g/cm^3) - sample III-5, with a clayey texture on the territory of Vadeni.

3.1.3. *Soil physical indices*

The study of the soil physical indices at a given time and under certain conditions is of particular importance in assessing the physical characteristics involved in the absorption and flow of nutrients and toxic or potentially toxic substances in the soil and from it to the organs of plants. It is also necessary to study the role they have in the process of biological processes.

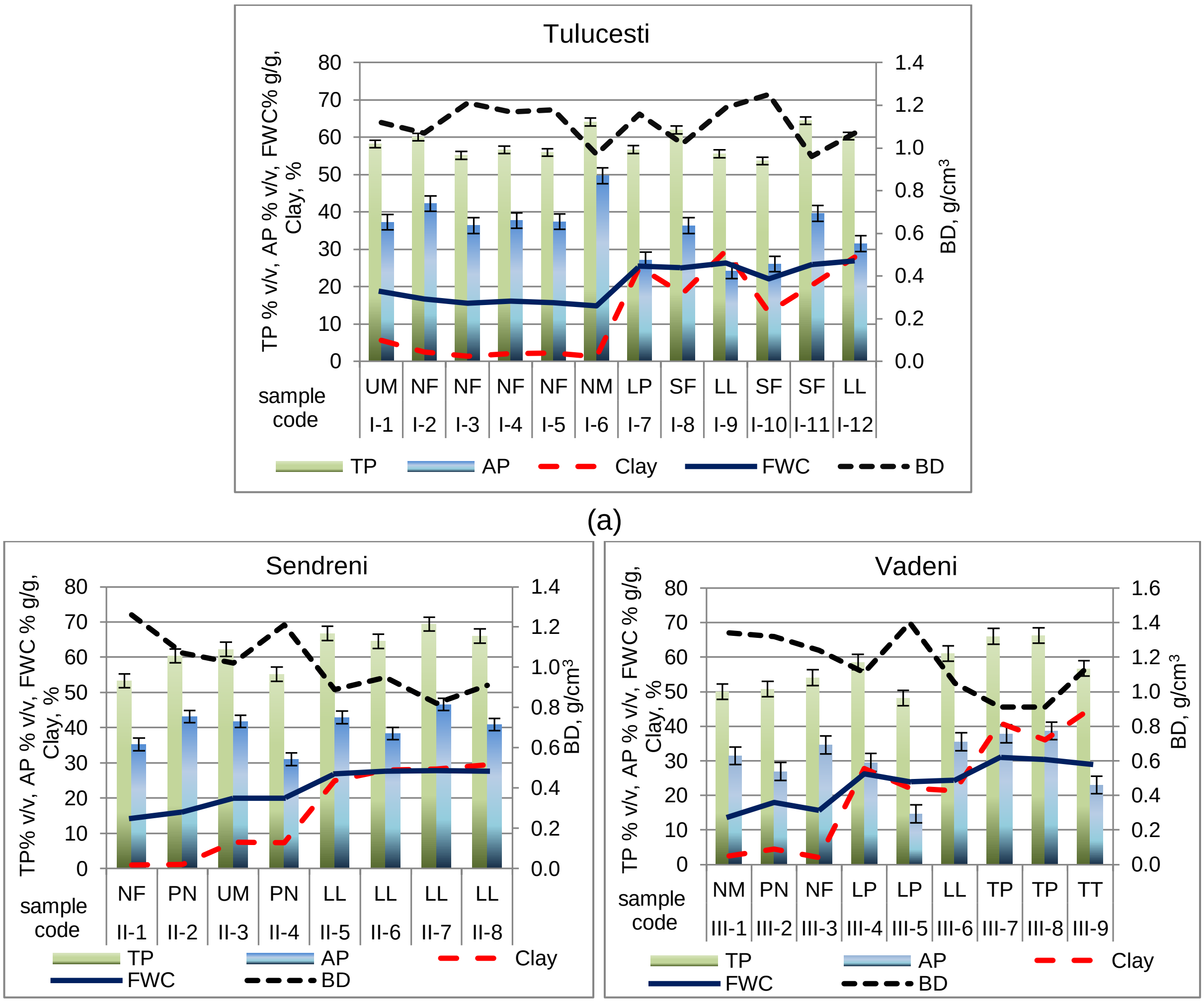
Among the physical indicators of importance in nutrients and pollutants transfer are the degree of compaction, total porosity and aeration porosity [Moraru, S.-S. et al., 2020]. Figure 3.2 shows the situation of the soil's total and aeration porosity to the bulk density, clay content and filed water capacity.

The *compaction degree* (CD) depends on the volumetric weight, the percentage of colloidal clay and the soil's total porosity (TP). The investigated soils have a compaction degree that varies from extremely low (-41.77- -19.32% v/v), very low (-18.28- -11.60% v/v) and low (-10.99- -8.35% v/v) to medium (0.96% v/v), which corresponds to a very loosened, loosened, non-compacted and slightly compacted state [Dumitru, E. et al., 2009].

The *total porosity* or the soil's total volume of pores is connected to the granulometric fractions of soil composition, bulk density and density, the latter being assessed according to the organic matter in the soil [Florea, N. et al., 1987c]. For the studied territories, TP is in the extremely high (55.97-69.40 % v/v) - medium (48.15 % v/v) variation range.

The *aeration porosity* (AP) is an indicator of the volume of soil pores occupied by air, essential in assessing the optimal conditions for the development of crops. This physical index is dependent on the content of colloidal clay, organic matter, bulk density and field water capacity. Near the Galati Iron and Steel Works, results highlighted values that fall in the low (14.65 % v/v) to very high (31.03-49.71 % v/v) variation range.

[Arbanas (Moraru), S.-S. et al., 2018c], [Moraru, S.-S. et al., 2018] reported similar values for arable and pasture soil on the Smardan territory, located in the north of Sendreni commune and the west of Tulucesti commune. Data on soil characterization in terms of physical indices were partially published in the paper [Moraru, S.-S. et al., 2020].



(a)

(b) (c)

Figure 3.2 Total porosity and aeration porosity variation with the clay content, bulk density and field water capacity in the 0-30 cm layer of the arable lands' soil on the territories: (a) Tulucesti, (b) Sendreni, (c) Vadeni (processed after [Moraru, S.-S et al., 2020])

3.2. Hydrophysical properties of agricultural soils study

3.2.1. *Field water capacity (FWC)*

The experimental data presented in Figure 3.3 show that the field water capacity is proportional to the soil content of colloidal clay. Therefore, soils with coarse and silty textures have a low field water capacity (samples I-1, I-2, I-3, I-4, I-5, I-6 Tulucesti; II-1, II-2, II-3, II-4 Sendreni; III-1, III-2, III-3 Vadeni). Soils of general-clay texture have medium and high field water capacity (samples I-7, I-8, I -9, I-10, I-11, I-12 Tulucesti; II-5, II-6, II-7, II-8 Sendreni; III-4, III-5, III-6 Vadeni), and the soils with general-clay texture have a very high field water capacity (samples III-7, III-8, III-9 Vadeni).

3.2.2. *Wilting coefficient (WC)*

The wilting coefficient, correlated with the soil's colloidal clay content and humus, has values in the order: very low (0.42-2.68% w/w)> medium (4.63-10.39 % w/w)> high (12.66-15.82 % w/w) - Figure 3.3. Thus, for *Tulucesti territory*, WC is very low (0.47-2.01 % w/w) for the coarse-textured soil, low (4.63-7.17 % w/w) - fine sandy loam texture and medium (8.90-

10.39 % w/w) - medium loam and silty loam texture of loamy soils. For *Sendreni territory*, WC is very low, with values of 0.42-2.68 % w/w for coarse and silty-textured soils and medium, with values of 8.79-10.34% w/w, for loamy-textured soils. Soils from *Vadeni* have a very low WC (0.73-1.60 % w/w) for coarse textures, low (7.51-7.80 % w/w), and medium (9.78 % w/w) for loamy-textured soils and high (12.66-15.82 % w/w) for clay soils.

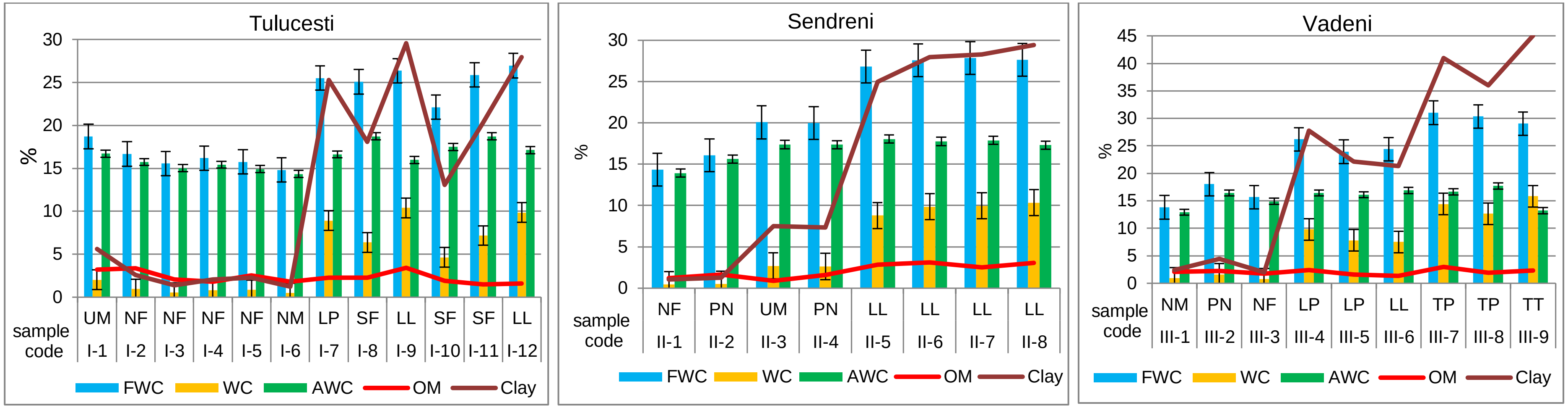
Dumitru, E. et al., 2009 report that, generally, the WC values of coarse-textured soils are in the range of 1.0-5.0 % w/w, while medium-textured soils have values of 5.1-11.0% w/w, and fine-textured soils values of 11.1-18.0 % w/w.

3.2.3. *Available water capacity (AWC)*

Available water capacity, also called potential water reserve [Dumitru E. et al., 2009], of the investigated soils 0-30 cm layer varies depending on the soil clay percentage (<0.002 mm) and volumetric weight as follows: high (14.34-15.98 % w/w) and very high (16.62-18.73% w/w) for *Tulucesti territory*; high (13.92-5.61% w/w) and very high (17.30-18.05% w/w) for *Sendreni area*; high (12.93-14.92% w/w) and very high (16.12-17.70% w / w) for *Vadeni territory* - Figure 3.3.

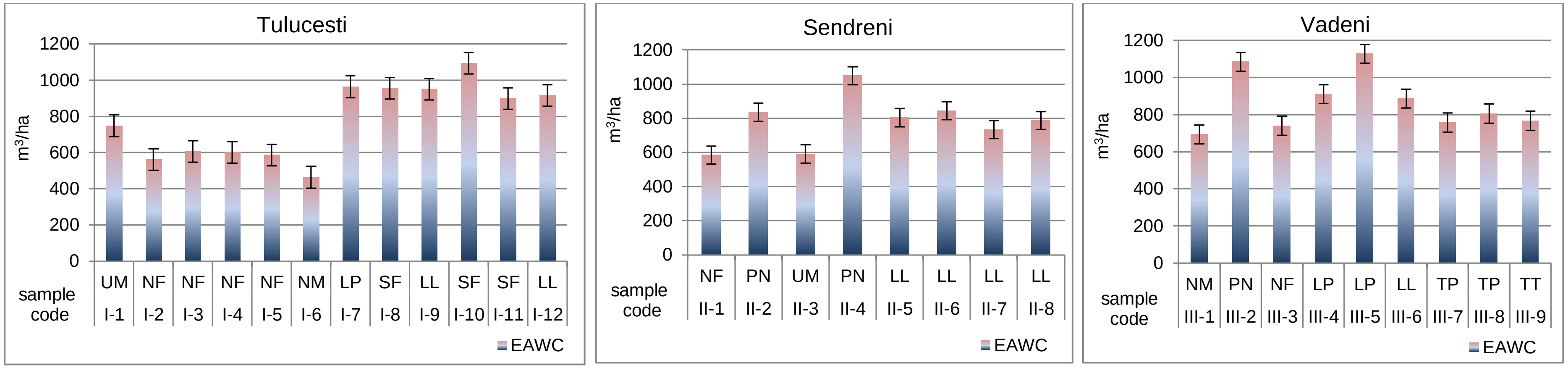
3.2.4. *Easily accessible water capacity (EAWC)*

The analytical data in Figure 3.4 show an increased water storage capacity in the soil as the values are higher and a drought susceptibility to low values of this coefficient. Overall, it is evident that depending on the soil texture, and the bulk density, the easily available water capacity is from low (463.67 m^3/ha) to very high (1093.2 m^3/ha). In the studied territories, the EAWC is: low (463.67 m^3/ha), medium (560.91-605.77 m^3/ha), high (748.15-898.82 m^3/ha) and very high (915.74-1093.72 m^3/ha) for *Tulucesti territory*; medium (584.40-590.88 m^3/ha), high (733.09-843.35 m^3/ha) and very high (1049.78 m^3/ha) for *Sendreni territory*; medium (693.06 m^3/ha), high (740.13-887.02 m^3/ha) and very high (1084.99-1128.38 m^3/ha) for *Vadeni area*.



(a) (b) (c)

Figure 3.3 Field water capacity, wilting coefficient and available water capacity variation in the 0-30 cm layer of the arable lands' soil on the territories: (a) Tulucesti, (b) Sendreni, (c) Vadeni (processed after [Moraru S.-S et al., 2020])

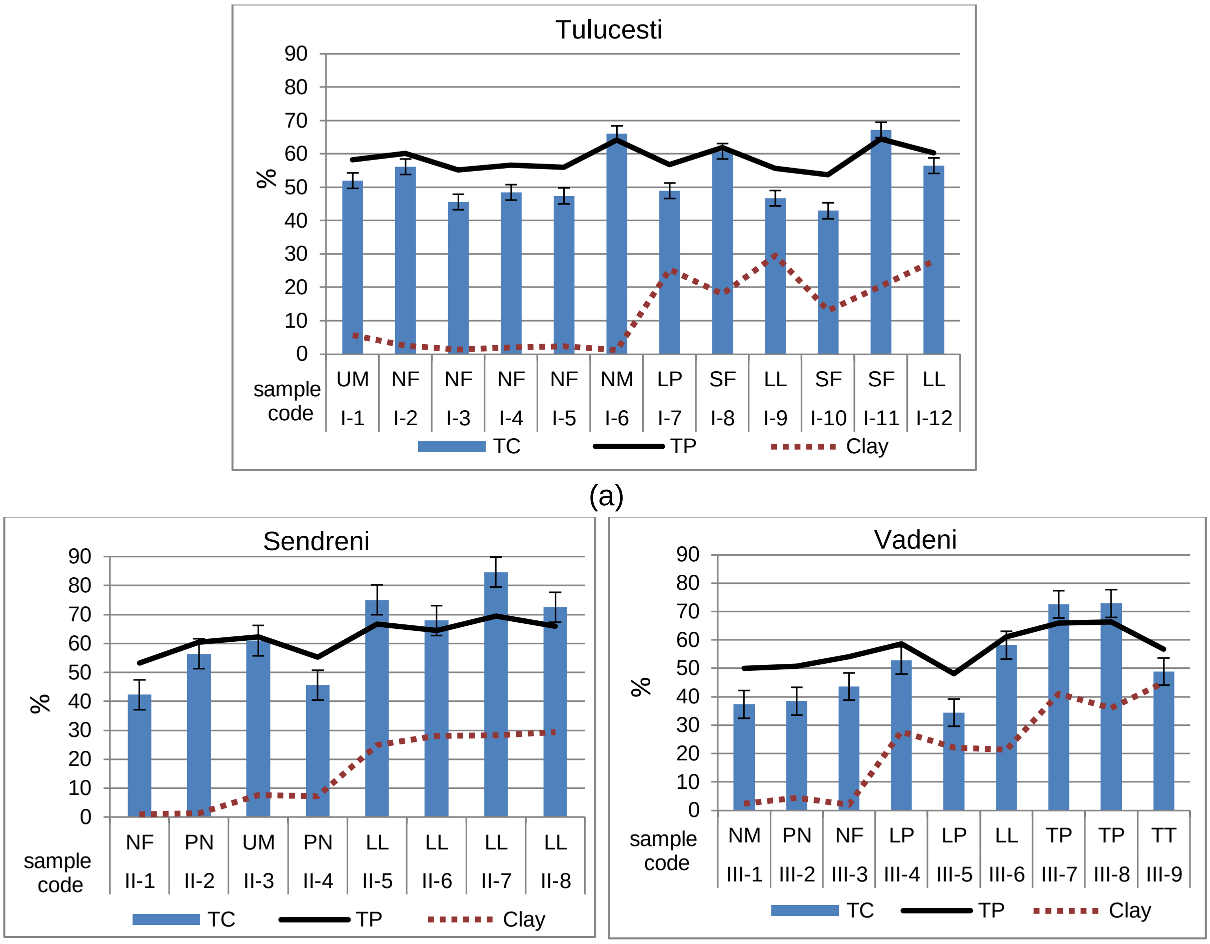


(a) (b) (c)

Figure 3.4 Easily accessible water capacity variation in the 0-30 cm layer of the arable lands' soil on the territories: (a) Tulucesti, (b) Sendreni, (c) Vadeni (processed after [Moraru S.-S. et al., 2020])

3.2.5. *Total capacity of water (TC)*

The total capacity of water in relation to the soil total porosity, clay content (<0.002 mm) and volumetric weight, in the 0-30 cm soil layer, records very high values (42.96-56.42 % w/w) and extremely high (66.06-67.13 % w/w) on *Tulucesti territory*; very high (42.33-56.42 % w/w) and extremely high (61.00-84.64 % w/w) on *Sendreni area*; high (34.39-38.44 % w/w), very high (43.61-58.20 % w/w) and extremely high (72.58-72.85 % w/w) on *Vadeni territory*. Figure 3.5 shows the total water capacity variation.



(b) (c)

Figure 3.5 The total water capacity variation with the total porosity and clay percentage in the 0-30 cm layer of the arable lands' soil on the territories: (a) Tulucesti, (b) Sendreni, (c) Vadeni (processed after [Moraru S.-S et al., 2020])

3.2.6. *Draining capacity (DC)*

The soil draining capacity in the 0-30 cm section has low (10.46 % mm) to very high (23.38-51.25 % mm) values. In the studied territories, this hydro-physical index varies as follows: high (20.35-20.83 % mm) and very high (23.38-51.25 % mm) on *Tulucesti territory*; very high (25.65-56.80 % mm) on *Sendreni area*; low (10.46 % mm), high (19.85-20.41 % mm) and very high (23.49-42.49 % mm) on *Vadeni territory* - Figure 3.6.

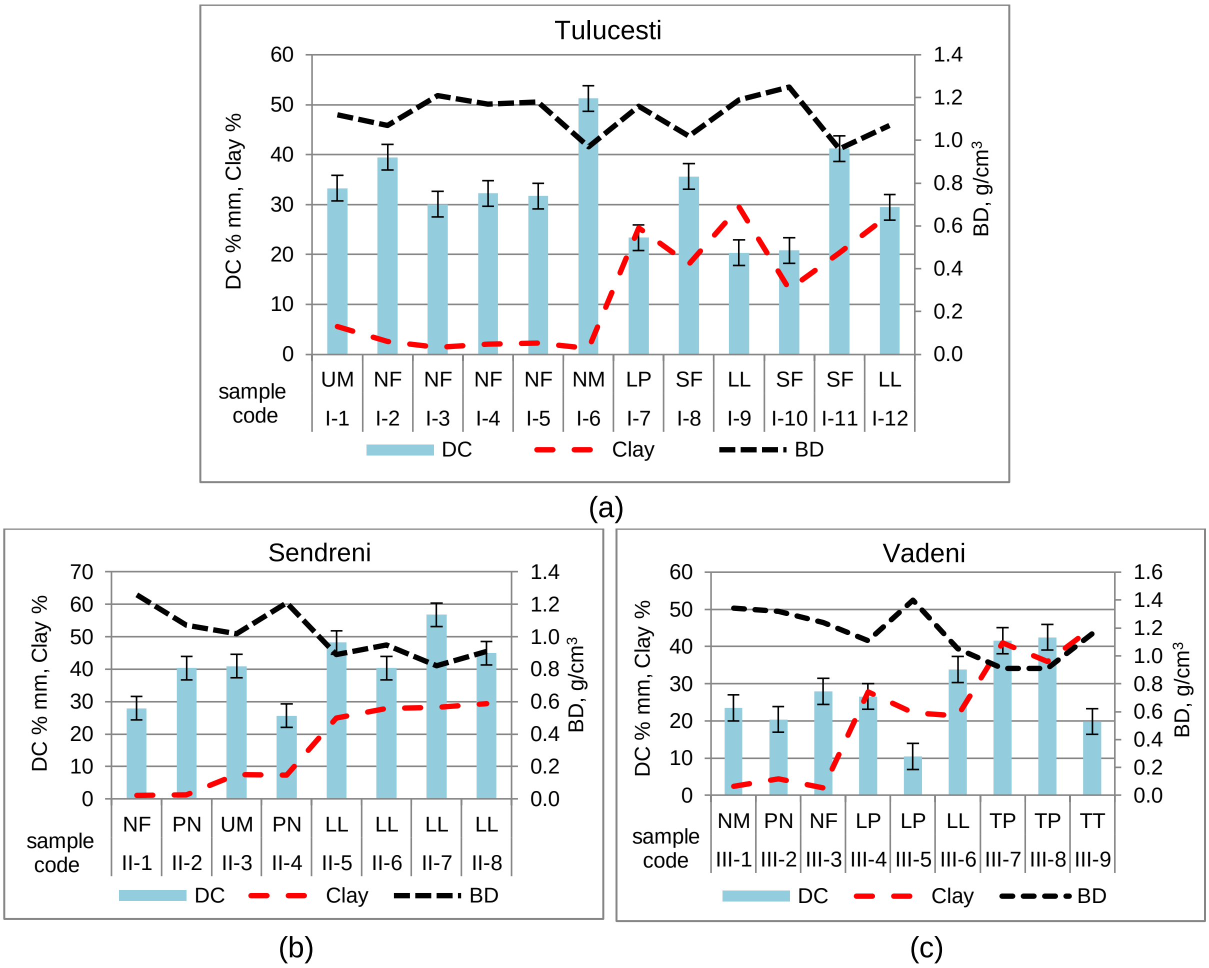


Figure 3.6 The drainage capacity variation with the clay percentage and bulk density in the 0-30 cm layer of the arable lands' soil on the territories: (a) Tulucesti, (b) Sendreni, (c) Vadeni (processed after [Moraru S.-S et al., 2020])

Data of soil's hydro-physical indices are published partly in [Moraru, S.-S. et al., 2020].

3.3. Partial conclusions

Overall, the arable soils of adjacent Galati steel industrial complex lands fall into the coarse (medium sand, fine sand, medium loamy sand), medium (medium loam, sandy silt) and fine (medium clayey loam and clayey silty loam) classes of texture. The volumetric weight in the 0-30 cm section is extremely low and very low due to the specific agricultural works. Total porosity and aeration porosity are inversely proportional to the volumetric weight of the soil. Thus, the total porosity in the 0-30 cm layer is extremely high and very high. The aeration porosity has values that place it in the low, high and very high classes.

The results showed a loosened 0-30 cm agricultural layer because of agrotechnical works. The retention and release of water with nutrients and toxic or potentially toxic elements varies depending on the soil particle size composition and bulk density.

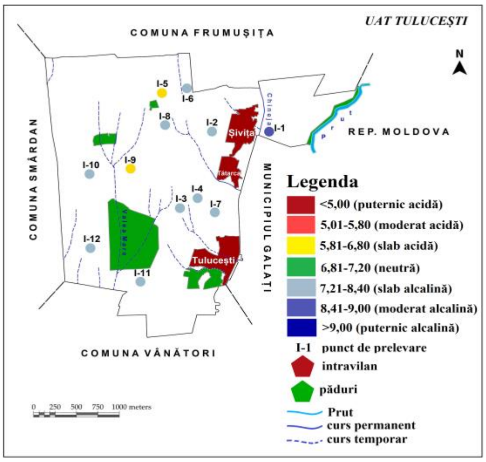
4. RESEARCH ON THE EVALUATION OF AGROCHEMICAL INDICES OF AGRICULTURAL SOILS IN THE AREAS ADJACENT TO THE GALATI STEEL PLANT

4.1. Soil reaction study

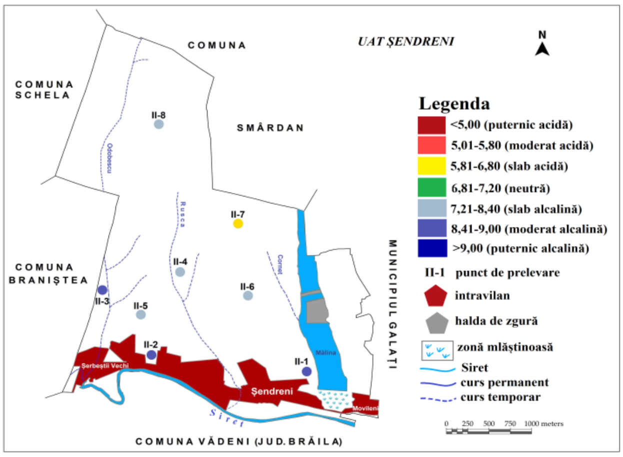
The soil reaction is an essential parameter in the favourable edaphic conditions for the development of the plants. For the studied area, at a depth of 0-30 cm, the soil pH does not vary within wide limits, being on the whole slightly acid-moderately alkaline [Arbanaș (Moraru), S.-S. et al., 2020a, b], [Moraru, S.-S. and Ene, A., 2020]. Figure 4.1 shows the spatial distribution of the reaction classes.

The soils on the territory of Tulucesti have a slightly acid pH (6.35-6.60), slightly alkaline (7.52-8.36) and moderately alkaline (8.42). In the Sendreni area, the soil's pH is slightly acid (6.34), slightly alkaline (7.85-8.02) and moderately alkaline (8.44-8.49). The Vadeni territory soils, developed on alluvial parent materials, are predominantly slightly alkaline (8.06-8.34) and moderately alkaline (8.46).

Overall, due to the same type of parent material (loess and loessoid deposits) and in the same ecological conditions, the reaction of the soils is similar [Arbanaș (Moraru), S.-S. et al., 2020a, b]. The results are comparable to the soil pH values reported by [Arbanaș (Moraru), S.-S. et al., 2018d, e], [Moraru, S. et al., 2017a, b], [Moraru, S.S. et al., 2019b] for arable and pasture lands located in the Covurlui High Plain, Chineja basin.



(a)



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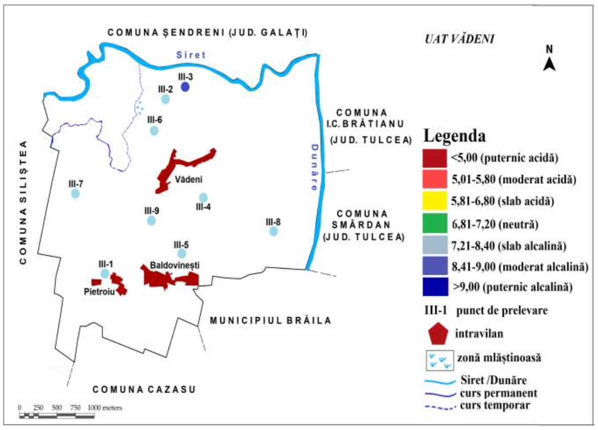


(c)

Figure 4.1 Spatial distribution of reaction classes on the 0-30 cm depth of the arable land: (a) Tulucesti territory, (b) Sendreni territory, (c) Vadeni territory (processed after [Arbanaș (Moraru), S.-S. et al., 2020 a, b], [Moraru, S.-S. and Ene, A., 2020])

4.2. Soil organic carbon and humus concentration study

Organic matter is an essential soil component, which gives it specific characteristics (physical, chemical, biological) and a certain level of fertility. Due to its high water and nutrient retention capacity, organic matter supports sustainable storage in the soil of accessible forms to plants. The humus soil supply assessment depends on particle size composition and the ecological factors in a particular area, which influences the degree of its accumulation in the soil. Figure 4.2 shows the organic matter (OM) and organic carbon (OC) content.

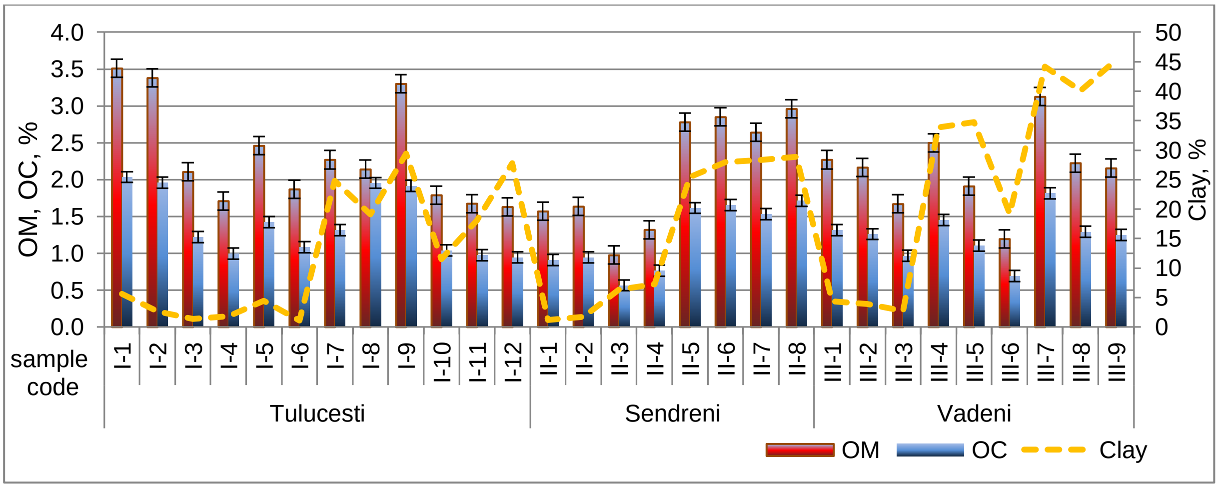


Figure 4.2 Organic matter and organic carbon content variation with the clay percentage in the 0-30 cm layer of the arable lands' soil on the territories of Tulucesti, Sendreni, and Vadeni (processed after [Arbanaș (Moraru), S.-S. et al., 2020 a, b])

Overall, the agricultural 0-30 cm layer is from very low in III-6 sampling point of Vadeni, on a medium loamy texture, to high, in I-2, I-3 sampling points of Tulucesti on a fine sandy texture, and in III-1 sampling point of Vadeni on a medium sandy texture. The agrotechnical works, the cultivated plants, and the slope of the land influence the agricultural soil humus supply.

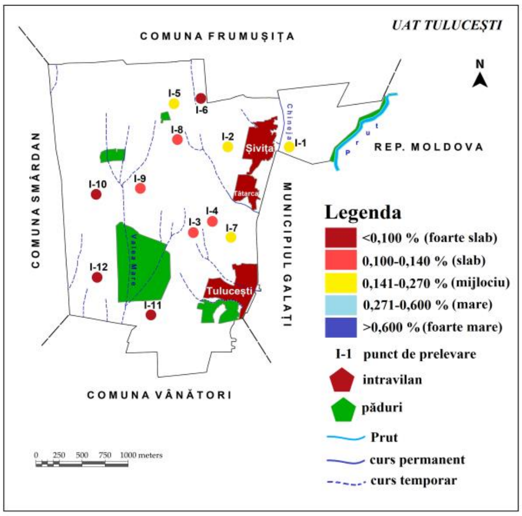
According to [Rusu, M. et al., 2005], the C/N ratio must be kept as low as possible to avoid immobilization of the macronutrients in the soil. The lower this ratio, the higher the soil fertility conditions. At high C levels, the microorganisms responsible for the breakdown of organic matter will consume nitrogen, creating imbalances in plant nutrition. The values of the C/N ratio are 8.05-14.50 % on the *Tulucesti territory*, 6.20-21.95 % in the *Sendreni area* and 7.10-17.00 % on the *Vadeni land*.

4.3. Soil macronutrients concentration study

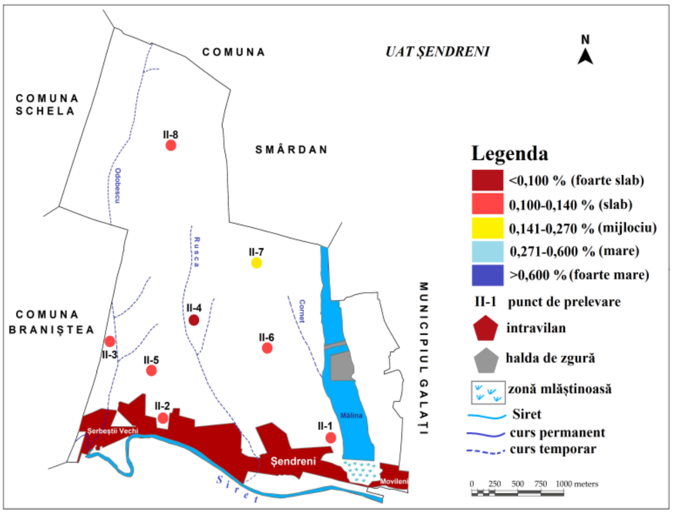
4.3.1. *Nitrogen in soil*

Nitrogen level in the soil can be appreciated by the total Kjeldahl nitrogen (TKN) or by the nitrogen index (NI), calculated based on humus content and degree of base saturation.

Figure 4.3 show the concentration of total Kjeldahl nitrogen in the 0-30 cm layer of the agricultural soils on Tulucesti, Sendreni and Vadeni territories.



(a)



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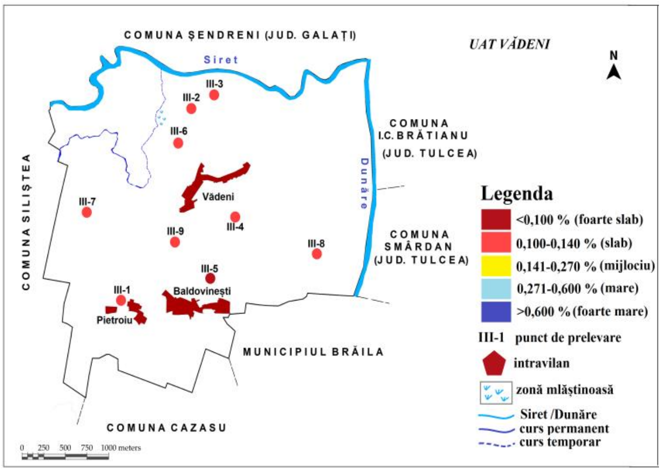
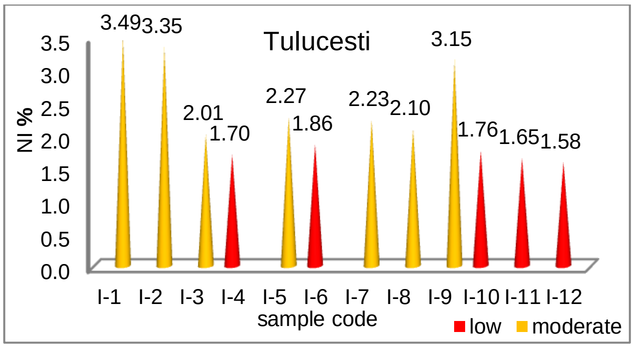


(c)

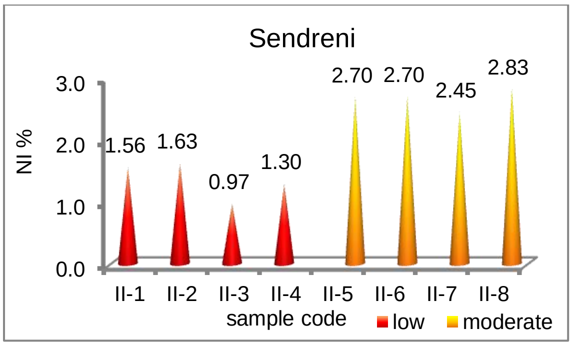
Figure 4.3 Figure 4.3 Total nitrogen content in the arable lands' 0-30 cm layer: (a) Tulucesti territory, (b) Sendreni territory, (c) Vadeni territory (processed after [Arbanaș (Moraru), S.-S. et al., 2020 a, b], [Moraru, S.-S. and Ene, A., 2020])

Overall, the investigated soils have a total nitrogen concentration ranging from very low (0.076-0.098 %) to moderate (0.159-0.175 %) [Arbanaș (Moraru), S.-S. et al., 2020 a, b], [Moraru, S.-S. and Ene, A., 2020]. The results are comparable to the values reported by [Arbanaș (Moraru), S.S. et al., 2018d, e], [Moraru, S. et al., 2017a, b], [Moraru, S.S. et al., 2019b] for arable and pasture lands located in the Covurlui High Plain, Chineja basin.

After IN results, the investigated agricultural soils are from low (0.97-1.87 %) to moderate (2.10-3.49 %) supplied with nitrogen [Arbanaș (Moraru), S.-S. et al., 2020 a, b], [Moraru, S.-S. and Ene, A., 2020]. Figure 4.4 shows the variation of the nitrogen index for each studied territory.



(a)



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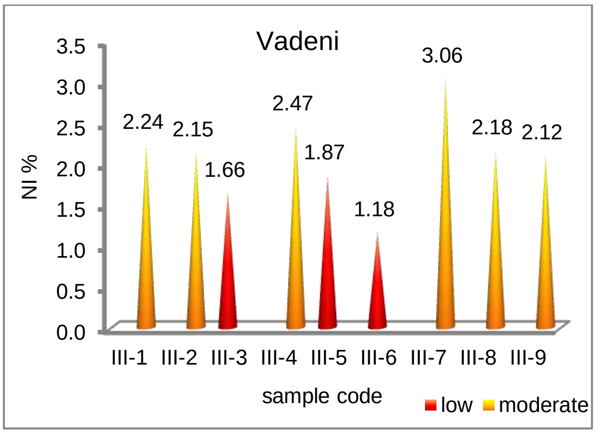


(c)

Figure 4.4 Nitrogen soil supply according to the nitrogen index in the 0-30 cm layer of the arable land soil: (a) Tulucesti, (b) Sendreni, (c) Vadeni [Arbanaș (Moraru), S.-S. et al., 2020 a, b]

It is to mention that the nitrogen supply on the 0-30 cm depth is comparable to the values reported by [OSPA Galați 2014, 2020] and [OSPA Brăila, 2018], both for the total Kjeldahl nitrogen and the nitrogen index in the 0-20 cm agrochemical layer and in the upper horizons of the soils.

The nitrogen content in the 0-30 cm layer of the soils on the Tulucesti, Sendreni and Vadeni territories was presented at the international conferences [Arbanaș (Moraru), S.-S. et al., 2019c], [Arbanaș (Moraru), S.-S. et al., 2020a], [Moraru, S.-S. and Ene, A., 2020] and published in [Arbanaș (Moraru), S.-S. et al., 2020b].

4.3.2. *Mobile phosphorus in soil*

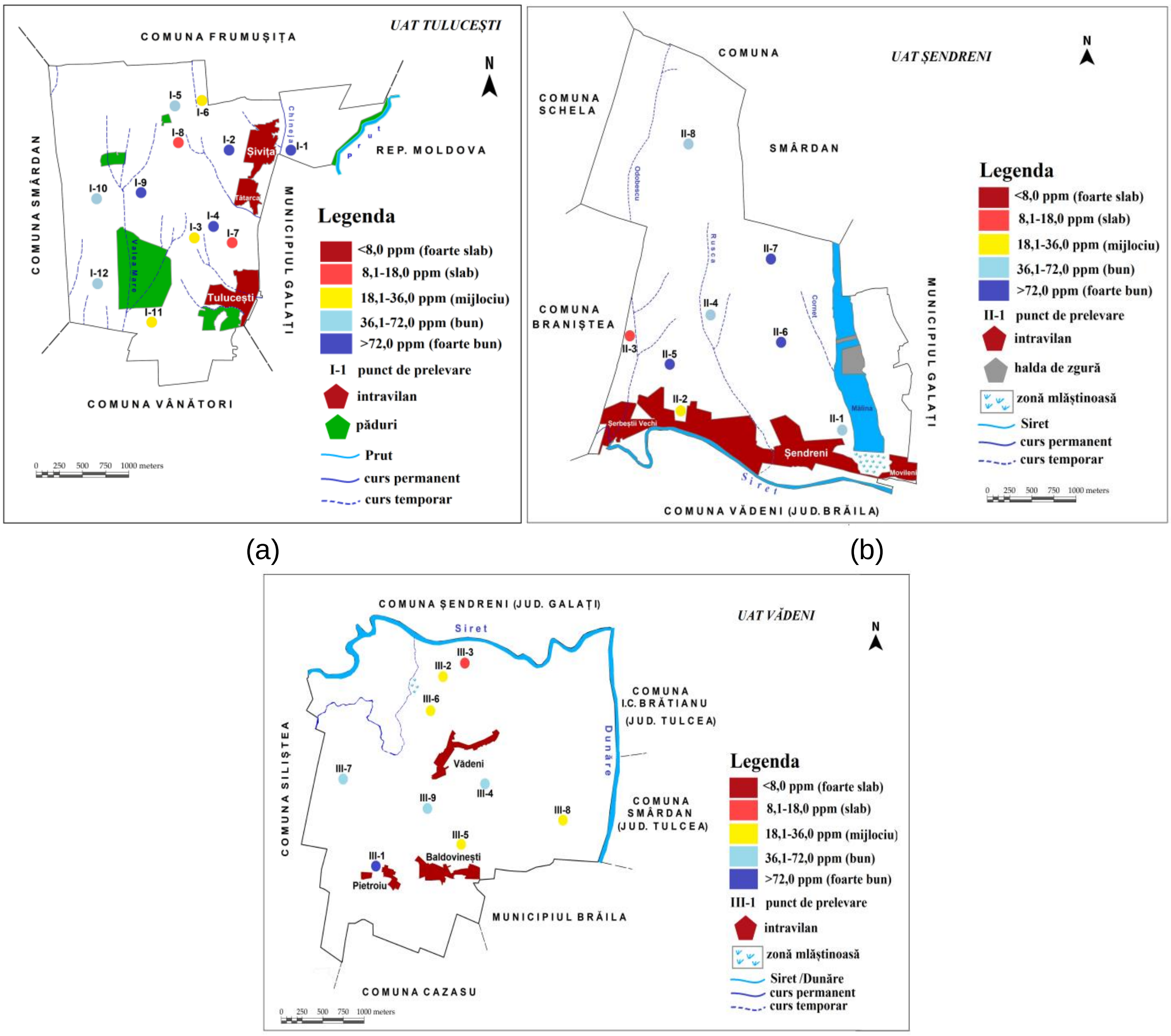
The data in Figure 4.5 show that accessible phosphorus is very low (9.80-15.75 ppm) to very high (80.85-161.75 ppm). The soil supply level of mobile phosphorus is similar to that reported by [OSPA Galați, 2014, 2020], [OSPA Brăila, 2018] and presented by [Arbanaș (Moraru), S.S. et al., 2018d, e], [Moraru, S. et al., 2017a, b], [Moraru, S.S. et al., 2019b].

The distribution of values in the *Tulucesti territory* falls into the following classes: low (11.30-15.75 ppm), medium (24.00-30.75 ppm), high (45.05-55.75 ppm), and very high (87.60-253.35 ppm).

In the *Sendreni area*, the supply of mobile phosphorus falls in the same field of variation. Thus, there is a low (9.80 ppm), moderate (35.80 ppm), high (37.30-49.80 ppm) and very high (80.85-161.75 ppm) supply of mobile phosphorus.

On the *territory of Vadeni* predominates the low (14.85 ppm) - very high (180.30 ppm) supply of mobile phosphorus.

The data regarding the mobile phosphorus content in the 0-30 cm layer of the soil from the Tulucesti, Sendreni and Vadeni were presented at the international conferences [Arbanaș (Moraru), S.-S. et al., 2019], [Arbanaș (Moraru), S.-S. et al., 2020a], [Moraru, S.-S. and Ene, A., 2020] and published in [Arbanaș (Moraru), S.-S. et al., 2020b].



(a) (b)

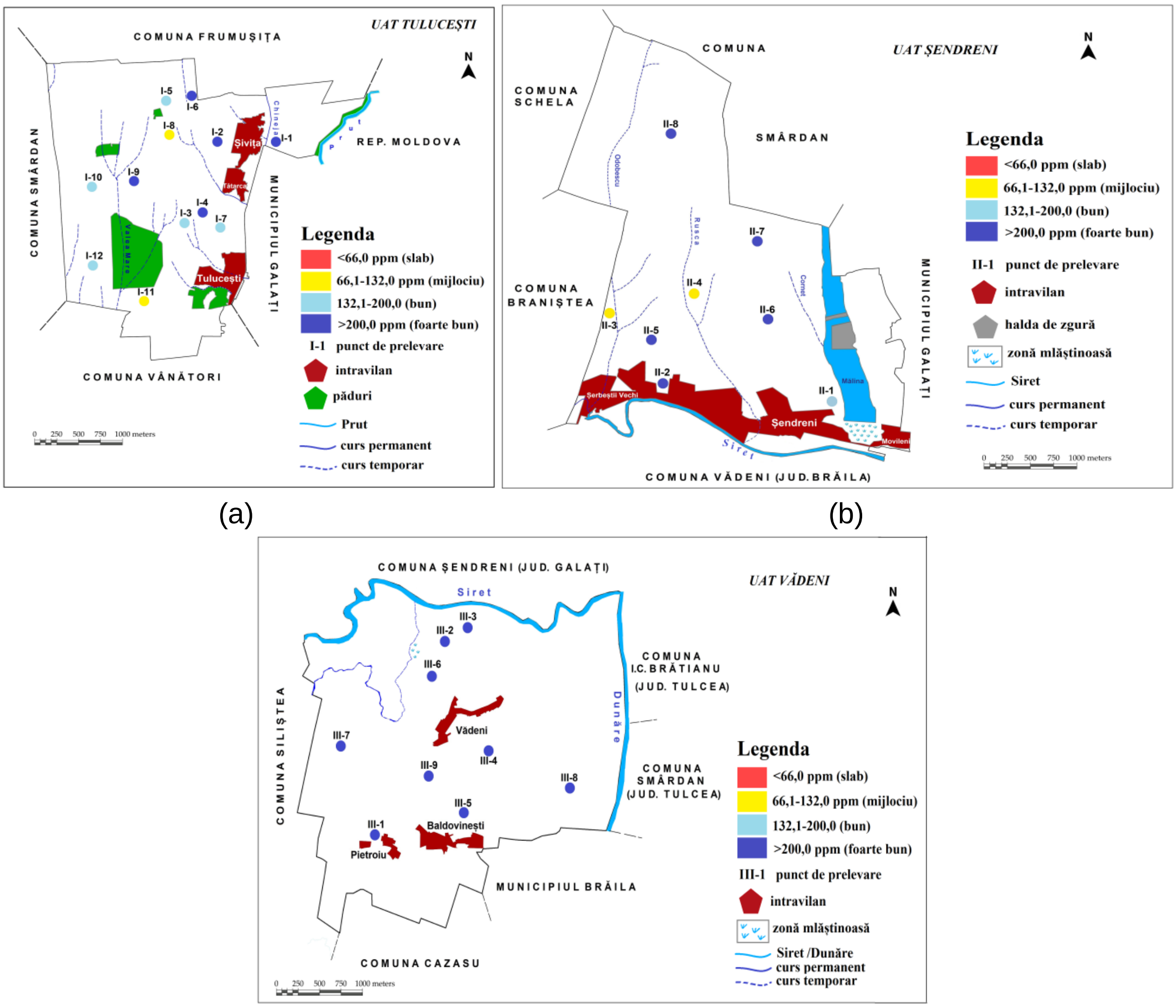
(c)

Figure 4.5 Mobile phosphorus content in the 0-30 cm soil layer of arable land: (a) Tulucesti territory, (b) Sendreni territory, (c) Vadeni territory (processed after [Arbanaș (Moraru), S.-S. et al., 2020 a, b], [Moraru, S.S. and Ene, A., 2020])

4.3.3. *Mobile potassium in soil*

The concentrations of mobile potassium in the 0-30 cm of the agricultural layer of soils adjacent to the steel industrial area of Galati vary from moderate (95-131 ppm) to very high (207-600 ppm) [Arbanaș (Moraru), S.-S. et al., 2020 a, b], [Moraru, S.-S. and Ene, A., 2020]. The level of soil supply with mobile potassium is comparable to that reported by [OSPA Galați, 2014, 2020] and [OSPA Brăila, 2018] for the 0-20 cm agrochemical layer and the upper horizons of the soils and presented by [Arbanaș (Moraru), S.-S. et al., 2018d, e], [Moraru, S. et al., 2017a, b], [Moraru, S.S. et al., 2019b]. Figure 4.6 shows the spatial variation of the level of mobile potassium in the studied agricultural soils.

On the *territory of Tulucesti*, the soils have a moderate (112-131 ppm), high (133-164 ppm) and very high (210-600 ppm) supply. The soils in the *territory of Sendreni* are moderate (95-103 ppm, high (166 ppm) and very high (268-600 ppm) supplied with mobile potassium. In the *Vadeni area*, the soils have a high (170 ppm) and very high (207 -506 ppm) level of mobile potassium.



(a) (b)

(c)

Figure 4.6 Mobile potassium content in the 0-30 cm soil layer of arable land: (a) Tulucesti territory, (b) Sendreni territory, (c) Vadeni territory (processed after [Arbanaș (Moraru), S.-S. et al., 2020 a, b], [Moraru, S.S. and Ene, A., 2020])

The data regarding the mobile potassium content in the 0-30 cm arable layer of the agricultural soils from the territories of Tulucesti, Sendreni and Vadeni were presented at the international conferences [Arbanaș (Moraru), S.-S. et al., 2019c], [Arbanaș (Moraru), S.-S. et al., 2020a], [Moraru, S.-S. and Ene, A., 2020] and published in [Arbanaș (Moraru), S.S. et al., 2020b].

4.7 Partial conclusions

The soil fertility can be assessed by evaluating the principle agrochemical indices. Nitrogen, humus, phosphorus, potassium and soil reaction are the main indicators involved in the optimal development of cultivated plants and maintaining them in balance to obtain economically and nutritionally valuable yields.

The agrochemical mapping of the 0-30 cm agricultural layer related to the soils cultivated with wheat, maize and sunflower in the vicinity of the Galati Iron and Steel Works highlighted the following:

- The analyzed agrochemical indices do not vary within broad limits on the three territories, which shows long exploitation without the sustainable management of the soil;

- From the reaction viewpoint, the soils are generally slightly alkaline. This reaction is a bit higher than the optimum preferred by most field crops.

- Regarding the supply of soil with nutrients, the analytical results showed imbalances. Thus, the nitrogen level is within the very low-low (according to the total Kjeldahl nitrogen) and low-moderate (according to the IN) range. The humus content is low to medium compared to the textural classes of the soil, which induces the soil's susceptibility to erosion. Humus also acts as a binder for structural aggregates.

- The mobile phosphorus content is in the low-very high range, while the mobile potassium level is medium-very high range.

- Due to the imbalances between nutrients, it is necessary to fertilize the soils with mineral and organic fertilizers by the physico-chemical properties, precursor crops, planned crops, and planned harvests.

Data on the content of nutrients in the arable layer 0-30 cm of arable land soils in the territories of Tulucesti, Sendreni and Vadeni were presented at the international conferences [Arbanaș (Moraru), S.-S. et al., 2020a], [Moraru, S.-S. and Ene, A., 2020] and published in [Arbanaș (Moraru), S.-S. et al., 2020b].

5. RESEARCH ON THE CONCENTRATION OF MACRO AND MICROELEMENTS IN SOILS AND AGRICULTURAL PLANTS IN THE AREAS ADJACENT TO THE GALATI STEEL PLANT

5.1. Micro and macroelements concentration in soil

Heavy metals in the soil can be of natural source by acquiring them from the parent material but also anthropogenic. The extraordinary development that the industry has seen in recent decades explains the soil heavy metals pollution, but agriculture (chemical fertilizers, pesticides, plant protection products, irrigation water), road traffic, and mining are also significant sources of contamination. Numerous studies have already put into evidence the impact of toxic elements on the environment and quality of life [Orisakwe, O.E. et al., 2012], [Ali, H. et al., 2019], [Kacholi, D.S. and Sahu, M., 2018], [Tóth, T. et al., 2009], [Krailertrattanachai, N. et al., 2019]. The research continues, given that not all the implications of the toxic elements can have through their combined action. Although some elements are essential for the physiological processes (Zn, Mn, Cr, Cu, Co, Ni, Fe), higher concentrations may cause diseases in the human population or disturbances in the development of plants and animals [Arif, N. et al., 2016], [Kumar, A. et al., 2020], [Nriagu, J., 2011], [Goldhaber, S.B., 2003], [Khan, A. et al., 2015].

Data on the concentration of heavy metals in 0-5 cm and 5-30 cm soil layers in the Tulucesti, Sendreni and Vadeni areas were partially presented at the international conferences [Arbanaș (Moraru), S.-S. et al., 2019c, d], [Ene, A. et al., 2019e], [Moraru, S.S. et al., 2019c] and published in part in [Arbanaș (Moraru), S.-S. et al., 2019e], [Ene, A. et al., 2019f].

5.1.1. *Continuous source high-resolution atomic absorbtion spectrometry (HR-CS AAS) results*

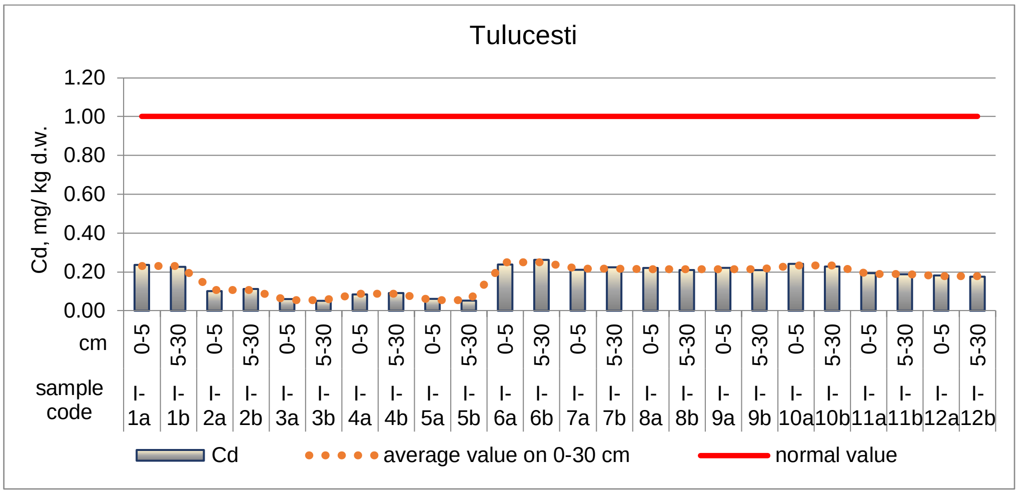
Cadmium in the soil

For the three studied territories, the average concentration of Cd in agricultural soils *did not exceed the normal limits* provided by [Order no. 756/1997] and is below the average value reported at the European and national levels. The level of Cd on the depths of 0-5 cm, 5-30 cm of agricultural soils varies as follows:

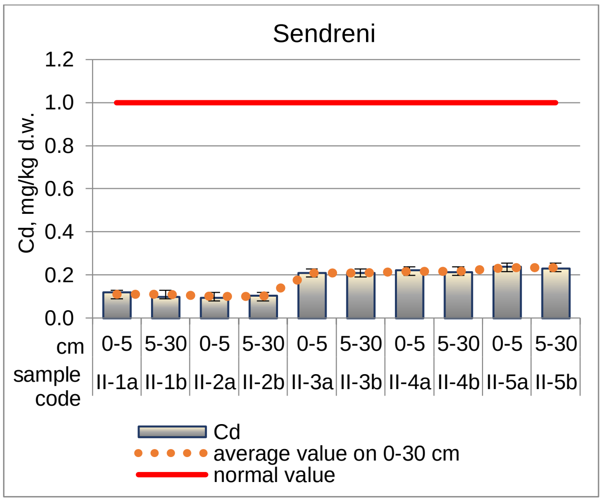
- On the agricultural soils of *Tulucesti territory*, the average Cd content varies in the limits of 0.060 ± 0.002-0.241 ± 0.007 mg/kg d.w., at 0-5 cm depth and the limits of 0.051 ± 0.001-0.262 ± 0.007 mg/kg d.w., at 5-30 cm depth. Figure 5.1a shows the variation of the Cd concentration on the two studied depths.

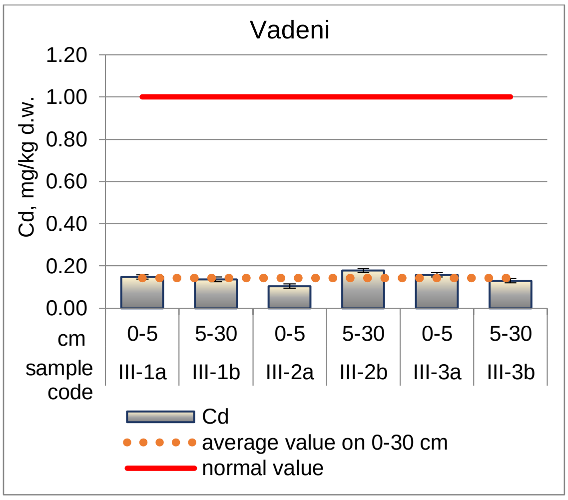
- In the *Sendreni area*, the average Cd content is between 0.142 ± 0.001 and 0.239 ± 0.005 mg / kg d.w., at 0-5 cm depth, and between 0.130 ± 0.002 and 0.231 ± 0.014 mg / kg d.w., at 5-30 cm depth. Figure 5.1b shows the distribution of Cd concentrations over the two depths.

- On the *territory of Vadeni*, the average level of Cd in the soils of the arable lands is between 0.127 ± 0.001 and 0.157 ± 0.003 mg/kg d.w. in the 0-5 cm layer and between 0.108 ± 0.001 and 0.155 ± 0.001 mg/kg d.w. in the 5-30 layer. Figure 5.1c shows the variation of the average Cd content in the 0-5 cm and 5-30 cm layers.



(a)





(b) (c)

Figure 5.1 Cadmium average concentration in the 0-5 cm, 5-30 cm and 0-30 cm layers of the arable lands' soil compared to the normal value: (a) Tulucesti, (b) Sendreni, (c) Vadeni

Cobalt in the soil

The results of Co concentration in the agricultural layer of the soils on the Tulucesti, Sendreni and Vadeni territories showed that the level of this element *does not exceed the normal threshold* in [Order no. 756/1997]. In the studied area is no significant variation between the two depths, the values being below the national average value. Figure 5.2 illustrate the element dynamics at the standard depths of 0-5 cm and 5-30 cm.

In the agricultural soil belonging to the three territories, the average Co content varies as follows:

- On *Tulucesti territory*, the Co concentration varies between 2,873 ± 0.451 and 11,060 ± 0.150 mg/kg d.w., on the 0-5 cm depth and between 5.380 ± 0.111 and 11.702 ± 0.256 mg/kg d.w., in the 5-30 cm layer.
- On *Sendreni territory*, the Co values are of 5.141 ± 0.715-8.833 ± 0.141 mg/kg d.w., on the depth of 0-5 cm, and of 5.537 ± 0.082 - 8.811 ± 0.162 mg/kg d.w., on the depth of 5-30 cm.
- On *Vadeni territory*, the Co content is between 5.289 ± 0.123-11.190 ± 0.225 mg/kg d.w., on the depth 0-5 cm, and between 4.876 ± 0.123-10.060 ± 0.225 mg/kg d.w., on the depth 5-30 cm.

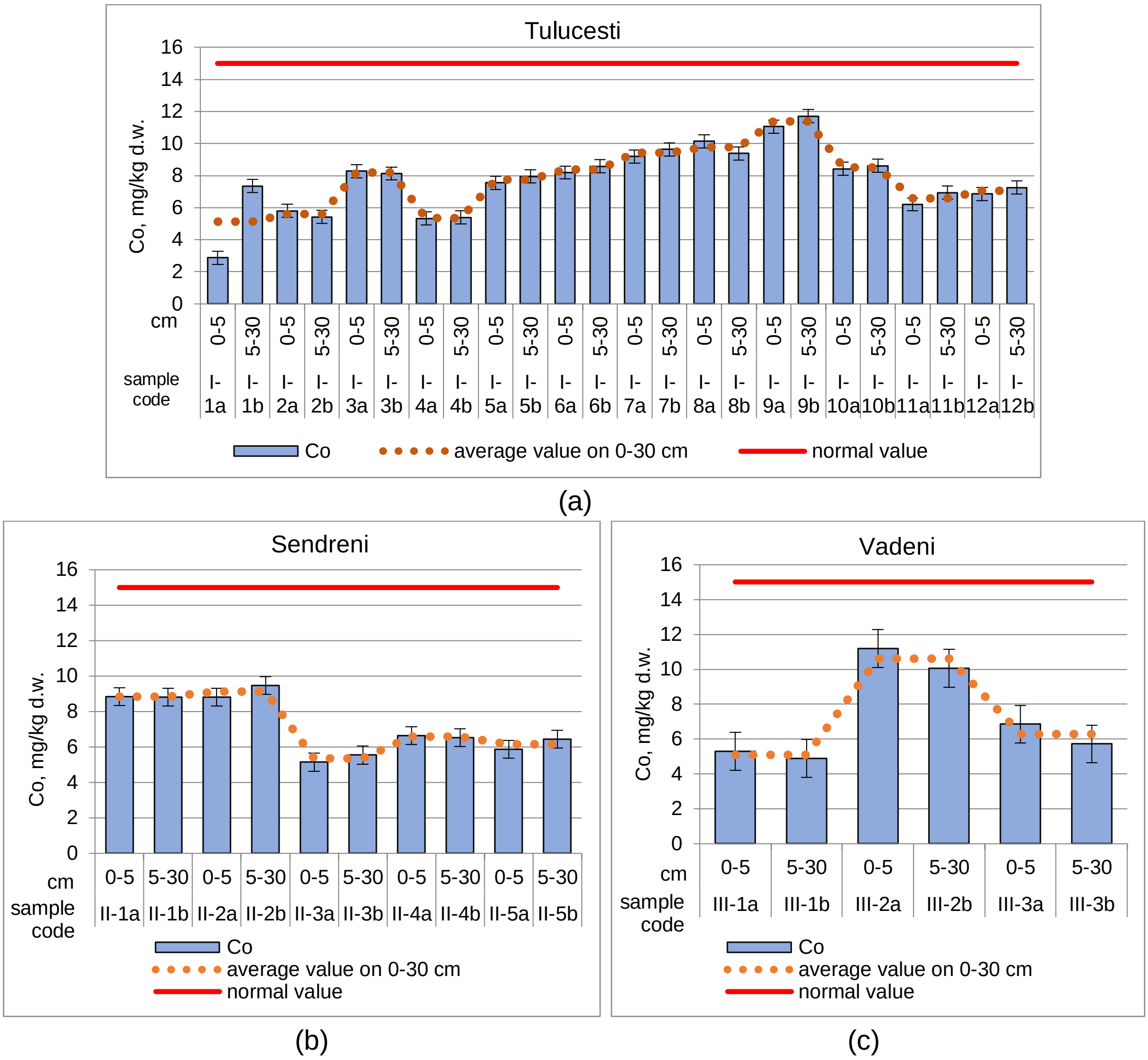
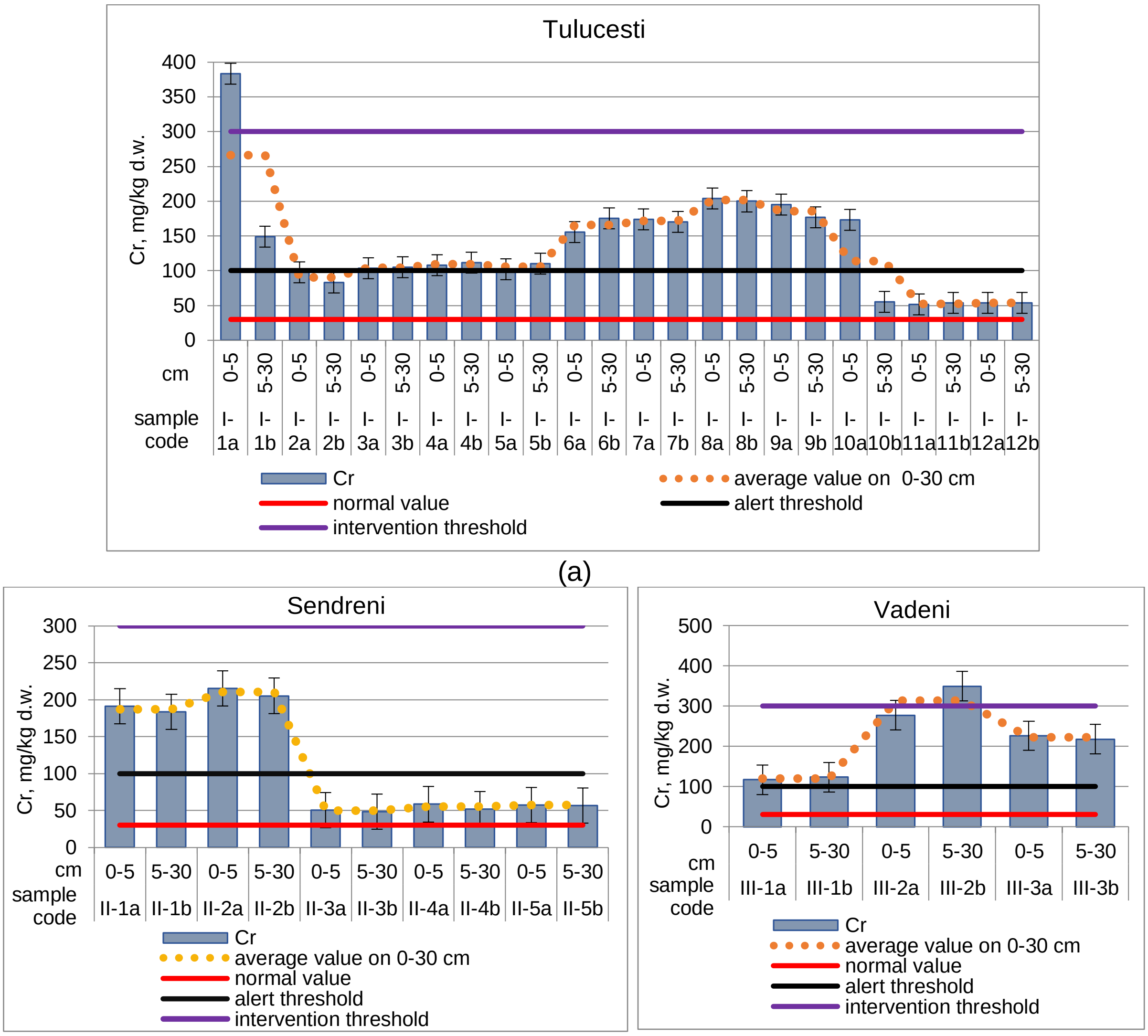


Figure 5.2 Cobalt average concentration in the 0-5 cm, 5-30 cm and 0-30 cm layers of the arable lands' soil compared to the normal value: (a) Tulucesti, (b) Sendreni, (c) Vadeni

Chromium in the soil

Compared to other metals investigated in this paper, Cr has **the highest values, exceeding the normal limits** (30 mg/kg d.w.). For the studied area, the concentration of this element varies from 48,620 ± 0.831 mg/kg d.w., in the north of Serbestii Vechi village, Sendreni commune, to 383,100 ± 5,378 mg/kg d.w., in Prut meadow, east of Sivita village, Tulucesti commune. Figure 5.3. shows the chromium variation in the three studied territories.



(a)

(b) (c)

Figure 5.3 Chromium average concentration in the 0-5 cm, 5-30 cm and 0-30 cm layers of the arable lands' soil compared to the normal value, alert threshold and intervention threshold: (a) Tulucesti, (b) Sendreni, (c) Vadeni

In the agricultural layer, the level of Cr varies as follows:

- In *Tulucesti*, **66.67%** of the analyzed samples **exceed the alert threshold for sensitive uses, which is a potentially significant level of pollution**, 29.16% are between normal values and the alert threshold for sensitive uses, and **4.17 %** of samples **exceed the intervention threshold for sensitive uses, which is significant pollution**. At a depth of 0-5 cm, the concentration of Cr in agricultural soils varies from 51,300 ± 0.320 mg/kg d.w. to 383,100 ± 5,378 mg/kg d.w., and in the 5-30 cm layer, from 53,590 ± 1,293 mg/kg d.w. to 199,900 ± 1,988 mg/kg d.w.
- On the *territory of Sendreni*, the Cr concentration, both in the 0-5 cm and in the 5-30 cm layers, **exceeds the normal value and the alert threshold for sensitive uses (potentially significant pollution)**. Thus, for the 0-5 cm layer, the Cr concentration is within the limits of 50.210 ± 0.782-215.400 ± 5.758 mg/kg d.w., and for the 5-30 cm layer, it is between 48.620 ± 0.831-205.300 ± 6.398 d.w.
- On the *territory of Vadeni*, the Cr content in both layers **exceeds the alert threshold for sensitive use (potentially significant pollution)**. At the III-2b (5-30 cm)

sampling point, **the Cr concentration exceeds the intervention threshold for sensitive uses (significant pollution)**, with a value of 349,600 mg/kg d.w. In the 0-5 cm layer the values are between 117,100 ± 1,039-277,400 ± 6,144 mg/kg d.w., and in the 5-30 cm layer, between 123,400 ± 2,345-344,600 ± 5,840 mg/kg d.w.

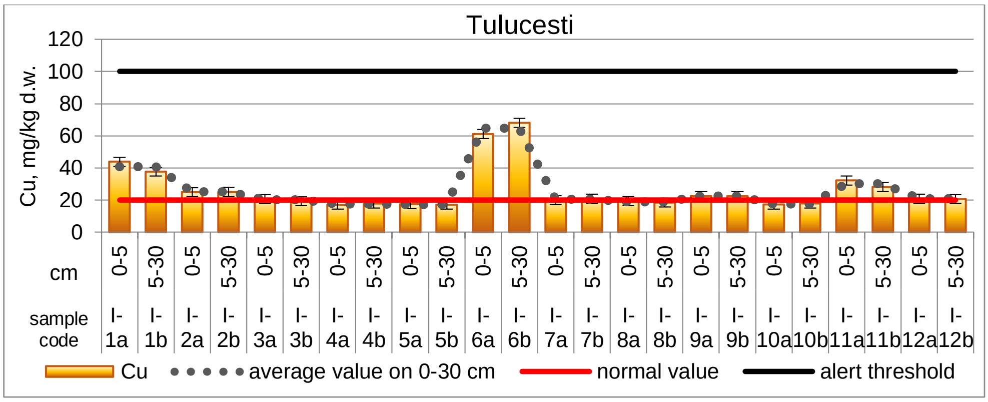
Cooper in the soil

Figure 5.4 illustrates the average content of Cu in the agricultural layer of soils in the Tulucesti, Sendreni and Vadeni areas. The concentration of this element shows variation, in some areas exceeding the normal limit provided by the legislation in force yet doesn't reach the alert threshold for sensitive (agricultural) uses. The analytical results for the three territories highlighted the following:

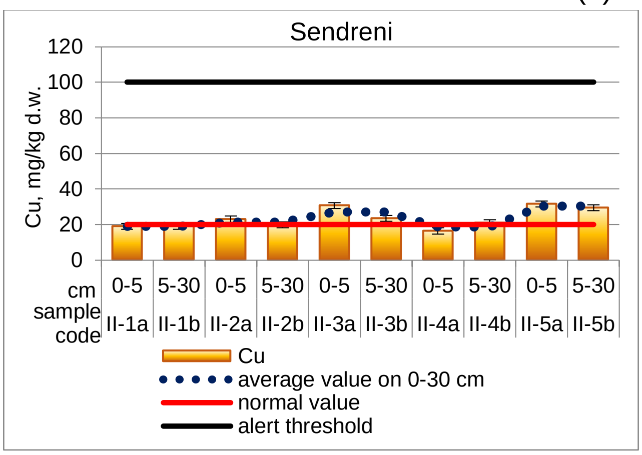
• On the *territory of Tulucesti*, **the Cu concentration exceeds the normal value of 20 mg/kg d.w.** for 62.50% of the analyzed samples. The average concentration in the 0-5 cm layer is 26.507 ± 0.175 mg/kg d.w., and for 5-30 cm, 26.162 ± 0.200 mg/kg d.w.

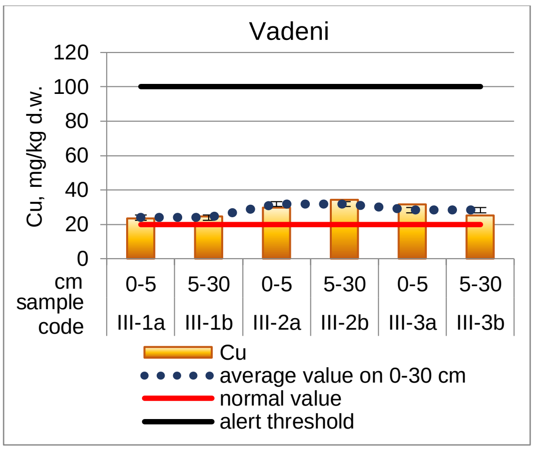
• In the *territory of Sendreni*, **the Cu concentration exceeds the normal value for 60% of the samples.** The average value on the depth of 0-5 cm is 24.107 ± 0.175 mg/kg d.w., and for the depth 5-30 cm, 22.520 ± 0.175 mg/kg d.w.

• On the *territory of Vadeni*, **all the analyzed samples exceed the normal value**. Thus, in the 0-5 cm layer, the Cu concentration is 23.350 ± 0.495-31.510 ± 0.256 mg/kg d.w., while in the 5-30 cm layer, it records values of 24.510 ± 0.168-34.180 ± 0.217 mg/kg d.w.



(a)





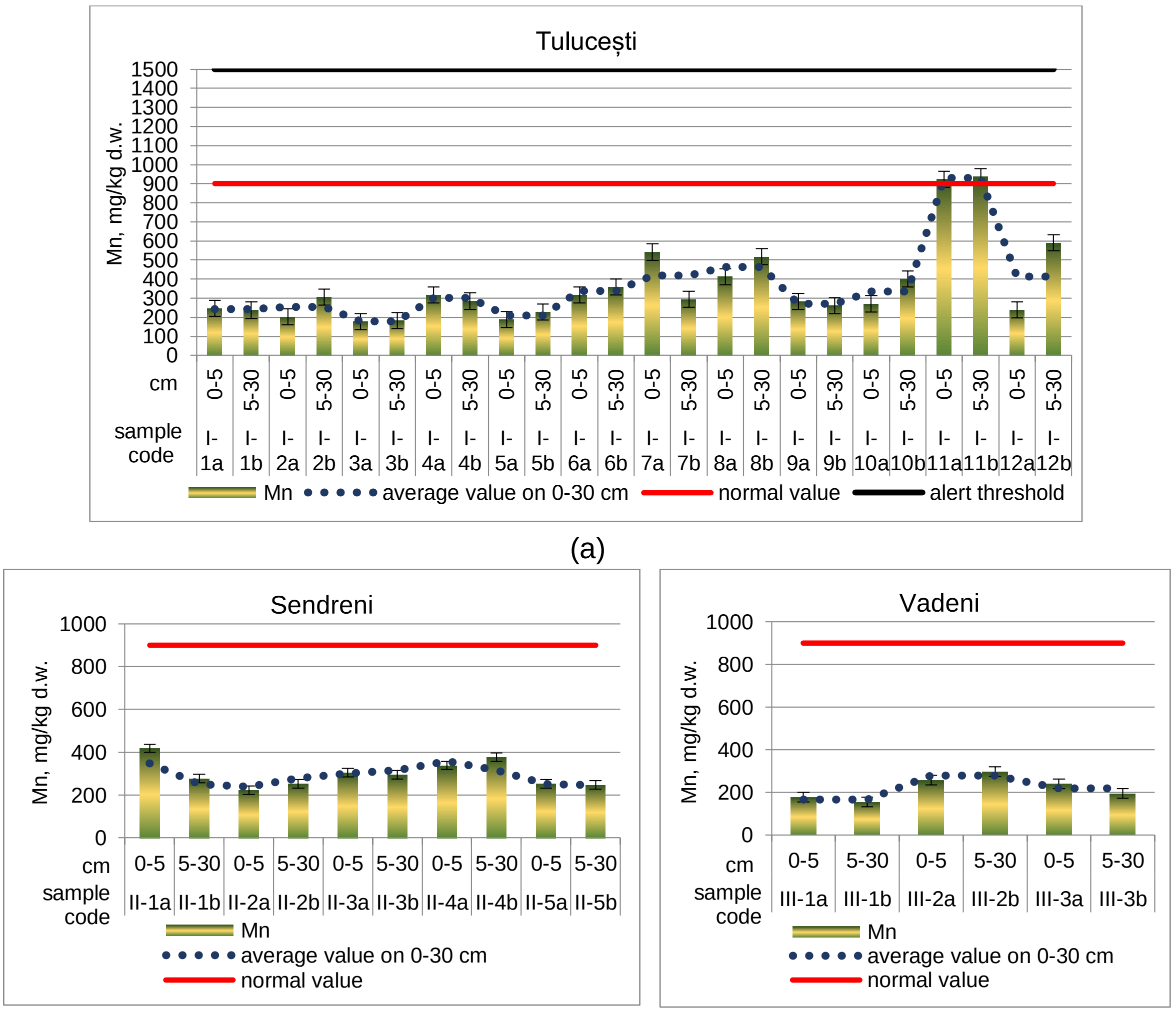
(b) (c)

Figure 5.4 Copper average concentration in the 0-5 cm, 5-30 cm and 0-30 cm layers of the arable lands' soil compared to the normal value and alert threshold: (a) Tulucesti, (b) Sendreni, (c) Vadeni

Manganese in soil

Generally, *the average concentration of Mn* in the arable soils from the Tulucesti, Sendreni and Vadeni communes *falls within the normal limit* established by [Order no. 756/1997]. Figure 5.5 shows the dynamics of Mn content in the three territories. Regarding the distribution and variation of Mn concentrations in each area, the situation is as follows:

- On the *territory of Tulucesti*, the average content of Mn in the agricultural layer varies from 177,045 ± 3,869 to 924,244 ± 14,760 mg/kg d.w., on the depth 0-5 cm, and from 182,245 ± 2,313 to 938,145 ± 12,860 mg/kg d.w., in the layer 5-30 cm.
- On the *territory of Sendreni*, the average concentration of Mn does not exceed the normal limit in the Romanian legislation. In agricultural soil, the Mn level is between 223,446 ± 5,828 and 417,846 ± 9,798 mg/kg d.w., on the depth of 0-5 cm, and between 247,045 ± 4,233 and 377,645 ± 3,334 mg/kg d.w., on a depth of 5-30 cm.
- On the *territory of Vadeni*, the Mn average values are similar to the territories of Tulucesti and Sendreni. The results for this element are below the normal value in the legislation. In the 0-5 cm layer, the Mn content varies within the limits of 176,146 ± 2,548-256,646 ± 11,610 mg/kg d.w., and between 153,845 ± 2,848 and 297,745 ± 4,852 mg/kg d.w., in the 5-30 cm layer.



(a)

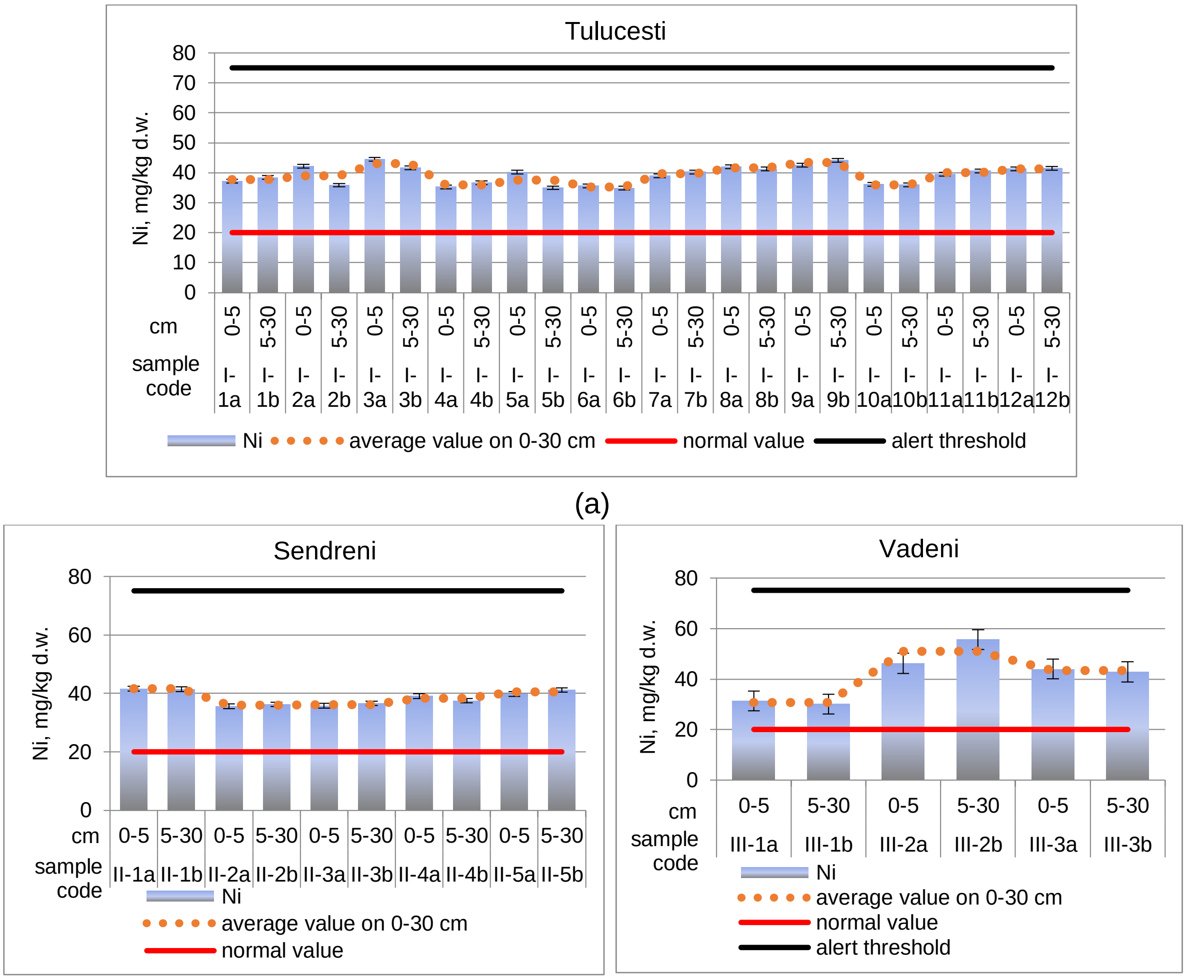
(b) (c)

Figure 5.5 Manganese average concentration in the 0-5 cm, 5-30 cm and 0-30 cm layers of the arable lands' soil compared to the normal value and alert threshold: (a) Tulucesti, (b) Sendreni, (c) Vadeni

Nickel in the soil

In the researched agricultural soils, **the average concentration of Ni exceeds the normal values for all the samples**. Figure 5.6 shows the dynamics of the Ni level on the two considered depths. In the studied territories, the nickel concentration presents the following situation:

- In the *Tulucesti area*, **the Ni content exceeds 1.7 to 2 times the normal value of 20 mg/kg d.w.**, **yet doesn't reach the alert threshold for sensitive uses**. In the 0-5 cm layer the concentration is between 35.265 ± 0.776-44.455 ± 0.356 mg/kg d.w.
- On the **territory of Sendreni**, **the Ni content is similar to that recorded for agricultural soils in Tulucesti**. The depth variation of Ni is as follows: for 0-5 cm, Ni is within the limits of 35.541 ± 0.569-41.570 ± 0.291 mg/kg d.w., and for 5-30 cm, it is within 36.199 ± 0.833-41.446 ± 0.333 mg/kg d.w.
- On the *territory of Vadeni*, **the concentration of Ni in the agricultural layer is slightly higher than in the other studied territories, exceeding 1.6-2.8 times the normal value**. **It doesn't reach the alert threshold for sensitive (agricultural) uses.** The Ni content registers values of 31.338 ± 0.094-46.197 ± 0.416 mg/kg d.w., in the 0-5 cm layer, and values of 30.157 ± 0.633-55.656 ± 0.167 mg/kg d.w., in the 5-30 cm layer.



(a) (b) (c)

Figure 5.6 Nickel average concentration in the 0-5 cm, 5-30 cm and 0-30 cm layers of the arable lands' soil compared to the normal value and alert threshold: (a) Tulucesti, (b) Sendreni, (c) Vadeni

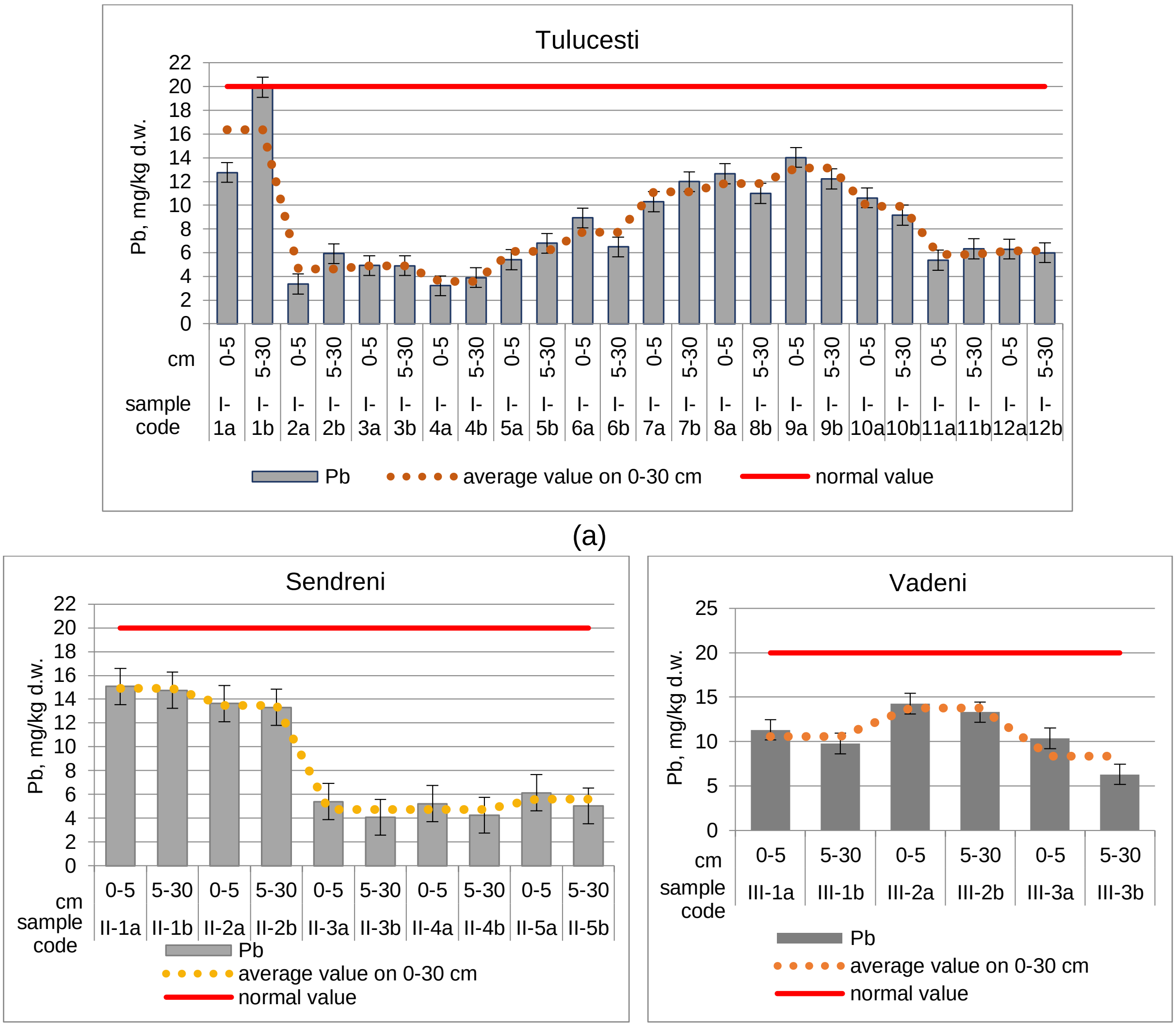
Lead in soil

The concentration of Pb on the territories of Tulucesti, Sendreni and Vadeni *is within normal limits*, without significant variations by soil layers. Figure 5.7 presents the Pb concentration on the two standard depths. For each area, the situation of lead in the upper layer of agricultural soils is as follows:

• On the *territory of Tulucesti*, the average content of Pb, on the 0-5 cm depth, is within the limits of 3.207±0.128-14.021±0.112 mg/kg d.w., and on the 5-30 cm depth, between 3.902 ± 0.382-19.940 ± 1.314 mg/kg d.w.

• On the *territory of Sendreni*, the lead registers average values of 5.216±0.125-15.070±0.255 mg/kg d.w., in the 0-5 cm layer, and values of 4.075±0.192-14.760±0.503 mg/kg d.w., in the 5-30 cm layer.

• On the *territory of Vadeni*, the average Pb content is within the limits of 10.370±0.767-14.250±0.354 mg/kg d.w., in the 0-5 cm layer, and 6.308±0.432-13.310±0.653 mg/kg d.w., in the 5-30 cm layer.



(a) (b) (c)

Figure 5.7 Lead average concentration in the 0-5 cm, 5-30 cm and 0-30 cm layers of the arable lands' soil compared to the normal value: (a) Tulucesti, (b) Sendreni, (c) Vadeni

Zinc in soil

Generally, *the average Zn content* in the investigated agricultural soils *is within normal limits*, except for a few areas on the territories of Tulucesti and Vadeni, where the concentration exceeds the normal limit of 100 mg/kg d.w. Figure 5.8 shows the variation of the average Zn content in the upper layers of agricultural soils in the studied territories. The situation of the soil's average level of Zn is the following:

- On the *territory of Tulucesti*, **the average Zn content is higher than the normal value, but it does not reach the alert threshold for sensitive uses.** Overall, the average Zn content is 60,395 ± 1.751-136.586 ± 1.229 mg/kg d.w., in the 0-5 cm layer, and 65.145 ± 0.847-122.690 ± 2.252 mg/kg d.w., at a depth of 5-30 cm.
- On the *territory of Sendreni*, *the average concentration of Zn is within normal limits*. In the 0-5 cm layer, the average Zn level is in the range of 69.370 ± 2.034-99.590 ± 0.571 mg/kg d.w., and in the 5-30 cm layer, it is 63.306 ± 1.805- 92.579 ± 0.898 mg/kg s.u.
- On the *territory of Vadeni*, the average Zn content is similar to the other two locations, registering values of 73.098 ± 0.333-102.587 ± 0.718 mg/kg d.w. in the 0-5 cm layer and of 73.457 ± 0.850-103.866 ± 0.325 mg/kg d.w. in the 5-30 cm layer.

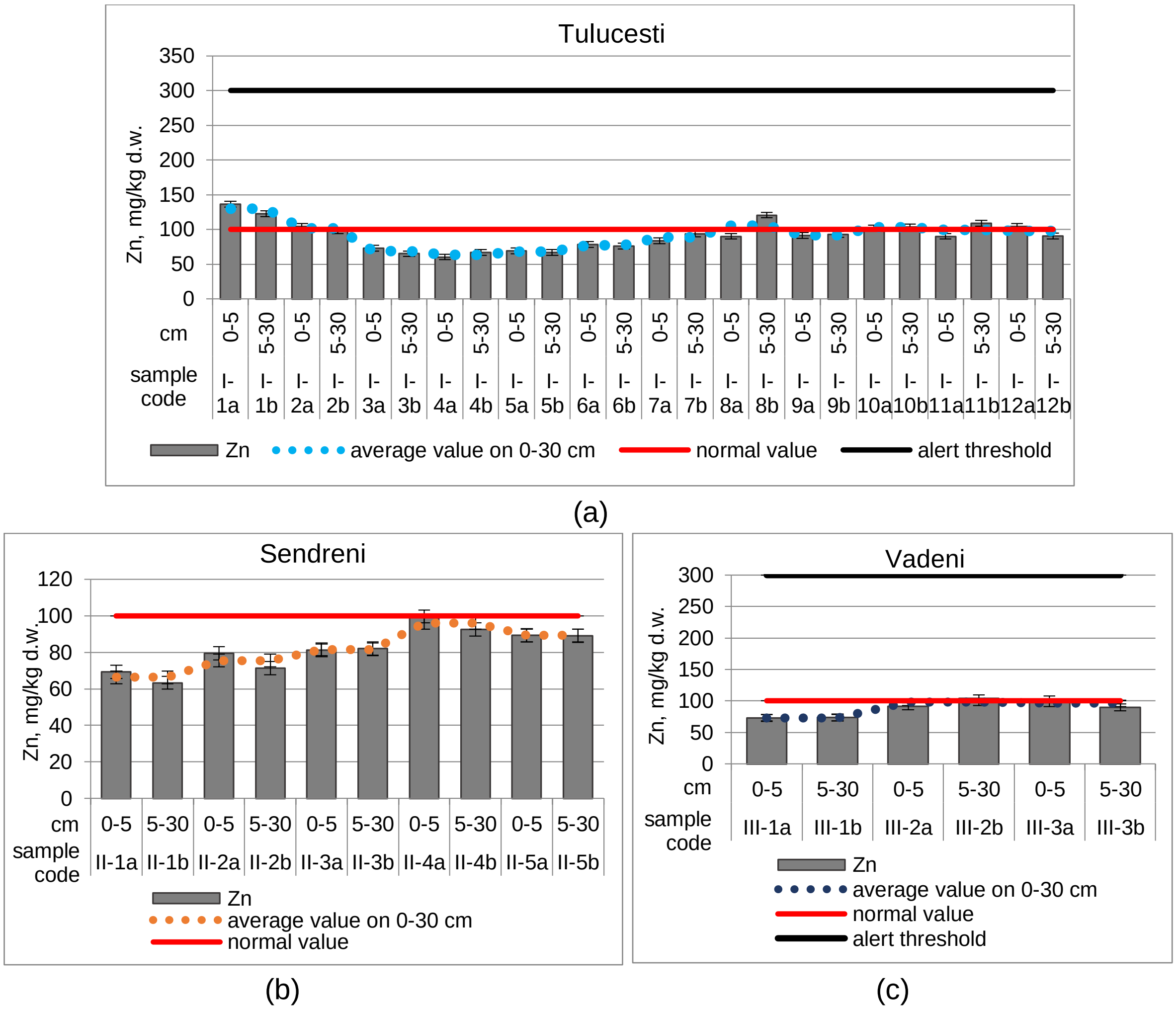


Figure 5.8 Zinc average concentration in the 0-5 cm, 5-30 cm and 0-30 cm layers of the arable lands' soil compared to the normal value and alert threshold: (a) Tulucesti, (b) Sendreni, (c) Vadeni

5.1.2. *Inductively coupled plasma mass spectrometry (ICP-MS) results*

The ICP-MS technique was employed to determine the stable isotope levels of metals in the selected environmental samples and assess the anthropogenic impact on agricultural soils near the steel industry complex. The following stable metal isotopes have been identified: ^{110}Cd, ^{111}Cd, ^{112}Cd, ^{113}Cd, ^{114}Cd, ^{59}Co, ^{52}Cr, ^{63}Cu, ^{65}Cu, ^{54}Fe, ^{56}Fe, ^{55}Mn, ^{58}Ni, ^{60}Ni, ^{206}Pb, ^{207}Pb, ^{208}Pb, ^{64}Zn, ^{66}Zn and ^{68}Zn.

Figures 5.9, 5.10, and 5.11 show the variation of the average concentrations of stable isotopes in the selected agricultural soils from the Tulucesti, Sendreni and Vadeni areas located in the vicinity of the Galati Iron and Steel Works.

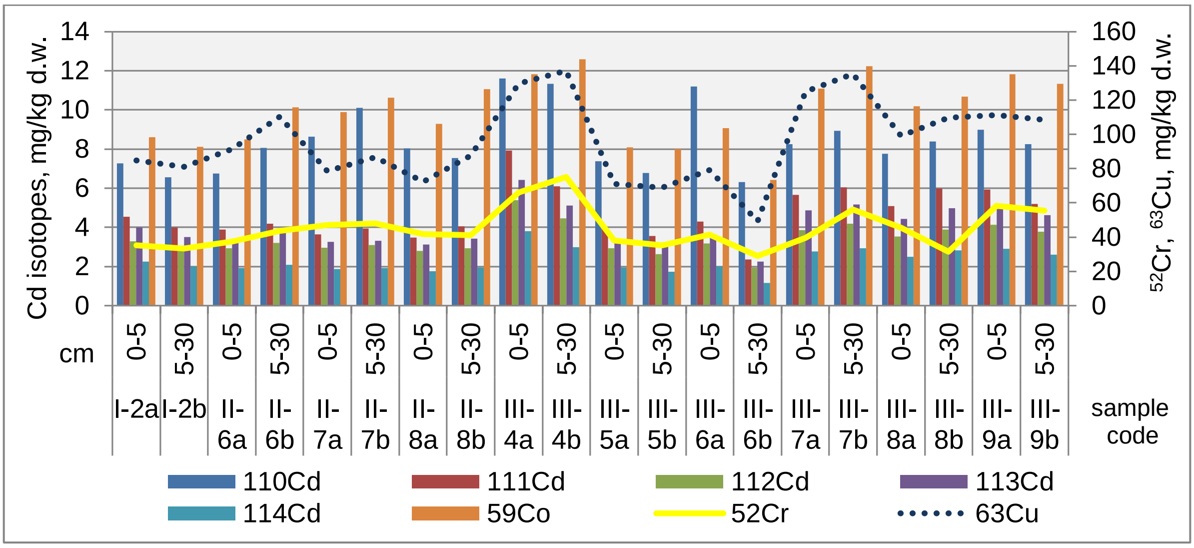


Figure 5.9 ^{110}Cd, ^{111}Cd, ^{112}Cd, ^{113}Cd, ^{114}Cd, ^{59}Co, ^{52}Cr și ^{63}Cu average concentration in 0-5 cm și 5-30 cm layer of the arable lands' soil from Tulucesti, Sendreni, Galati county and Vadeni, Braila county

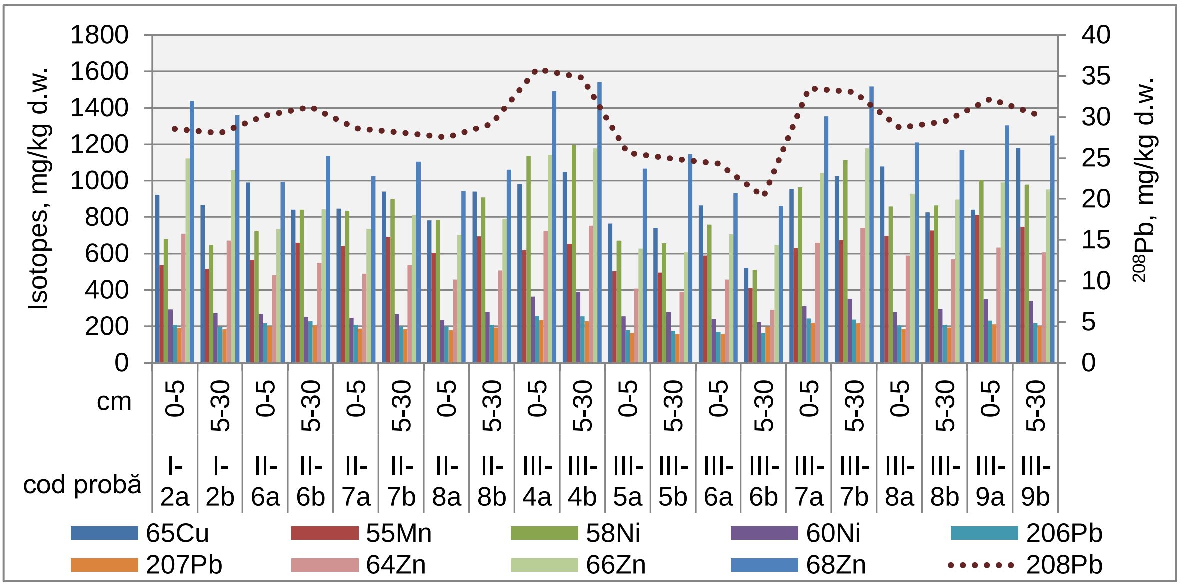


Figure 5.10 ^{65}Cu, ^{55}Mn, ^{58}Ni, ^{60}Ni, ^{206}Pb, ^{207}Pb, ^{208}Pb, ^{64}Zn, ^{66}Zn și ^{68}Zn average concentration in 0-5 cm și 5-30 cm layer of the arable lands' soil from Tulucesti, Sendreni, Galati county and Vadeni, Braila county

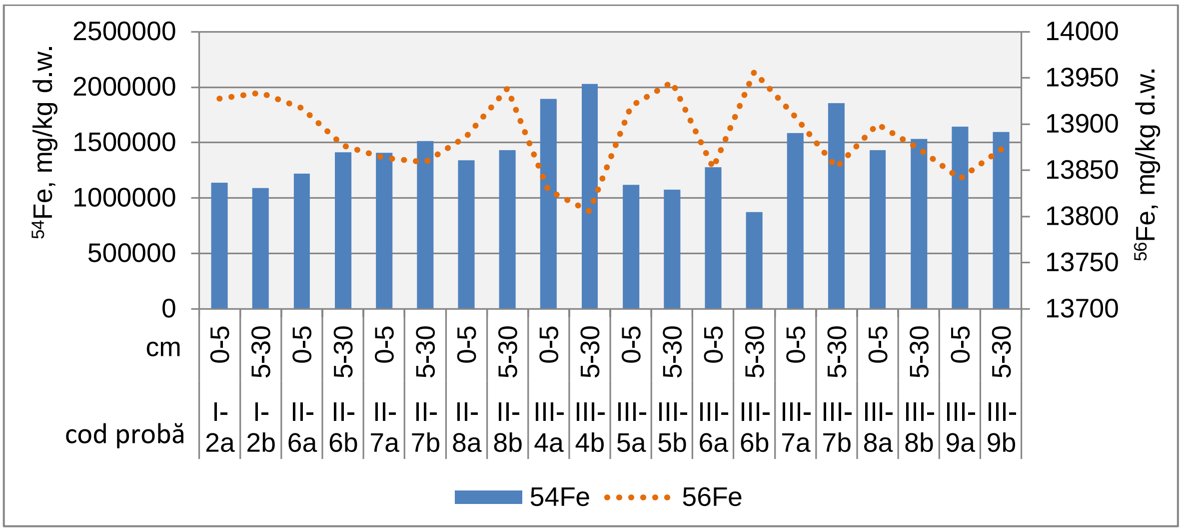


Figure 5.11 ^{54}Fe și ^{56}Fe average concentration in 0-5 cm și 5-30 cm layer of the arable lands' soil from Tulucesti, Sendreni, Galati county and Vadeni, Braila county

The study of stable isotopes has proved very useful in assessing the heavy metals and trace elements exchangeable forms in the soil [Ayoub, A.S. et al., 2003], [Oliver, Y.W. et al., 2006], as well as in identifying the source of pollution [Wang, L.W. et al., 2021], [Rabinowitz, M.B. et al., 1972]. For the analyzed soils, the following was found:

- The identified stable isotope concentrations in the soil do not show significant variations between the three territories and between the standard depths, 0-5 cm and 5-30 cm. Overall, the analytical results showed homogeneity of values.

- Of the six stable isotopes of Cd, ^{110}Cd, ^{111}Cd, ^{113}Cd, ^{112}Cd, and ^{114}Cd were identified. On the *territory of Tulucesti*, the soils have higher average concentrations of Cd isotopes in the upper layer of 0-5 cm compared to the layer of 5-30 cm. The soil in the *Sendreni area* has higher values in the 5-30 cm layer compared to the upper one of 0-5 cm. On the *Vadeni territory*, there is a tendency of accumulation in the upper layer of 0-5 cm of the soils located in the southwest direction of the Galati Iron and Steel Works, on an imaginary line between the northeastern extremity of Vadeni and Baldovinesti villages. In the bordering areas of Vadeni territory, the content of Cd isotopes is higher in the underlying layer of 5-30 cm compared to the upper one of 0-5 cm.

- On the *Tulucesti and Sendreni territories*, the accumulation of the ^{59}Co in the soil is similar, as the highest values are in the 5-30 cm layer. In the *Vadeni area*, the isotope ^{59}Co shows the same tendency of accumulation as the stable isotopes of Cd, respectively a higher concentration in the underlying layer of 5-30 cm in the eastern and western areas and a higher accumulation in the upper layer of 0-5 cm in central-western areas between Vadeni and Baldovinesti.

- Among the stable isotopes of Cr, the identified ^{52}Cr shows a tendency of accumulation in the upper layer of 0-5 cm on all the studied territories.

- For Cu, both stable isotopes, ^{63}Cu and ^{65}Cu, are present. For the *territories of Tulucesti and Sendreni*, a higher accumulation of ^{65}Cu in the 0-5 cm layer is observed compared to ^{63}Cu. The soils on *Vadeni* show the same tendency of higher accumulation in the upper layer of 0-5 cm in the area between Vadeni and Baldovinesti villages. The rest of the territory records higher values on the 5-30 cm layer.

- Among the stable isotopes of iron, the ^{54}Fe concentration is higher in the 0-5 cm layer than in the 5-30 cm layer on the *Tulucesti territory*, while the agricultural soils on the *Sendreni and Vadeni areas* show a higher accumulation of this isotope in the 5-30 cm section. In III-5, III-6, and III-9 sampling points between Vadeni and Baldovinesti, respectively north-west of Vadeni, the ^{54}Fe content is higher in the 0-5 cm layer. The distribution of ^{56}Fe shows no significant variations in depth between the three studied territories.

- The stable isotope ^{55}Mn has a higher accumulation in the 5-30 cm layer on all the studied territories. A higher concentration is in the upper layer of 0-5 cm on the Vadeni-Baldovinesti line.

- Among the stable isotopes of the element Ni, ^{58}Ni and ^{60}Ni were identified. While the 58Ni isotope has lower concentrations in the 0-5 cm layer, except for the area between Vadeni and Baldovinesti villages, the ^{60}Ni isotope has higher values in the 5-30 cm layer.

- Regarding the stable isotopes of the Pb, ^{206}Pb, ^{207}Pb and ^{208}Pb are present in the selected soils. For all stable Pb isotopes, the concentrations are higher in the upper layer of 0-5 cm.

- Among the stable isotopes of Zn, in the analyzed agricultural soils are present ^{64}Zn, ^{66}Zn and ^{68}Zn. Overall, there are higher concentrations in the 5-30 cm layer, except for the soils in the Vadeni-Baldovinesti direction, where the content of all stable Zn isotopes has higher values in the 0-5 cm layer.

The influence of anthropogenic factors on agricultural soils' is also evaluated by comparing the soil ratios of isotope concentrations with those of the corresponding natural abundances. By these ratios, the source of pollution, the variation of concentrations with the distance from the source of contamination [Wang, J. et al., 2021], [Chen, Z. et al., 2022], [Huang, Y. et al., 2021], the influence of physico-chemical and biological processes at ground level on the isotopic footprint [Gao, T. et al., 2021], [Schmitt, A.-D. et al., 2012], as well as the variation of the isotope concentration in the soil profile [Baieta, R. et al., 2022] may be identified.

The isotopic footprint of cadmium, expressed by the isotope ratios (^{114}Cd/ ^{110}Cd, ^{114}Cd/ ^{111}Cd, ^{114}Cd/ ^{112}Cd, ^{114}Cd/ ^{113}Cd, ^{113}Cd/ ^{110}Cd, ^{113}Cd/ ^{111}Cd, ^{112}Cd/ ^{111}Cd, ^{112}Cd/ ^{110}Cd, ^{111}Cd/ ^{110}Cd), is below those of the ratios calculated based on natural abundances. The exception is the isotopic footprint of $^{113}Cd/^{112}Cd$, which has values higher than the ratio of natural abundances (0.5064), without significant variations between the two sections (0-5 cm and 5-30 cm). The use of chemical fertilizers, especially those with phosphates, and the deposits of particles loaded with Cd from industrial emissions [Yuan, Z. et al., 2019] bring an important contribution of Cd in the soil [Loganathan, P. et al., 2003]. The isotopic footprint of copper ($^{65}Cu/^{63}Cu$) has a higher value than the natural abundance ratio (0.4461), indicating potential soil contamination from anthropogenic sources. The $^{65}Cu/^{63}Cu$ ratio value is similar in all the studied territories, with no notable variations by section.

The results of the $^{56}Fe/^{54}Fe$ ratio are below the value of the natural abundance ratio (15.6838). There are no differences in the $^{56}Fe/^{54}Fe$ isotopic ratio distribution between the three territories.

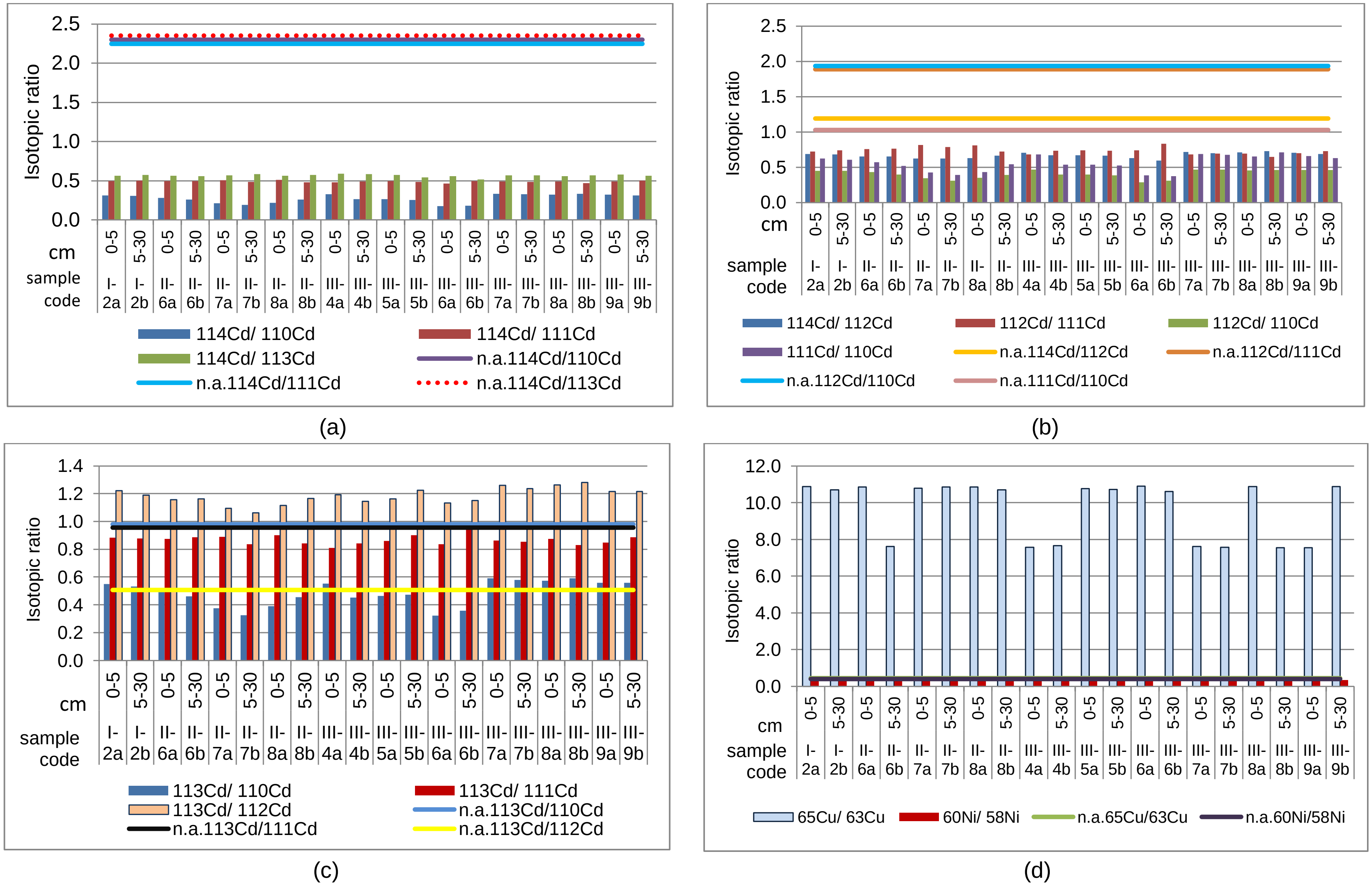
Overall, the ratio $^{60}Ni/^{58}Ni$ records a value below that of natural abundances (0.3852).

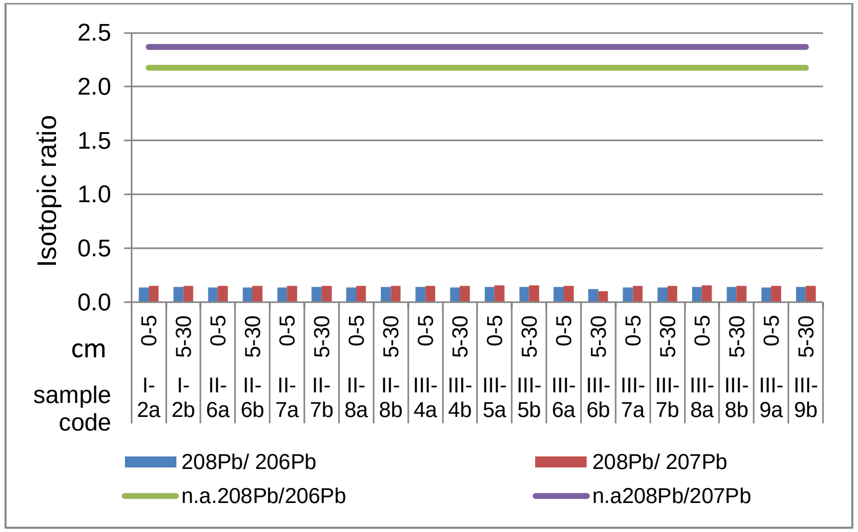
The isotopic footprints of lead based on $^{208}Pb/^{206}Pb$, and $^{208}Pb/^{207}Pb$ did not show higher values than the ratio of natural abundances, and $^{206}Pb/^{207}Pb$ have values around 1.0905. No significant variations are between the three studied territories.

A distinct situation is the isotopic ratio $^{207}Pb/^{208}Pb$, which registers a very high level compared to the natural abundances. The results of the $^{207}Pb/^{208}Pb$ exceed the European average of this ratio for agricultural soils (0.403 [Reimann, C. et al., 2012]).

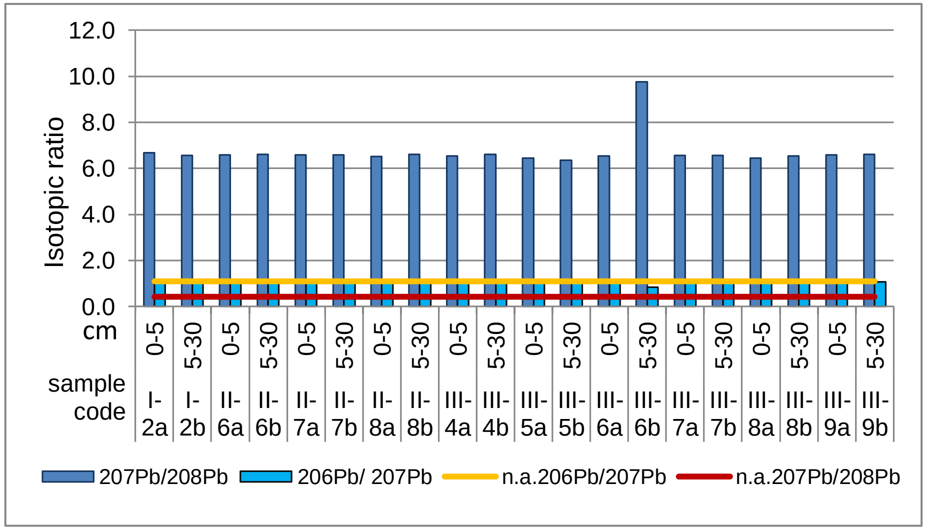
Concerning zinc, the values $^{66}Zn/^{64}Zn$, $^{68}Zn/^{66}Zn$, and $^{68}Zn/^{64}Zn$ ratios are higher than those of the natural abundances ratio, which indicates an anthropogenic accumulation of this element, the industrial activity being an important source of contamination. The isotopic footprint state is similar in all the three studied territories in the vicinity of the steel industrial complex and doesn't show significant variations in the analyzed soil sections.

Figure 5.12 shows the variation of the isotopic ratios characteristic of agricultural soils taken from the territories in the vicinity of the steel industrial complex compared to the natural abundance ratios of the identified isotopes.

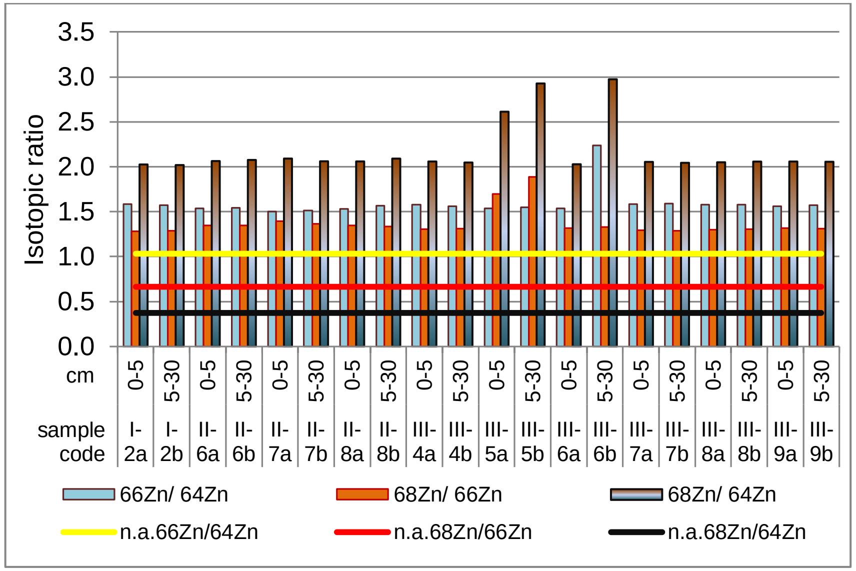

Isotopic ratio
cm
sample code
114Cd/ 110Cd
114Cd/ 111Cd
114Cd/ 113Cd
n.a.114Cd/110Cd
n.a.114Cd/111Cd
n.a.114Cd/113Cd
(a)
114Cd/ 112Cd
112Cd/ 111Cd
112Cd/ 110Cd
111Cd/ 110Cd
n.a.114Cd/112Cd
n.a.112Cd/111Cd
n.a.112Cd/110Cd
n.a.111Cd/110Cd
(b)
113Cd/ 110Cd
113Cd/ 111Cd
113Cd/ 112Cd
n.a.113Cd/110Cd
n.a.113Cd/111Cd
n.a.113Cd/112Cd
(c)
65Cu/ 63Cu
60Ni/ 58Ni
n.a.65Cu/63Cu
n.a.60Ni/58Ni
(d)



(e)



(f)



(g)

Figure 5.12 Characteristic isotopic ratios variation in 0-5 cm and 5-30 cm layer of the arable lands' soil from the territories of Tulucesti, Sendreni, Galati county and Vadeni, Braila county compared to the isotopic ratio of natural abundances (a) - (g)

n.a. - natural abundance

5.1.3. *Nuclear methods of analysis (PIGE, PIXE) results*

The HR-CS AAS, ICP-MS and EDX results were completed by the PIGE and PIXE nuclear analysis techniques employment, which allowed the determination of both light and heavy elements in the soil samples harvested from agricultural lands under the influence of steel activities.

The PIXE technique was used as a qualitative method, confirming the results of the advanced analysis methods mentioned above. Ca, Ti, Cr, Mn, Fe, Ni, and Zn were determined using an HPGe (High Purity Germanium) type detector, and Na, Mg, Al, Si, P, and S were identified with an external beam and a Si(Li) type detector.

Figure 5.13 shows an example of a characteristic PIXE spectrum of soils near Galati Iron and Steel Works.

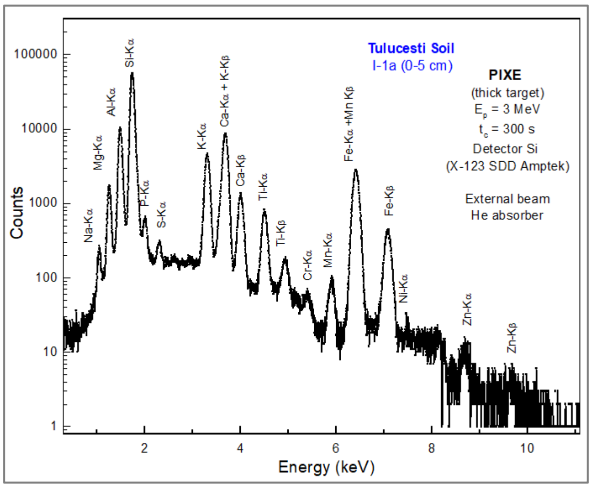


Figure 5.13 Characteristic PIXE spectrum of I-1a soil, taken from 0-5 cm layer, Tulucesti commune, Galati county [Ene, A. et al., 2019a]

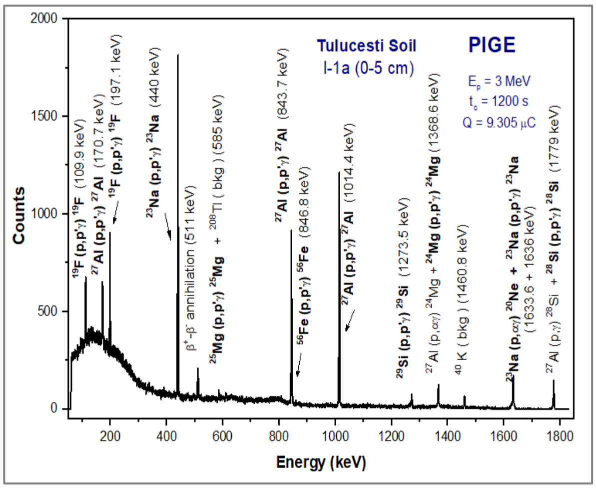


Figure 5.14 Characteristic PIGE spectrum of I-1a soil, taken from 0-5 cm layer, Tulucesti commune, Galati county

The light elements (Z<15) F, Al, Na, Mg, Si, Cl, and K were determined by the PIGE technique, also used for Fe and Ti identification. Tables 5.5 and 5.6 show the micro, macroelements and trace elements concentrations in the agricultural soils collected from Tulucesti, Sendreni, Galati county and Vadeni, Braila county.

The PIGE spectrum shown in Figure 5.14 highlights the characteristic gamma lines of the elements present in the soil sample, as well as the types of nuclear reactions (p,p'γ - ^{18}F, ^{23}Na, ^{24,25}Mg, ^{27}Al, ^{28,29}Si, ^{31}P; p,αγ - ^{23}Na, ^{31}P; p,nγ - ^{48}Ca) [Ene, A. et al., 2019a].

The PIGE results reveal that the level of the elements does not show significant variations between the three studied territories nor between the soil layers. Figures 5.15, 5.16, 5.17, and 5.18 illustrate the elements' concentration variation at 0-5 cm and 5-30 cm depth.

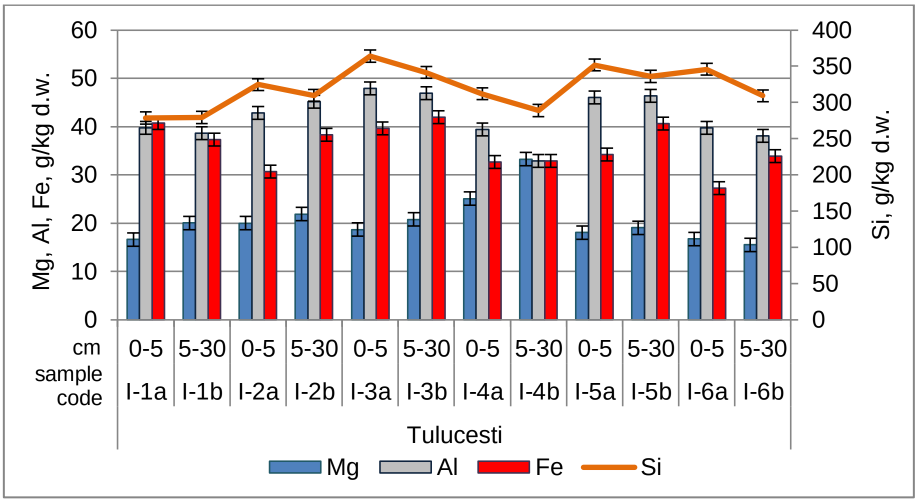


Figure 5.15 Mg, Al, Fe și Si average concentration in 0-5 cm și 5-30 cm layer of agricultural soils taken from Tulucesti, Galati county

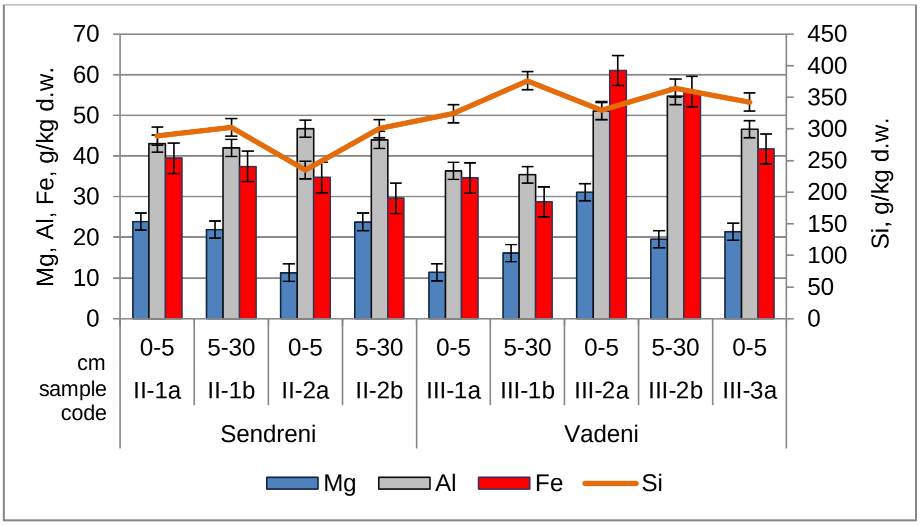


Figure 5.16 Mg, Al, Fe și Si average concentration in 0-5 cm și 5-30 cm layer of agricultural soils taken from Sendreni, Galati county and Vadeni, Braila county

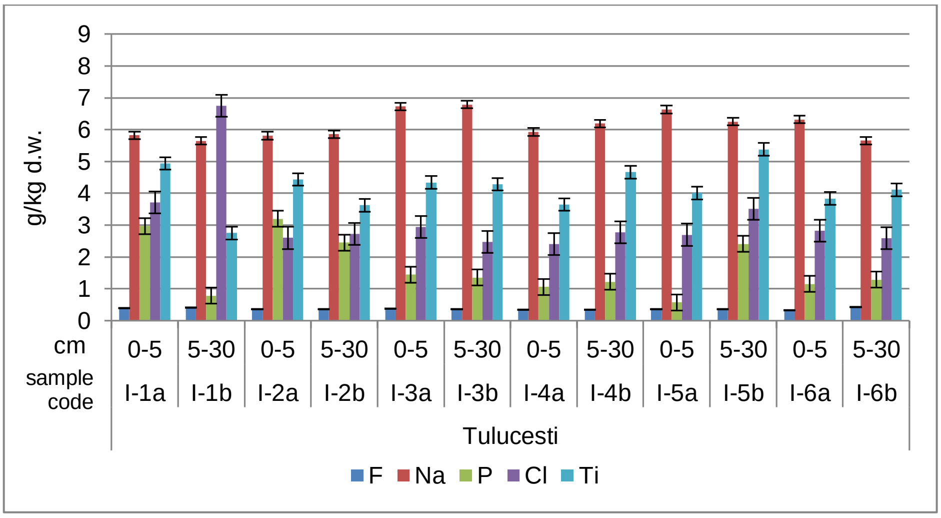


Figure 5.17 F, Na, P, Cl și Ti average concentration in 0-5 cm și 5-30 cm layer of agricultural soils taken from Tulucesti, Galati

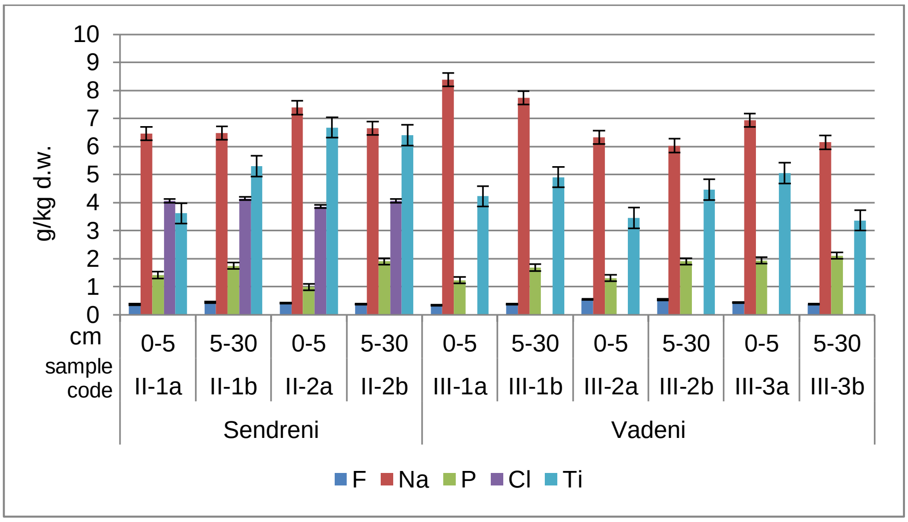


Figure 5.18 F, Na, P, Cl și Ti average concentration in 0-5 cm și 5-30 cm layer of agricultural soils taken from Sendreni, Galati county and Vadeni, Braila county

Partial results of PIXE and PIGE techniques were presented at the conferences [Ene, A. et al., 2018], [Ene, A. et al., 2019b, c, d] and published in [Ene, A. et al., 2019a].

5.2. Micro and macroelements concentration in wheat (*Triticum vulgare* Vill.), maize (*Zea mays* L.) și sunflower (*Helianthus annuus* L.)

The study of crop plant contamination with toxic elements is essential to understand the transfer of pollutants through the soil-plant system, especially in agricultural areas near industrial platforms, mining areas, heavily trafficked roads, and natural gas and oil extraction platforms.

The bioavailability and accumulation of heavy metals in plants connect with the soil's physical, chemical and biological properties they grow. The most important factors influencing the mobility of elements ions are soil reaction, texture (mainly colloidal clay), $CaCO_3$ and organic matter content [Kabata-Pendias, A., 2011], [Smical, A.I. et al., 2008], [Băjescu, I. and Chiriac, A., 1984], [Davidescu, D. and Davidescu, V., 1981]. At the same time, the bioaccumulation of metals varies depending on the species, vegetation stage, and climatic and relief conditions in which the plants grow. Some plants may be biomonitors for air, soil, and water, as they can accumulate toxic elements and metabolize them.

5.2.1. *Continuous source high-resolution atomic absorbtion spectrometry (HR-CS AAS) results*

Cadmium in plants. In *wheat*, the mean Cd concentration varies between 0.0011 ± 0.0001 and 0.2165 ± 0.0020 mg/kg d.w. in leaves and between 0.0002 ± 0.0000 and 0.1819 ± 0.0007 mg/kg d.w. in caryopsis (Figure 5.19). The average Cd content in caryopsis is in line with the FAO/WHO recommendations for all sampling points, except for wheat harvested from the I-7 point (Tulucesti), where it slightly exceeds the permissible value of 0.10 mg/kg provided by [https://eur-lex.europa.eu/homepage.html] and [FAO / WHO, 1995]. In *maize*, the mean Cd content is present within 0,0320 ± 0,0016-0,0930 ± 0,0045 mg/kg d.w., in leaves, and between 0,0017 ± 0,0001 and 0,0209 ± 0, 0000 mg/kg d.w., in caryopsis (Figure 5.21), the values being within the maximum admissible limit recommended by [https://eur-lex.europa.eu/homepage.html] and [FAO / WHO, 1995]. In *sunflower*, Cd varies in the range

0.0759 ± 0.0064-0.3608 ± 0.0069 mg/kg s.u. in leaf and 0.0928 ± 0.0041-0.3995 ± 0.0041 mg/kg d.w. in achenes (Figure 5.23). Values do not exceed the maximum allowed Cd limit set by [https://eur-lex.europa.eu/homepage. html] and [FAO / WHO, 1995].

Cobalt in plants. In *wheat*, the concentration of Co is in the range 0.0298 ± 0.0015-0.5627 ± 0.0152 mg/kg d.w., in leaves and 0.0056 ± 0.0003-0.0624 ± 0.0052 mg/kg d.w., in caryopsis (Figure 5.19). The leaves in the II-1 sampling point near the slag heap record a maximum of 0.5627 ± 0.015 mg/kg d.w. In *maize*, the Co content is present within 0.0134 ± 0.0002-0.0499 ± 0.0026 mg/kg d.w. in leaves and 0.0010 ± 0.0000-0.0079 ± 0.0006 mg/kg d.w. in caryopsis (Figure 5.21). In *sunflower*, the accumulation of Co follows the same trend as in wheat and corn. The average Co content varies between 0.0757 ± 0.0049-0.1276 ± 0.0111 mg/kg d.w. in leaves, and 0.0055 ± 0.0003-0.0474 ± 0.0042 mg/kg d.w. in achenes (Figure 5.23).

Cobalt has no maximum allowable threshold set for cereals and sunflower seeds in the European regulations. The accumulation of cobalt is in the order leaf>caryopsis/achene.

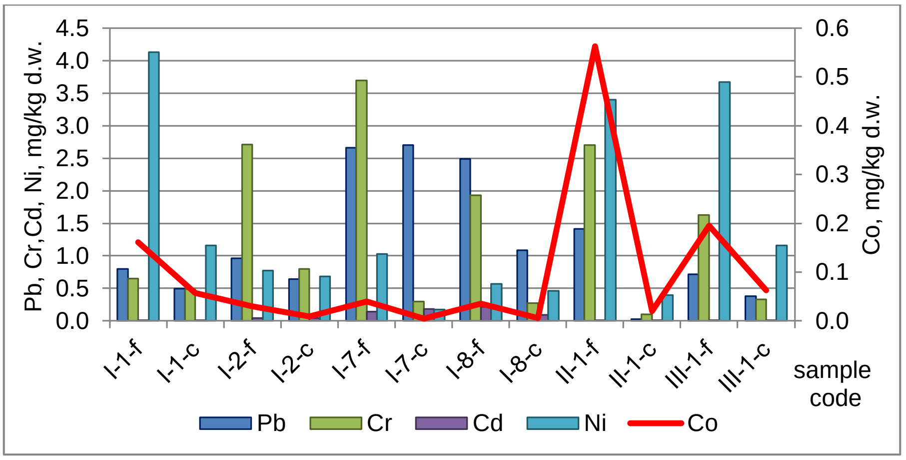


Figure 5.19 Pb, Cr, Cd, Ni și Co average concentration in wheat leaves and grains taken from Tulucesti, Sendreni, Galati county and Vadeni, Braila county

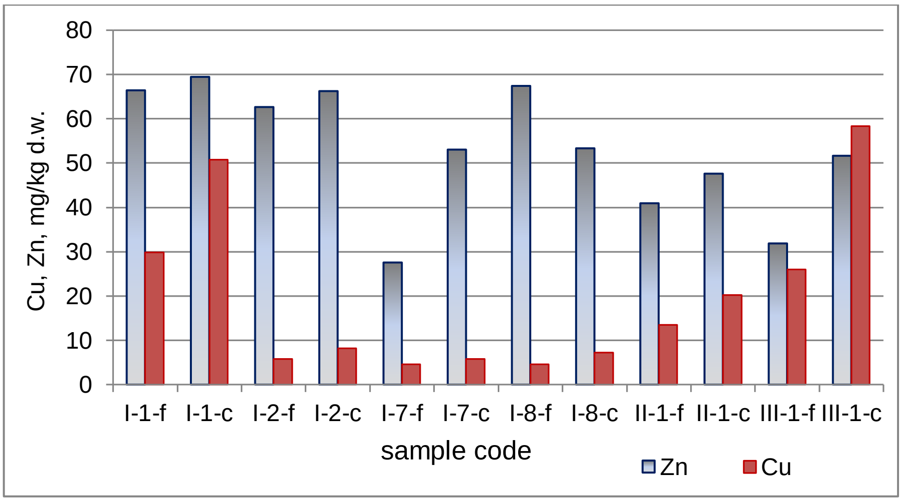


Figure 5.20 Zn și Cu average concentration in wheat leaves and grains taken from Tulucesti, Sendreni, Galati county and Vadeni, Braila county

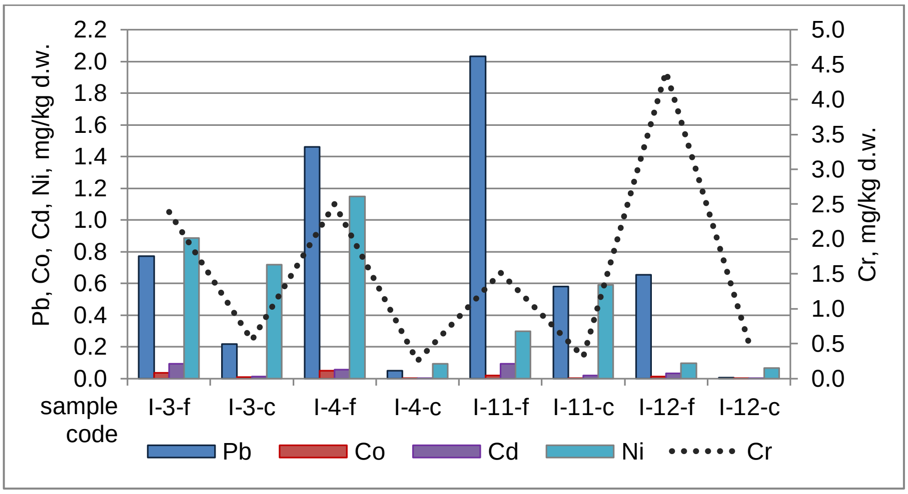


Figure 5.21 Pb, Co, Cd, Ni și Cr average concentration in maize leaves and grains taken from Tulucesti, Galati county

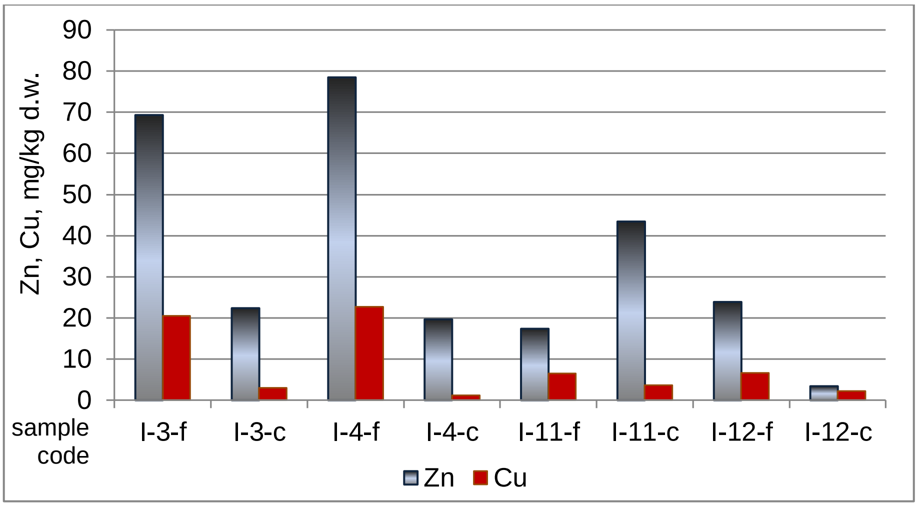


Figure 5.22 Zn și Cu average concentration in maize leves and grains taken from Tulucesti, Galați county

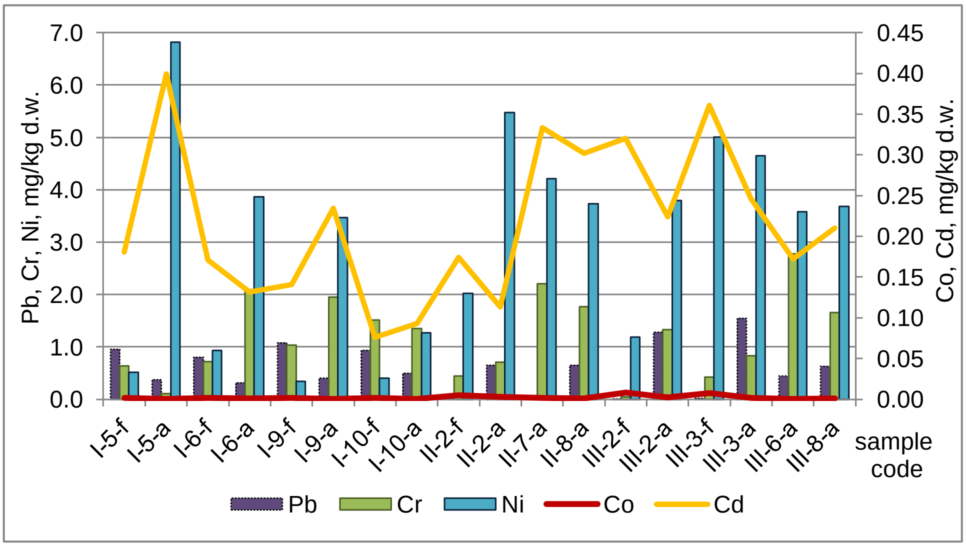


Figure 5.23 Cr, Cd, Ni, Co și Pb average concentration in sunflower leaves and seeds taken from Tulucesti, Sendreni, Galati county and Vadeni, Braila county

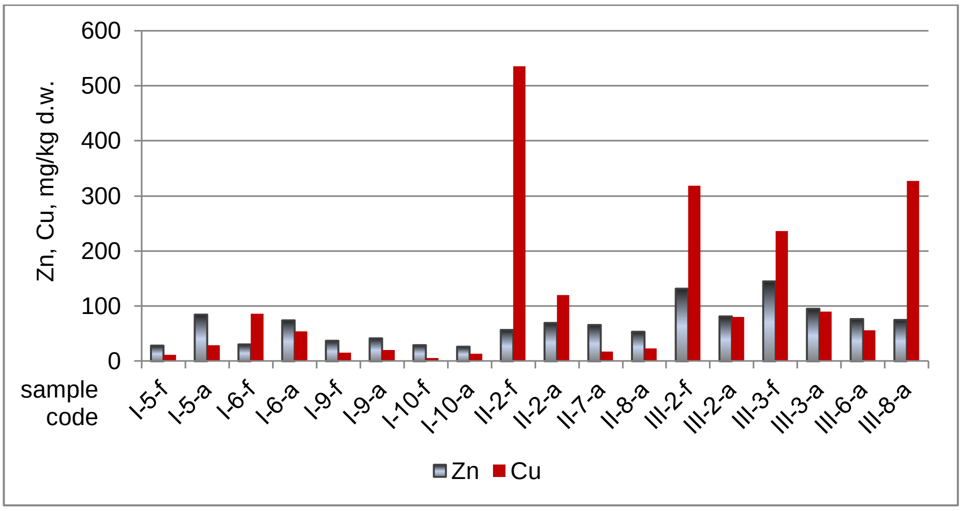


Figure 5.24 Zn și Cu average concentration in sunflower leaves and seeds taken from Tulucesti, Sendreni, Galati county and Vadeni, Braila county

Chromium in plants. Regarding the average Cr content in *wheat*, the results show a tendency for this element to accumulate in higher quantities in the leaves, the values being between 0.6488 ± 0.0554 and 3.6972 ± 0.2210 mg/kg d.w. In caryopsis, Cr is between 0.1004 ± 0.0044 and 0.8014 ± 0.0502 mg/kg d.w. (Figure 5.19). In the case of *maize*, the highest Cr concentrations are in the leaves (1.5153 ± 0.0220-4.4003 ± 0.2692 mg/kg d.w.), and the concentration in caryopsis is within the limits of 0.2465 ± 0.0157-0.5470 ± 0.0521 mg/kg d.w. (Figure 5.21). For *sunflower* is a different trend of Cr accumulation. Achenes record the highest values (0.1033 ± 0.0099-2.7785 ± 0.1882 mg/kg d.w.), while in the leaf, this element is in a concentration of 0.0423 ± 0.0020-1.5088 ± 0.0211 mg/kg d.w. (Figure 5.23).

This element has no maximum allowed value for the edible sections. After [Al-Othman, Z.A. et al., 2016], the maximum permitted threshold of Cr for crops is 2.30 mg/kg. After this value, the analyzed samples of caryopsis and achenes have low concentrations of Cr, except sunflower achenes taken from the point III-6, Vadeni.

Cooper in plants. The average Cu content in the *wheat* samples varies as follows: the lowest average values are between 4.6635 ± 0.0345 and 29.9108 ± 0.2094 mg/kg d.w. in the leaf. In caryopsis, the mean level of Cu varies between 5.7913 ± 0.0293-58.3966 ± 0.4672 mg/kg d.w. (Figure 5.20). For all the caryopsis samples, the mean Cu level exceeds the maximum permissible limit set by [Order no. 975/1998], even in a proportion of 4 to 10 times more than the maximum in the I-1, II-1, and III-1 sampling points but are below the maximum value recommended by [Al-Othman, Z.A. et al., 2016]. In the case of maize, the element Cu accumulates more in the leaf (6.5535 ± 0.2709-22.7788 ± 0.3072 mg/kg d.w.) compared to caryopsis (1.2676 ± 0.0738- 3.7025 ± 0.0127 mg/kg d.w.) - Figure 5.22. The concentration of Cu in maize caryopsis is within the maximum permitted limit set by [Order 975/1998] for cereals and is below the maximum value in crops after [Al-Othman, Z.A. et al., 2016]. For sunflower, the results show that, in the samples taken from Tulucesti, the lower average concentrations of Cu are in the leaves (5.8448 ± 0.2235-15.2186 ± 0.0988 mg/kg d.w.). In achenes the concentration is between 13.6488 ± 0.68 and 54.3190 ± 0.3626 mg/kg d.w. For Sendreni and Vadeni territories is a different trend of accumulation, the results highlighting situations when the values are higher in the leaf than in achene and vice versa. For the two territories, the average Cu content in the leaves is 535.3477 ± 0.5353 mg/kg d.w.

at Sendreni and 235.9469 ± 6.8425-318.2454 ± 8.9109 mg/kg d.w. in Vadeni. In achenes, Cu is between 13.6488 ± 0.0968 and 120.1464 ± 8.2901 mg/kg d.w. at Sendreni, and 327.2088 ± 12.3200 mg/kg d.w. in Vadeni (Figure 5.24). Cooper has no maximum permissible limits for sunflower seeds in the European regulations. After [Al-Othman, Z.A. et al., 2016], the concentration of this element in crops should not exceed 73.30 mg/kg.

Nickel in plants. The analytical data for *wheat* show that Ni accumulated in higher average concentrations in leaves (0.5680 ± 0.0608 and 4.1301 ± 0.2354 mg/kg d.w.) than in caryopsis (0.1771 ± 0.0264-1.1637 ± 0.1152 mg/kg d.w.) - Figure 5.19. For *maize*, the same trend is maintained, in leaves the average concentration is 0.0950 ± 0.0636-1.1471 ± 0.0014 mg/kg d.w., and in caryopsis, 0.0660 ± 0.0239-0.7181 ± 0 , 0342 mg/kg d.w. (Figure 5.21). In the case of *sunflower* the situation is different, as the Ni has accumulated in higher average concentrations in achenes (1.2731 ± 0.0609-6.8157 ± 0.1438 mg/kg d.w.) compared to leaves (0.3737 ± 0.0203-5.0053 ± 0.4455 mg/kg d.w.) - Figure 5.23.

Nickel has no maximum allowable value in food in the European regulations. [Al-Othman, Z.A. et al., 2016] state that the level of Ni in crops should not exceed the threshold of 67.90 mg/kg. The results for Ni levels in plants are below this limit.

Lead in plants. *Wheat* accumulated more Pb in leaves than in caryopsis, with values between 0.7139 ± 0.0521 and 2.6630 ± 0.0548 mg/kg d.w., respectively between 0.0259 ± 0.0023 and 1.0830 ± 0.1045 mg/kg d.w. - Figure 5.19. The average concentrations for caryopsis exceed the maximum value set by [https://eur-lex.europa.eu/ homepage.html] and [FAO / WHO, 1995]. In the case of *maize*, the mean values of this element are higher in the leaves (0.6534 ± 0.0570-2.0330 ± 0.16273 mg/kg d.w.) compared to those in caryopsis, in which it varies between 0.0046 ± 0.0001 and 0.5821 ± 0.0046 mg/kg d.w. (Figure 5.21). In the I-3 and I-11 points (Tulucești), the average values for Pb exceed the maximum allowed of 0.20 mg/kg [FAO / WHO, 1995]. The accumulation of lead in the *sunflower* sections involves two distinct situations (Figure 5.23):

1. On the territory of Tulucesti, the results showed a higher accumulation in leaves (0.8046 ± 0.0358 and 0.9552 ± 0.00301 mg/kg d.w.) than in achenes (0.3718 ± 0.0343 and 0.4912 ± 0.0405 mg/kg d.w.).
2. In the Sendreni and Vadeni territories, Pb records higher average values in achenes (0.4399 ± 0.0172 and 1.6180 ± 0.0970 mg/kg d.w.) compared to those in leaves (0.0067 ± 0.0003 and 0.0114 ± 0.0009 mg/kg d.w.). The average Pb concentration in achenes taken from these territories exceeds the maximum allowable value set by [https://eur-lex.europa.eu/homepage.html] and [FAO / WHO, 1995].

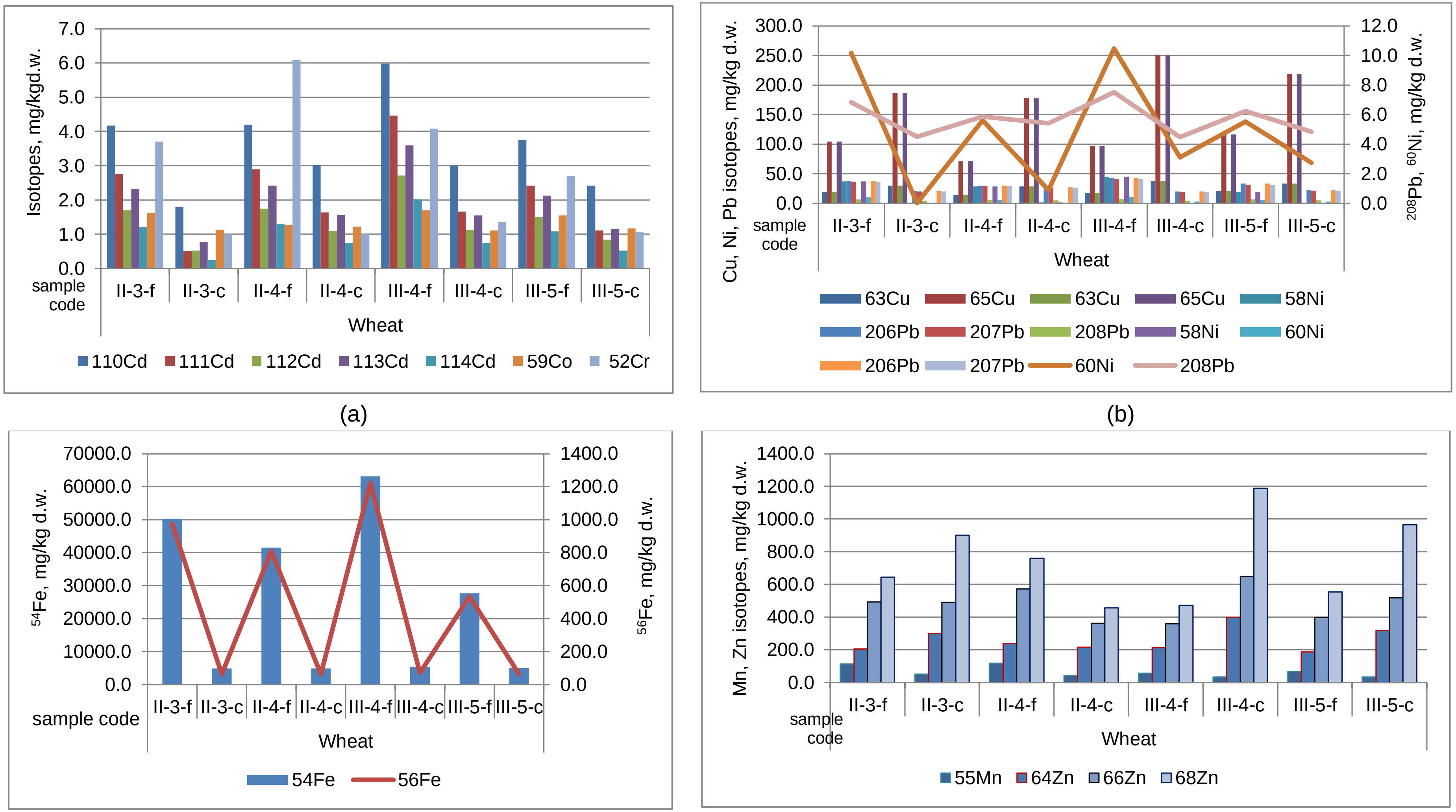
Zinc in plants. The average zinc in *wheat* sections shows a tendency for high accumulation in caryopsis (47.5840 ± 1.5703-69.4203 ± 0.4165 mg/kg d.w.). In leaves, Zn varies between 27.5370 ± 0.4530 and 67.3812 ± 0.7668 mg/kg d.w. (Figure 5.20). [Order no. 975/1998] provided a maximum of 15 mg/kg Zn for cereals. Compared to the maximum mentioned by [Al-Othman, Z.A. et al., 2016], the concentration of Zn in caryopsis is low. Compared with wheat, maize accumulated more Zn in the leaves (17.3812 ± 0.6334-78.4678 ± 0.2328 mg/kg d.w.) than in the caryopsis (3.4229 ± 0, 5423-43.4355 ± 0.8903 mg/kg d.w.) - Figure 5.22. The values exceed the maximum allowed for cereals [https://eur-lex.europa.eu/homepage.html] and [FAO / WHO, 1995] and are below the value mentioned by [Al-Othman, Z.A. et al., 2016]. The accumulation of Zn in sunflower tissues is highlighted, in general, by the higher average values in achenes than in leaves. The average concentration of Zn in the leaf varies between 28.2430 ± 0.9340 and 144.8514 ± 2.0279 mg/kg d.w., and in achenes between 26.2678 ± 0.8287 and 95.1353 ± 0.8562 mg/kg d.w. (Figure 5.24). Zinc has no maximum permissible limits in sunflower seeds. The values recorded in achenes are lower than that recommended by [Al-Othman, Z.A. et al., 2016].

5.2.2. *Inductively coupled plasma mass spectrometry (ICP-MS) results*

The ICP-MS technique was employed to assess the level of plant contamination with macro, microelements and toxic or potentially toxic trace elements in the vicinity of the Galati steel industrial complex. The same soil stable isotopes of metals are present in plants, namely ^{110}Cd, ^{111}Cd, ^{112}Cd, ^{113}Cd, ^{114}Cd, ^{59}Co, ^{52}Cr, ^{63}Cu, ^{65}Cu, ^{54}Fe, ^{56}Fe, ^{55}Mn, ^{58}Ni, ^{60}Ni, ^{206}Pb, ^{207}Pb, ^{208}Pb, ^{64}Zn, ^{66}Zn and ^{68}Zn. Figures 5.25, 5.26, and 5.27 show the variation of stable isotopes' average concentration in the tissues of wheat, maize and sunflower taken from the territories near Galati Iron and Steel Works.

The analytical results indicate the tendency of micro and macroelements to accumulate, especially in the leaves. The isotopes concentrations in plants show the relationship between soil and plant relationships in the transfer of nutrients and toxic or potentially toxic elements.

The isotopic footprint assessment relied on the isotope ratios determination. As in the soil, $^{113}Cd/^{112}Cd$, $^{65}Cu/^{63}Cu$, $^{66}Zn/^{64}Zn$, $^{68}Zn/^{66}Zn$ and $^{68}Zn/^{64}Zn$ recorded higher values than the natural abundance ratios. It indicates the isotopic specificity of the studied area and the relation between soil and plants are grown on it.



(c) (d)

Figure 5.25 Metal stable isotopes average concentration in wheat tissues taken from Sendreni Galati county and Vadeni, Braila county

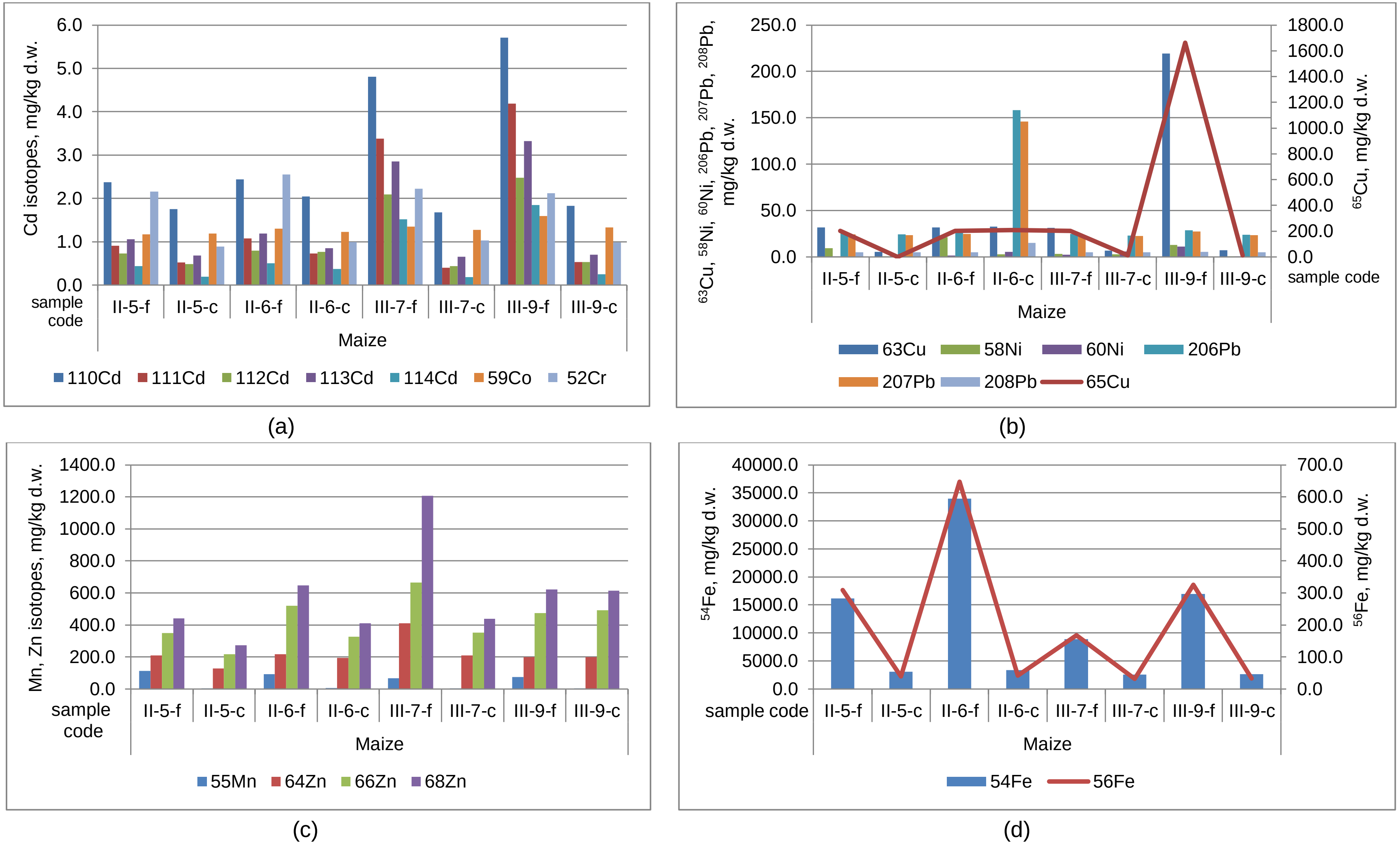


Figure 5.26 Metal stable isotopes average concentration in maize tissues taken from Sendreni Galati county and Vadeni, Braila county

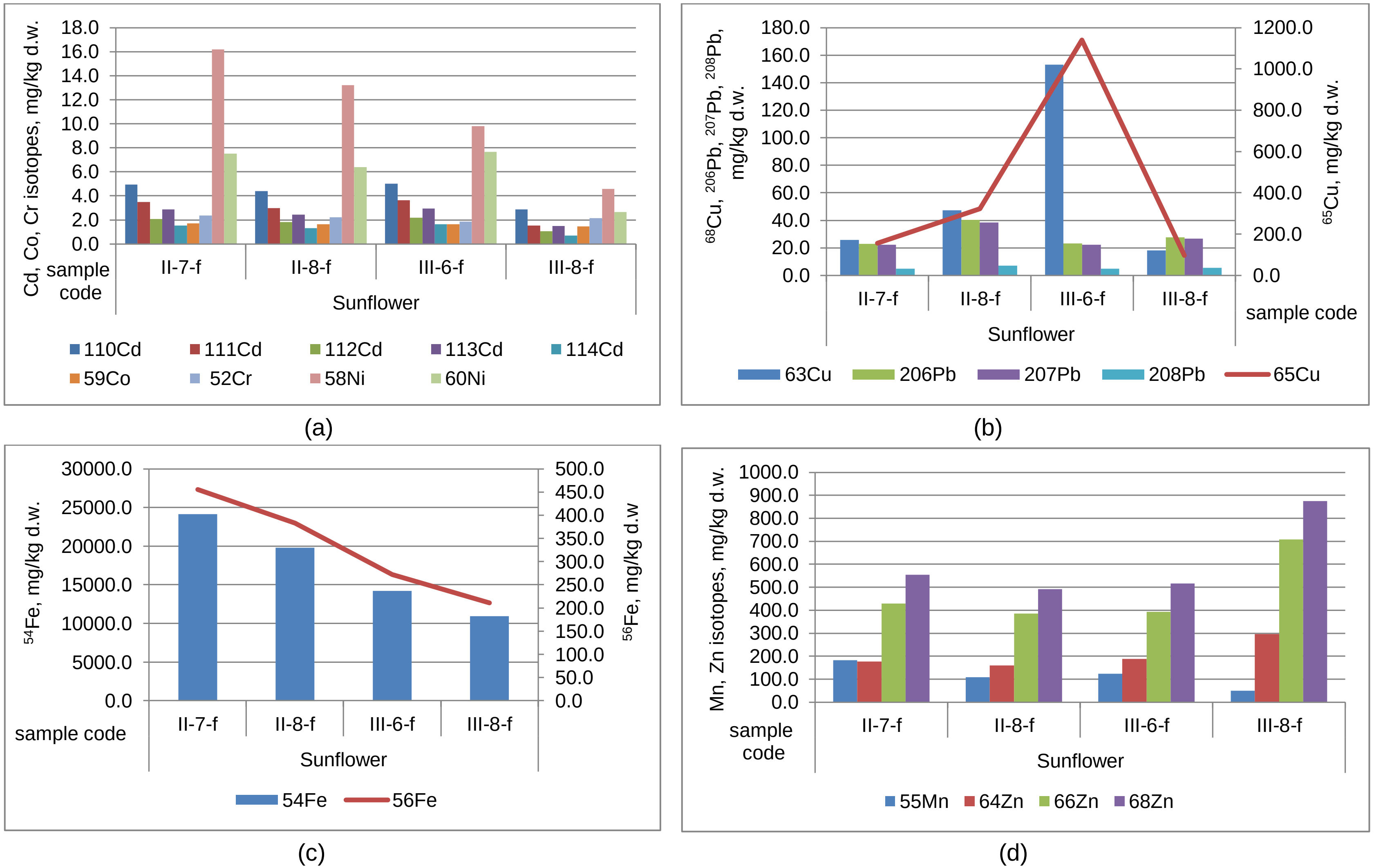
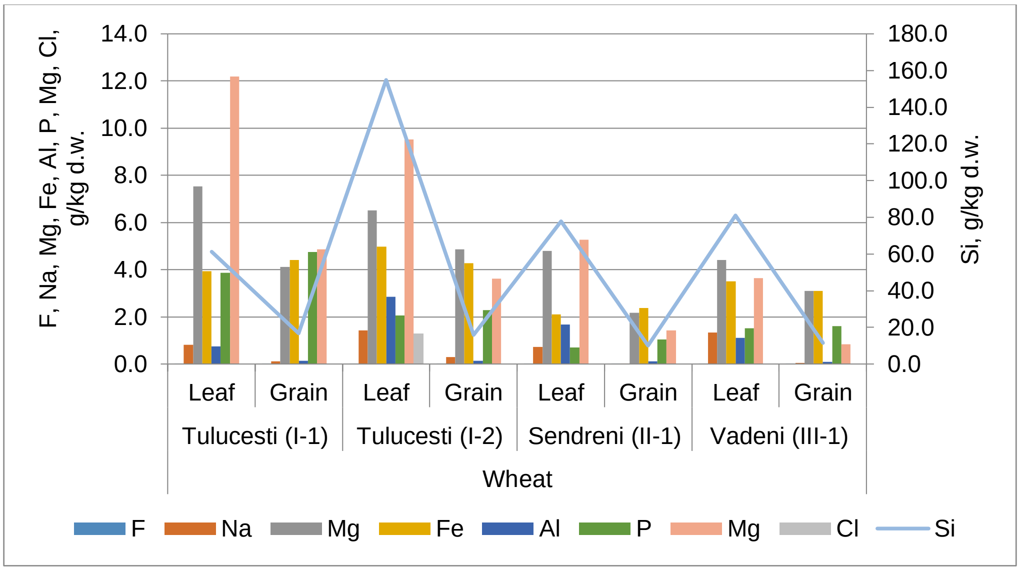


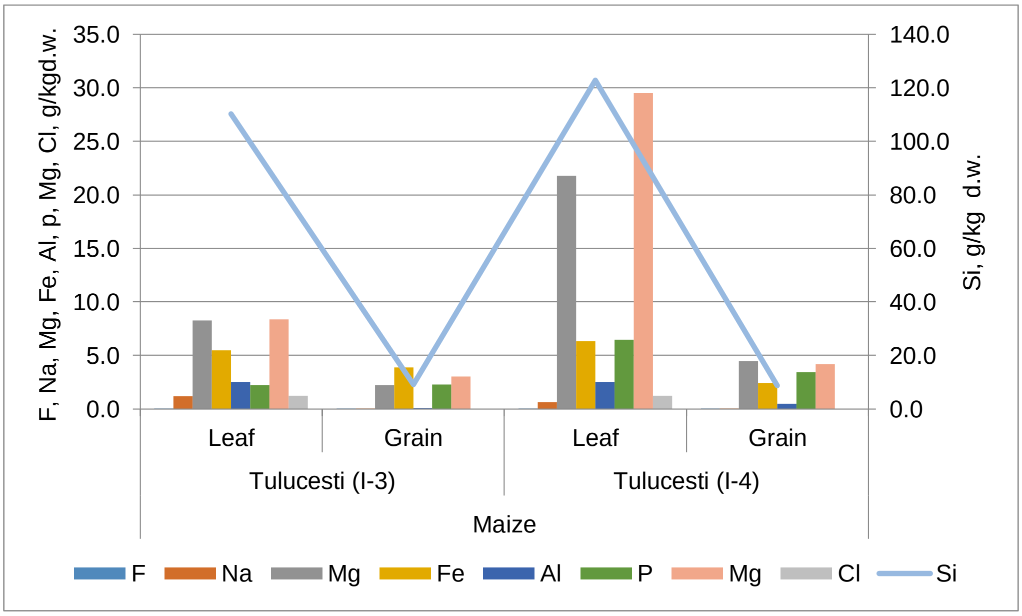
Figure 5.27 Metal stable isotopes average concentration in sunflower tissues taken from Sendreni Galati county and Vadeni, Braila county

5.2.3. *Nuclear methods of analysis (PIGE, PIXE) results*

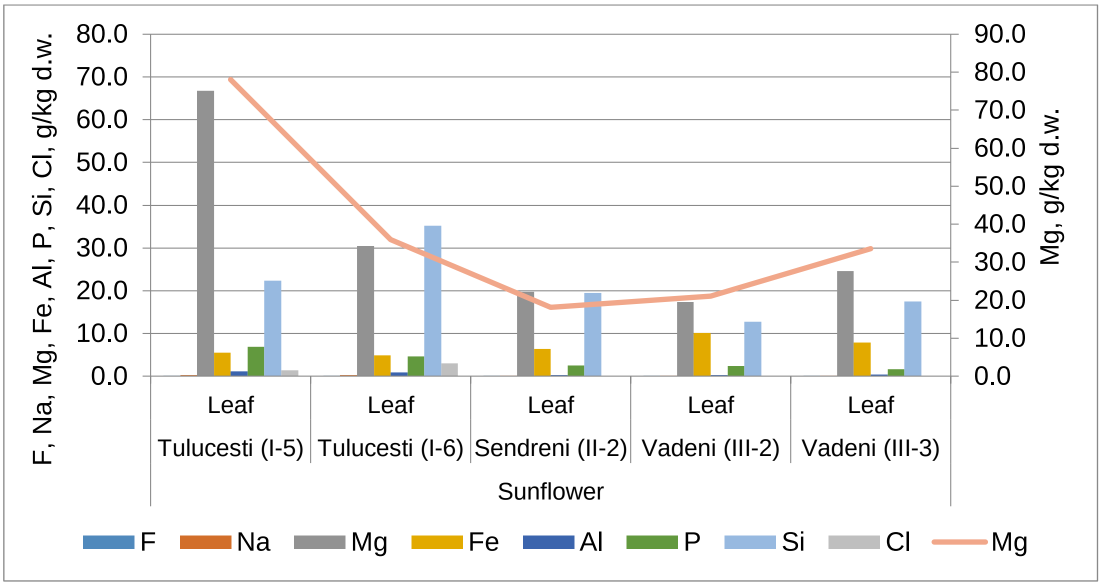
The PIGE and PIXE nuclear techniques served for the complex evaluation of crop plants and to identify and quantify the elements that the other advanced methods didn't assess. Figures 5.29 and 5.31 illustrate the PIGE spectra of the following light and heavy elements: Ca, Ti, Cr, Mn, Fe, Ni, Zn, Na, Mg, Al, Si, P, S. The light elements in the composition of the environmental samples matrix (C, O, Ca, Si, K) were identified by the RBS technique - Figure 5.30. Figure 5.28 shows the PIGE elemental concentration variation in the vegetal tissues.



(a)

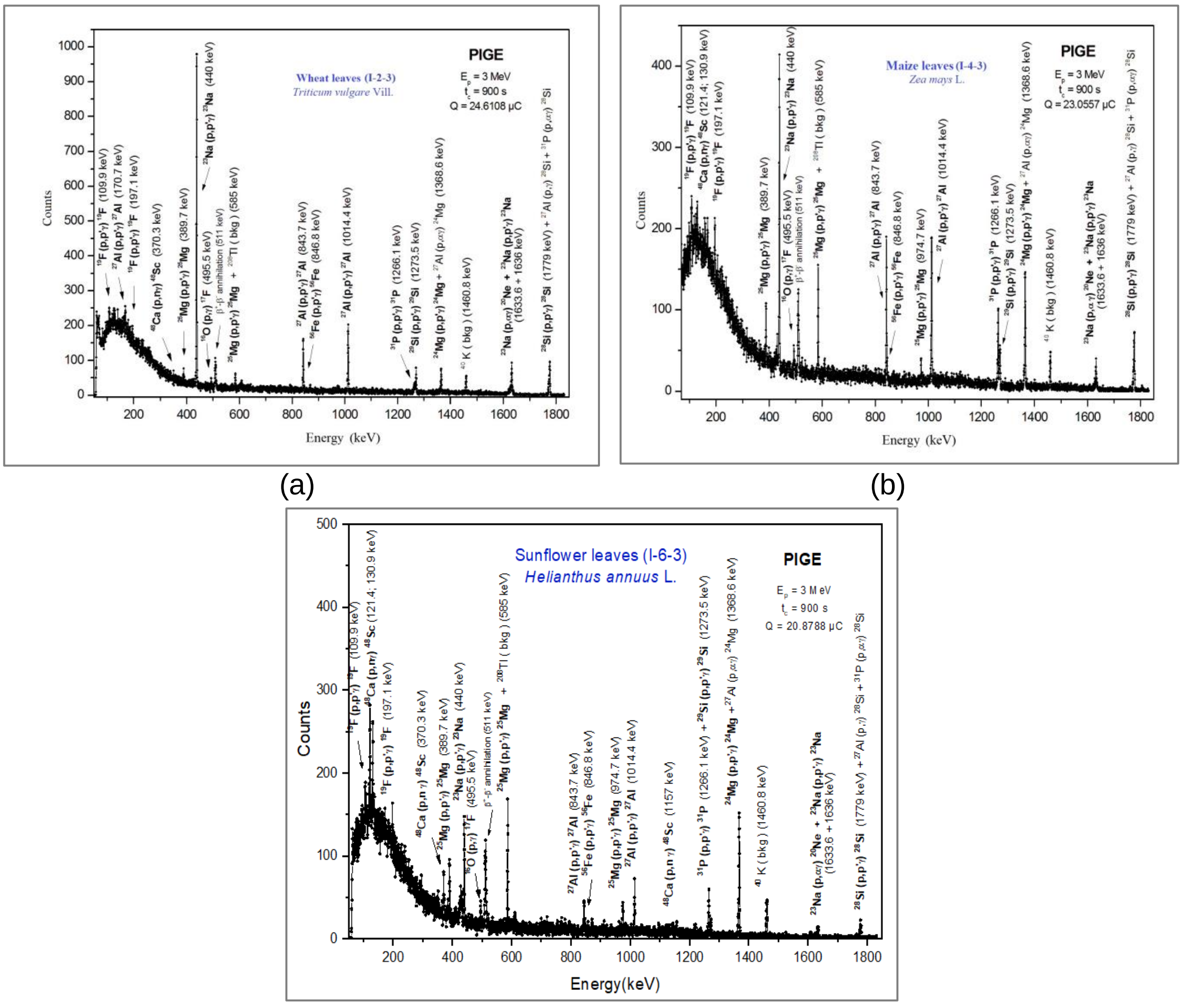


(b)



(c)

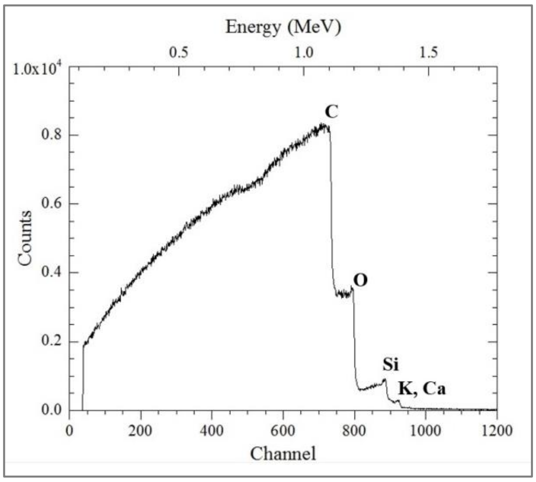
Figure 5.28 F, Na, Mg, Fe, Al, P, Si, Cl, Mg average concentration in vegetal tissues: (a) wheat leaves and grains, (b) maize leaves and grains, (c) sunflower leaves



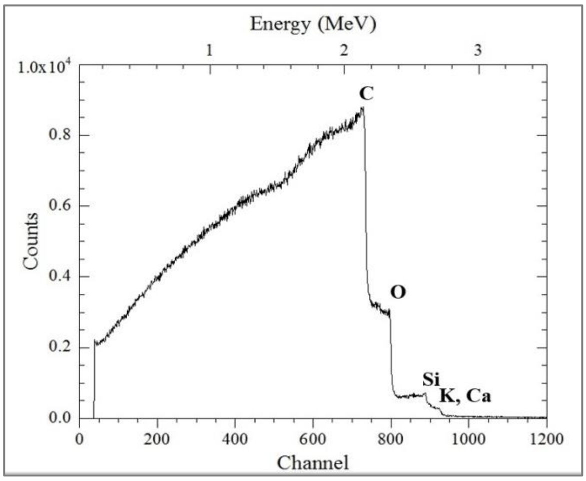
(a) (b)

(c)

Figure 5.29 Characteristic PIGE spectra of leaves of (a) wheat, (b) maize, (c) sunflower taken from the vicinity of Galati Steel Plant [Ene, A. et al., 2019a, b]



(a)



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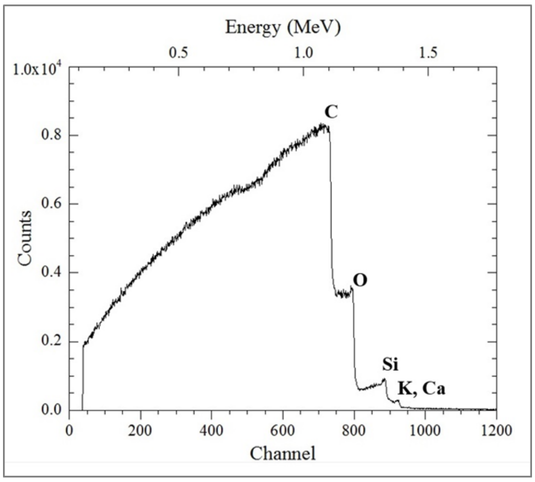


(c)

Figure 5.30 Characteristic RBS spectra of leaves of (a) wheat, (b) maize, (c) sunflower taken from the vicinity of Galati Steel Plant [Ene, A. et al., 2019a, b]

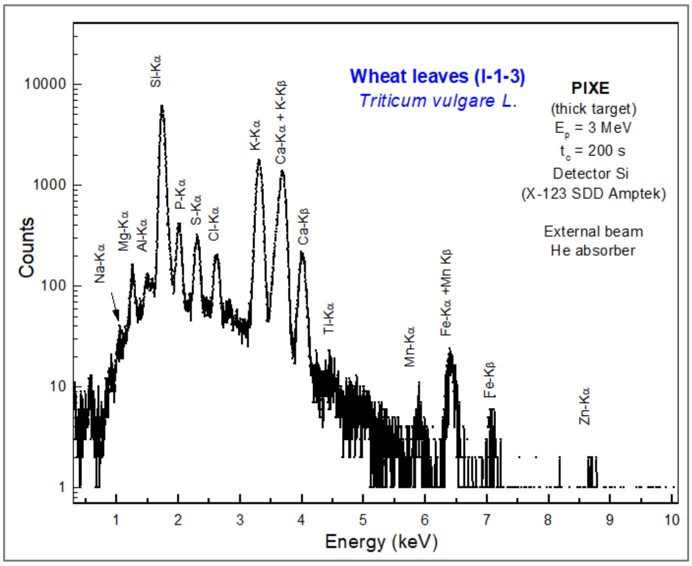


Figure 5.31 Characteristic PIXE spectrum of wheat leaves taken from Tulucesti, Galati [Ene, A. et al., 2019a, b]

5.3. Partial results

The experimental results showed the following:

Soil analysis

The HR-CS AAS method allowed the quantification of Cd, Co, Cr, Cu, Mn, Ni, Pb and Zn concentrations. *The results showed that the Cd, Co, Mn, Pb, and Zn level does not* *exceed the set values* regulated by [Order no. 756/1997]. **Chromium recorded the highest concentrations, exceeding the alert threshold for sensitive uses** in the 0-5 cm and 5-30 cm layers of agricultural soils in all three territories. **For this element, there were also punctual exceedances of the intervention threshold for sensitive uses**.

Regarding the copper content, the analytical results showed that, on the whole, the average values are around the normal threshold provided by law on both standard depths, *except for a few areas where concentrations exceed normal values but do not reach the alert threshold for sensitive uses*. The nickel concentration registers exceedings in the normal regulated values in the 0-5 cm layer and the 5-30 cm layer in all the studied territories. The Vadeni area has higher values compared to the Sendreni and Tulucesti territories.

ICP-MS technique completed the results obtained by the HR-CS AAS method, which allowed the identification of the isotopic footprint specific to the soils in the vicinity of the steel industrial complex. The isotope ratios were compared with the values of their natural abundance ratios. The isotopic signature of Cd (113Cd/112Cd), Cu (65Cu/63Cu), Pb (207Pb/208Pb), and Zn (66Zn/64Zn, 68Zn/66Zn, and 68Zn/64Zn) show higher values than the ratio of the natural abundances of these isotopes. 65Cu/63Cu, 207Pb/208Pb and 68Zn/64Zn recorded the highest results, which indicate the impact of neighbouring industrial activities. These values may also suggest other anthropogenic sources of contamination (road traffic, plant protection products and chemical fertilizers).

Nuclear analysis methods allowed the detection of light and heavy elements existing in agricultural soils in the Galati-Braila area. PIXE technique provided the qualitative assessment of micro and macroelements in the soil matrix, which enabled the detection of Na, Mg, Al, Si, P, S, Ca, Ti, Cr, Mn, Fe, Ni, and Zn. The PIGE method allowed us to identify and quantify mainly the light elements F, Al, Na, Mg, Si, Cl and K but also Fe and Ti. The PIGE technique detected the F element, which is more difficult to quantify by other analytical methods. Fluorine has higher values than the average concentration of this element in world soils, the primary source of contamination being the steel activity adjacent to the studied territories. Si and Al, inherited from the geochemical structure of the parent material, have the highest values.

Plant analysis (leaves and grains/seeds of wheat, maize and sunflower)

The HR-CS AAS method experimental results on micro and macroelements content in the leaves and caryopsis/achenes of wheat, maize and sunflower showed that their accumulation varies depending on the species and the physiological section. Overall the studied plants tend to accumulate metals, particularly in the leaves. In the case of sunflower, the elements Ni, Pb, Cr and Zn have higher average concentrations in achenes. On the territories of Vadeni and Sendreni, *the Pb content exceeds the maximum limit allowed by the European regulations*. *The ICP-MS* identified the same stable isotopes of micro and macroelements in the soil. The values of the isotopic ratios highlight the soil-plant connection in the transfer of chemical elements. The highest values were recorded for Cd (^{113}Cd/^{112}Cd), Cu (^{65}Cu / ^{63}Cu), Zn (^{66}Zn/^{64}Zn, ^{68}Zn/^{66}Zn and ^{68}Zn/^{64}Zn) and, punctually, Ni (^{60}Ni/^{58}Ni).

PIXE and PIGE nuclear techniques have shown the presence in plant tissues of both light (Na, Mg, Al, Si, P, S) and heavy (Ca, Ti, Cr, Mn, Fe, Ni, Zn) elements that exist in the soil matrix on which they were grown. The results obtained by the PIGE method confirmed the predilection of chemical element accumulation in higher quantities in the leaves compared to caryopsis and achenes.

6. MORFOLOGICAL AND MINERALOGICAL RESEARCH OF AGRICULTURAL SOILS AND CROPS IN THE AREAS ADJACENT TO THE GALATI STEEL PLANT

6.1. Identifican of soil minerals by ATR-FTIR spectroscopy

The ATR-FTIR analysis aim was to identify the soil spectral signature and the types of minerals from their composition. The ATR-FTIR spectra shown in Figure 6.1 illustrate that the absorption bands for the selected soil samples are similar, but their peak intensities are different. Thus, the absorption bands attributed to the characteristic vibrations of the functional groups of the chemical elements present in the analyzed samples highlighted the presence of clay and non-clay minerals [Arbanaș (Moraru), S.-S. et al., 2019a, b, c], [Ene, A. et al., 2020b], [Moraru, S.-S. et al., 2019a, d, e].

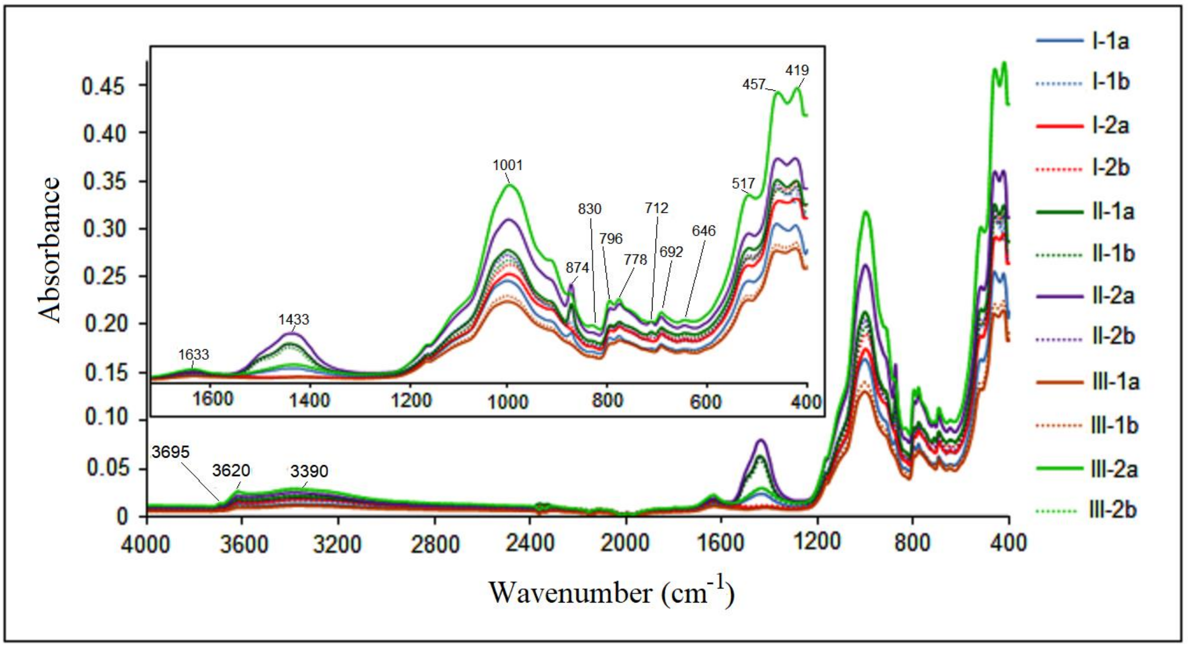


Figure 6.1 Characteristic ATR-FT-IR spectra of some soil samples collected from 0-5 cm (continuous line) and 5-30 cm (dotted line) depth on territories: Tulucesti (I-1a, I-1b; I-2a, I-2b), Sendreni (II-1a, II-1b; II-2a, II-2b) and Vadeni (III-1a, III-1b; III-2a, III-2b) (processed after [Moraru, S.-S. et al., 2019a, d])

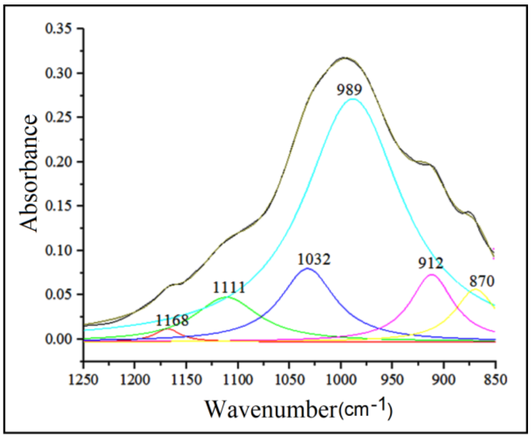


Figure 6.2 Deconvolution of complex IR absorption band comprised between 1250-850 cm^{-1}
[Moraru, S.-S. et al., 2019 a, d]

The spectral range 1500-400 cm^{-1}, also called the "spectral fingerprint range" [Bulgariu, D. et al., 2018], shows the absorption bands specific to clay minerals (montmorillonite and kaolinite) and non-clay minerals (quartz (SiO_2), feldspars (orthosis ($KAlSi_3O_8$), albite ($NaAlSi_3O_8$)) and calcite ($CaCO_3$)). The deconvolution of the ATR-FTIR spectra highlights the bands that make up the absorption band complex within the range 850-1250 cm^{-1} (Figure 6.2).

The characteristic ATR-FTIR spectra of the selected soils are similar to those obtained for that taken by [Sion, A. et al., 2020] from the parks of

Galati, which confirms the mineralogical signature of the research region.

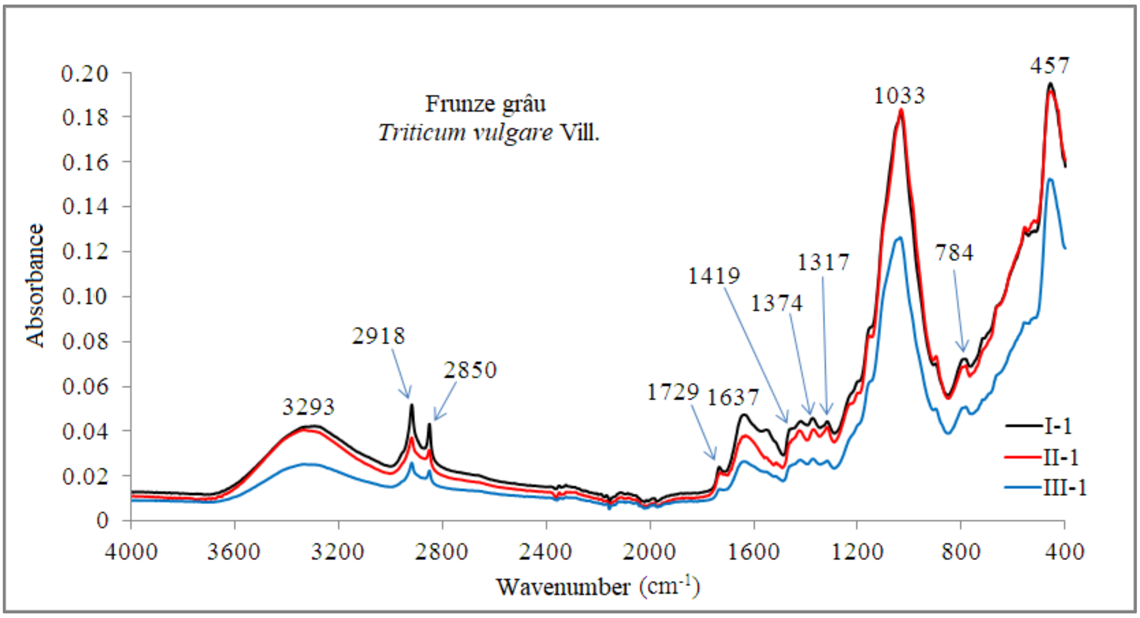
The ATR-FTIR method results on the soil samples collected from the territories of Tulucesti, Sendreni, and Vadeni were partially presented at the national conference [Arbanaș (Moraru), S.-S. et al., 2019a], at international conferences [Arbanaș (Moraru), S.-S. et al., 2019 b, c], [Ene, A. et al., 2020b], [Moraru, S.-S. et al., 2019d] and partially published in [Moraru, S.-S. et al., 2019a, e].

6.2. Plant organic compound functional groups identification by ATR-FT-IR spectroscopy

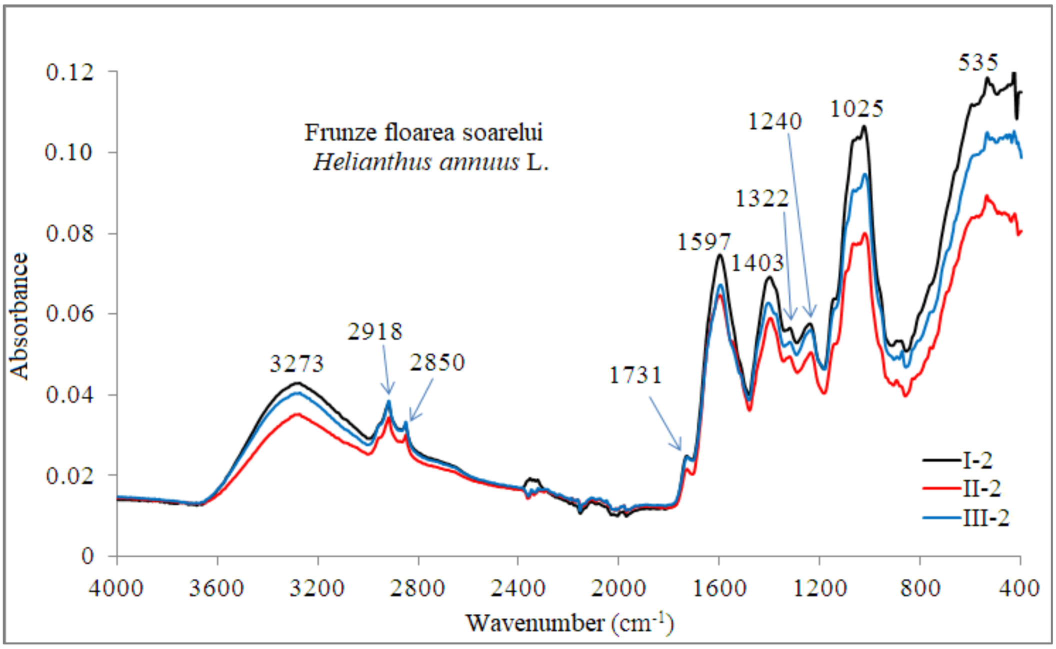
Plant tissues are complex structures consisting of organic compounds in various combinations such as cellulose, lignin, sugars, proteins, starches, lipids, waxes, et cetera. All this has a defining role in the development of the physiological processes of plants and the nutrition of final consumers, humans and animals. Figure 6.3 shows the characteristic ATR-FT-IR spectra of wheat and sunflower tissues taken from the territories of Tulucesti, Sendreni, Galati county and Vadeni, Braila county.

The characteristic ATR-FT-IR spectra of the vegetal tissues showed the presence of the following functional groups: alkynes, alcohols, phenols, amides I and II, carboxylic acids, and alkanes, which indicates the presence of lipids, proteins, carbohydrates and nucleic acids. In the case of wheat leaves, these peaks are much more pronounced compared to those identified in the spectrum of sunflower leaves (Figure 6.3). Functional groups specific to polysaccharides, phospholipids, hemicellulose, xyloglucans and pectin are present in plant tissues.

The results obtained by the ATR-FT-IR method on plant samples taken from Tulucesti, Sendreni and Vadeni were presented at the international conference [Moraru, S.-S. et al., 2019d] and partially published in [Moraru, S.-S. et al., 2019a].



(a)



(b)

Figure 6.3 Characteristic ATR-FT-IR spectra of some plants tissues collected from Tulucesti, Sendreni, Galati county and Vadeni, Braila county: (a) wheat leaves, (b) sunflower leaves (processed after [Moraru, S.-S. et al., 2019 a, d])

6.3. Soil mineralogical composition and morphological structure identification by scanning electron microscopy (SEM) coupled with energy dispersive X-ray spectrometry (EDX)

The semi-quantitative SEM-EDX technique results completed the ATR-FT-IR method, providing the determination of the following chemical elements from the soil and plant elemental composition: C, N, O, Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Gd, Fe, Co, Ni, Cu, Zn, Ga, and Hg. Figure 6.4a shows the SEM micrography, the EDX spectrum and the map of the elements present in the analyzed soil sample.

SEM micrographs show the presence of clay minerals, calcite, quartz and chemical element oxides, identified by spherical formations and flattened structures with rounded or shaped edges.

For the three analyzed territories, the results show a homogeneity of the elemental composition. The characteristic EDX spectra of the soils on the territory of Tulucesti show a percentage distribution of the elements in the order: O> Si> C> Al> Fe> Ca> K> N> Mg> Zn> Co> Ni> Ti> Na> Mn> P> Cr > S> Cl> Ga> Gd, for the 0-5 cm depth, and O> Si> C> Al> Fe> Ca> K> Mg> Gd> Ga> Ni> Co> Zn> Ti> Mn> Na> P > S> N> Cr> Cl, for the 5-30 cm depth. The percentage content of the elements identified in the soils from Sendreni commune decreases as follows: O> Si> C> Al> Ca> Fe> Hg> K> Gd> Mg> Ga> Cu> Zn> Ni> Co> Na> Ti> Mn> Cr > P> S (0-5 cm) and O> Si> C> Al> Ca> Fe> K> Mg> Ga> Zn> Gd> Cu> Co> Ti> Mn> Ni> Na> V> Cr> P > S (5-30 cm). On the territory of Vadeni the vertical distribution of the elements is similar to the previously mentioned territories: O> Si> C> Al> Fe> K> Ca> Gd> Ga> Zn> Mg> Cu> Co> Mn> Ti> Ni> Cr> Na> P> S (0-5 cm) and O> Si> C> Al> Fe> K> Ca> Co> Mg> Ti> N> Na> P> S (5-30 cm).

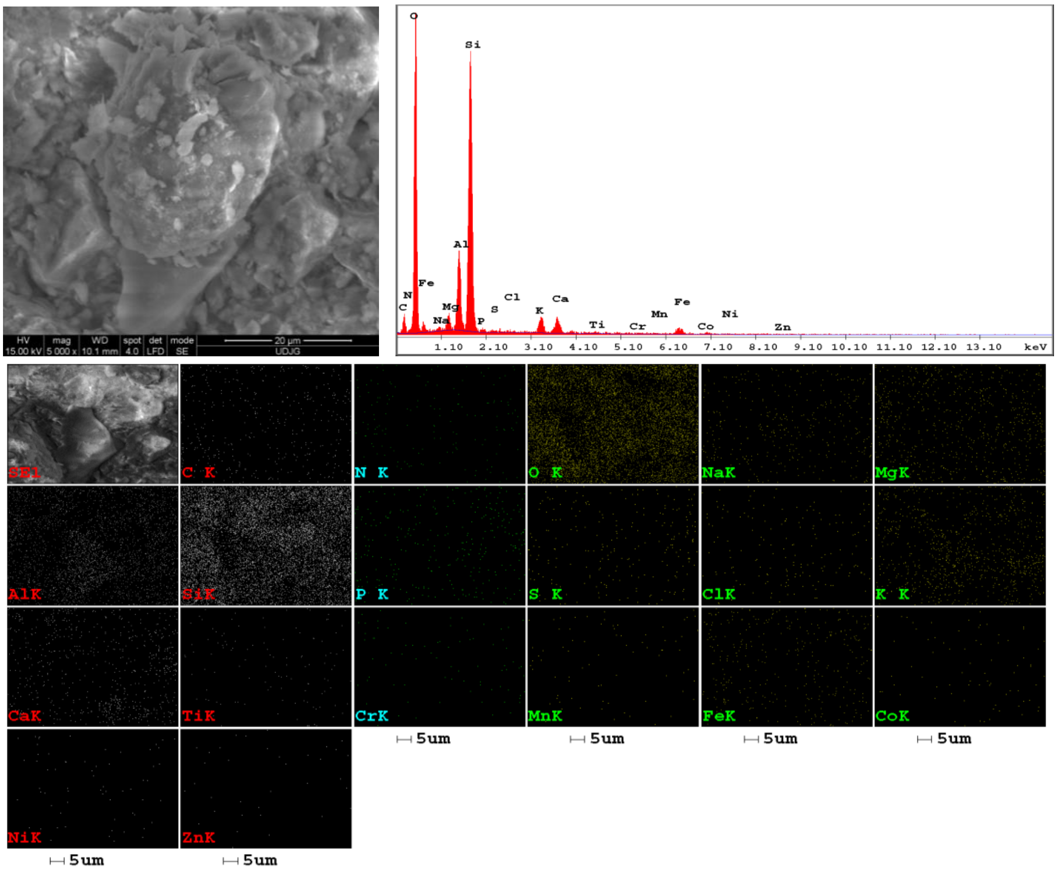
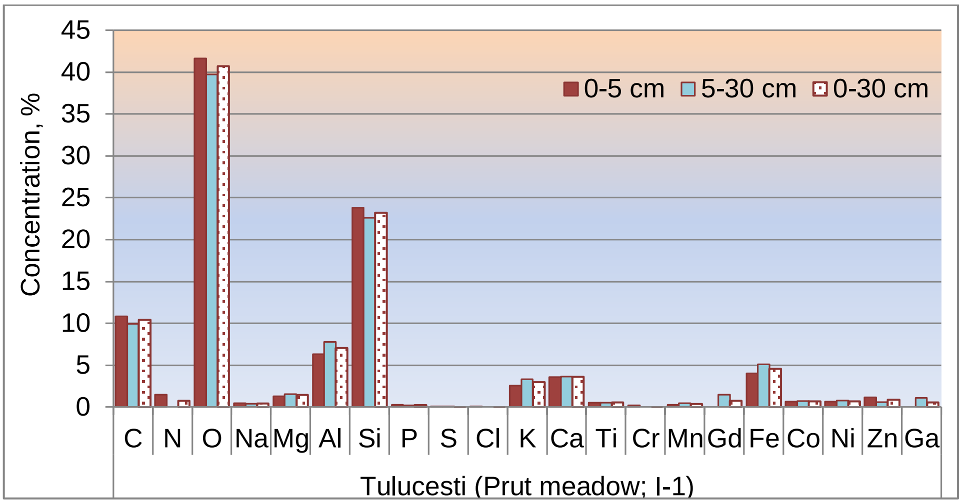


Figure 6.4a Characteristic SEM micrograph (5000x), EDX spectrum and map of the elements distribution of a soil sample from the territory of Tulucesti (extract from Figure 6.4 - thesis in extenso)

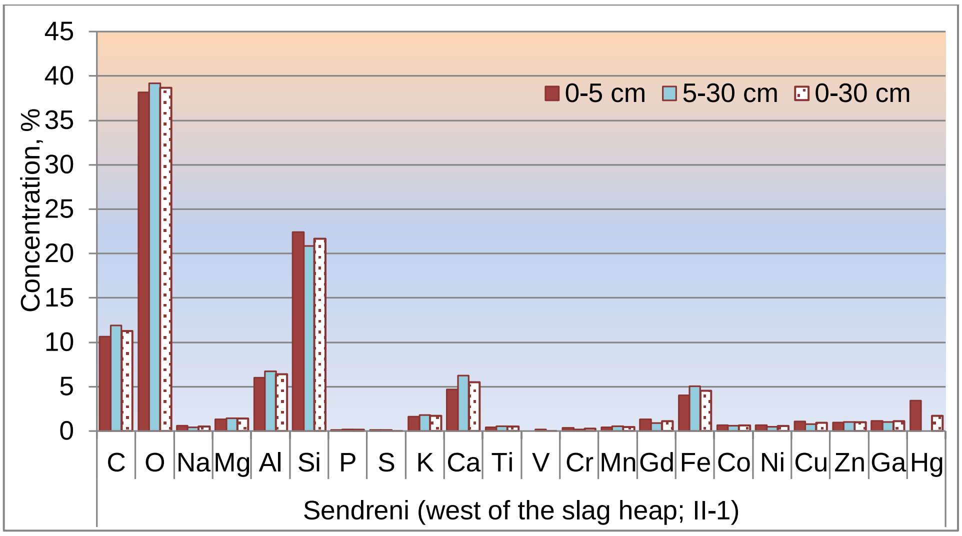
Of the 24 elements identified, O and Si have the highest concentration. Oxygen level in the agricultural 0-30 cm layer has an average value of 40.69 % in the soils from *Tulucesti*, 38.67 % for *Sendreni* commune and 32.47 % for *Vadeni* territory. The O content in the researched soils is slightly lower than the average value reported for the earth's crust (46 % [https://ptable.com/#Properties/Series]). In the agricultural 0-30 cm layer, Si registers an average of 23.22 % on *the territory of Tulucesti*, 21.64 % in *the Sendreni area* and 25.78 % at *Vadeni*.

In the 0-30 cm layer of agricultural soil, the carbon level is low, registering average values of 10.41 % on *the territory of Tulucesti*, 11.25 % in *the Sendreni area*, and 12.48 % at *Vadeni*. The level of C is much higher than the European average (2.48 %) reported by [Salminen, R., 2005] for the upper soil horizon and compared to the average value of this element in the earth's crust (0.18 % [https://ptable.com/#Properties/Series]).

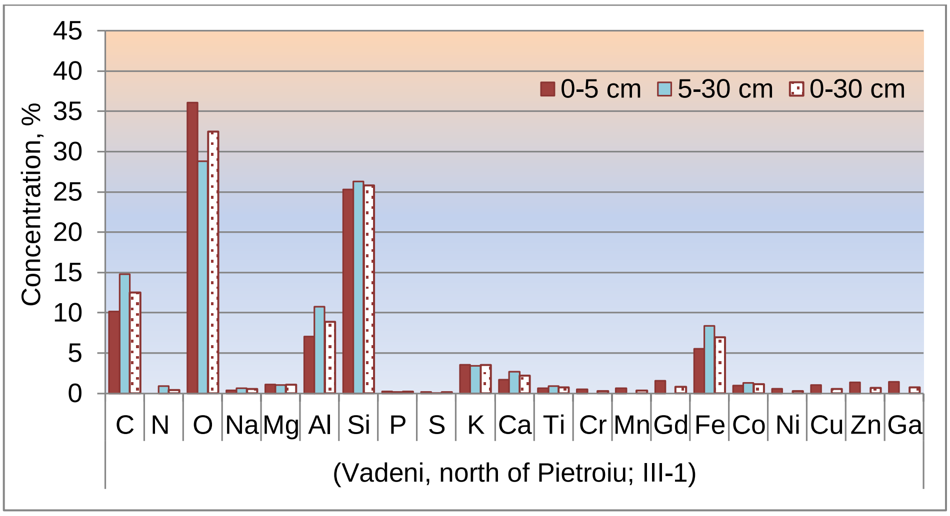
The EDX method allowed the identification of the alkaline elements (Na, Mg, K, Ca), primary and secondary elements with a role in plant nutrition (N, P and S), as well as heavy metals and rare elements (Cr, Mn, Fe, Co, Ni, Cu, Zn, Hg, Ga and Gd). Figure 6.5. show the percentage distribution of elements.



(a)



(b)



(c)

Figure 6.5 Distribution of the average elemental concentration obtained by the EDX method specific to the selected soils from the studied area: (a) Tulucesti (Prut meadow, I-1), (b) Sendreni (west of slag heap; II-1), (c) Vadeni (north of Pietroiu; III-1)

6.4. Plant mineralogical composition and morphological structure identification by scanning electron microscopy (SEM) coupled with energy dispersive X-ray spectrometry (EDX)

The application of the SEM-EDX technique for the investigation of plant tissues from the territories adjacent to the steel plant proved to be a practical method for morphological and structural analysis. Figure 6.6 shows the specific SEM micrographs (5000x), the EDX spectra and the maps of the elements of some samples of wheat, maize and sunflower caryopsis (Tulucesti, Sendreni communes, Galati county, and Vadeni, Braila county). Arrows and acronyms marked the morphological parts identified for the physiological sections of the grains/seeds according to [Heneen, W.K. and Brismar, K., 1987], [Shorstkii, I.A. et al., 2019], [Scheuer, P.M. et al., 2011]. From a morphological viewpoint, SEM micrographs highlighted the structural elements of plant tissues with physiological functions (cell walls, cell membranes) and organic substances (lipids, starch, proteins).

The EDX method indicated the presence of twenty-three macro, microelements and trace elements (C, O, N, P, K, Mg, Ca, Cu, Zn, Mn, Fe, Co, Na, Cl, Al, Si, S, Cr, Pb, Hg, Ni, Cd, Ti), which are also found in the soil they grew on. Figure 6.6a shows the SEM micrograph (5000x), the EDX spectrum and the map of the characteristic elements of a sample of wheat caryopsis..

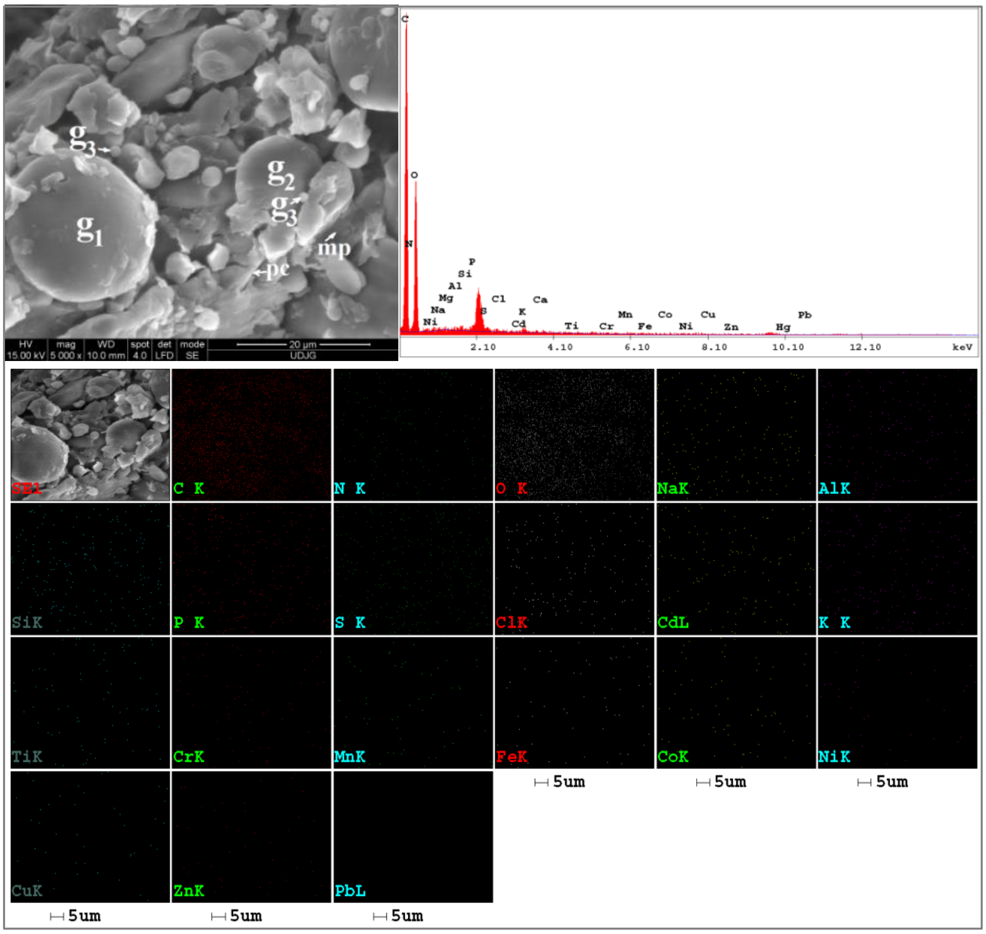


Figure 6.6a Characteristic SEM micrography (5000x), EDX spectrum and elements map of a wheat grain sample (*Triticum vulgare* Vill.) - large starch granule (g_1), medium starch granule (g_2), small starch granule (g_3), protein matrix (mp), cell wall (pc) - excerpt from Figure 6.6 (thesis in extenso)

6.5. Partial conclusions

ATR-FTIR and SEM-EDX techniques were employed for the morphology and mineralogical composition research of agricultural soils and plants grown on them.

By characteristic spectra, the ATR-FTIR technique provided the functional groups of minerals and organic compounds in soil and plants.

The spectral analysis of the soil showed the presence of clay (montmorillonite and kaolinite) and non-clay minerals (calcite, dolomite, gypsum, feldspars and quartz). In plant tissues, the functional groups of organic compounds such as cellulose, lignins, sugars, proteins, starch, et cetera have been identified.

The SEM-EDX technique allowed the estimation of the mineralogical composition and micro and macro elements on the surface of the specimen. By the SEM method, the micromorphological and structural analysis of the soil and plant samples was performed, confirming the results obtained by analyzing the ATR-FTIR spectra. The EDX method made it possible to get semi-quantitative information on the specimens' concentration of elements. C, N, O, Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Gd, Co, Ni, Cu, Zn, Ga, and Hg were found in soil and plant samples alike. As the EDX spectra and the maps of the elemental distribution highlighted, O and Si are the dominant elements in soil, which, by association with the other chemical elements present in the soil matrix, form complex compounds. C and O are also present in wheat, maize and sunflower tissues.

7. ASSESSMENT OF SOIL POLLUTION LEVELS THROUGH SIMPLE AND COMPLEX POLLUTION INDICES

7.1. Soil degree of pollution assessment through simple pollution indices

The following simple pollution indices, calculated according to the methods specified in subchapter 2.3.3 of this paper, assessed the pollution level of the soil in relation to the natural geochemical background for each of the two standard depths of 0-5 cm and 5-30 cm: Geoaccumulation Index (Igeo), Enrichment Factor (EF) and Pollution Index (PI).

Soil pollution assessment by the Geoaccumulation Index (Igeo)

The Igeo index estimates the soil pollution according to the ratio between the metal concentration in the analyzed samples and the average metal concentration in the natural background. Figure 7.1 show the variation of Igeo in the studied area.

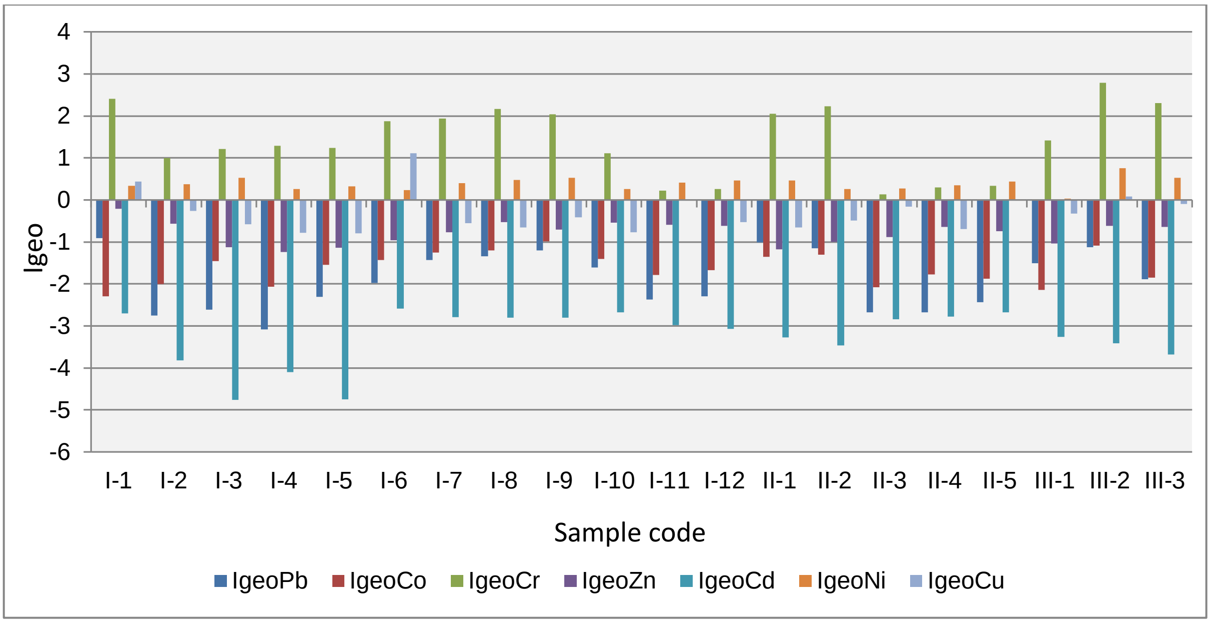


Figure 7.1 Average Geoaccumulation Index (Igeo) variation on the 0-30 cm depth of agricultural soils taken from the territories of Tulucesti, Sendreni, Galati county and Vadeni, Braila county

$Igeo_{Cd}$, $Igeo_{Co}$, $Igeo_{Pb}$, $Igeo_{Zn}$, and $Igeo_{Cu}$ recorded the lowest values, classifying the soil at class 0 (unpolluted).

Unlike the previously mentioned geoaccumulation indices, Cr and Ni have different states. The average $Igeo_{C}$r include the soil in class 3 (moderately-heavily polluted soil) in the sites where the average Cr concentration exceeds the alert threshold for sensitive uses on the 0-30 layer. The average values of IgeoNi place the soil in class 1 (unpolluted-moderately polluted).

Therefore the values for the geoaccumulation index decrease in the order: $Igeo_{Cd}$>$Igeo_{Pb}$>$Igeo_{Co}$>$Igeo_{Zn}$ >$Igeo_{Cu}$>$Igeo_{Ni}$>$Igeo_{Cr}$.

Soil pollution assessment by the Enrichment Factor (EF)

The Enrichment Factor (EF) was calculated according to Al, Ca and Mn. The results highlighted the following issues:

EF calculated according to Al shows the lowest Pb, Co, and Cd values for all the samples, which set the soil in *class 1* (deficiency to minimal enrichment). For the Cr, Zn, Ni, and Cu, the results show a variability, ranging the soil from *class 0* to *class 4*, respectively between deficiency-minimal enrichment (unpolluted soil-slightly polluted soil) and a very high enrichment (highly polluted soil).

Summarizing the results of the enrichment factors by Al, there is an evident accumulation of Cr and a less one of Cd, Co and Pb. EF decreases in order: EFCr> EFNi> EFCu> EFZn> EFCo> EFPb> EFCd, without differences between the three territories.

According to EF calculated according to Ca, the soil enrichment with Cd, Co, and Pb is from deficiency to minimal enrichment (unpolluted to slightly polluted-class 1) for all analyzed samples. EFCr ranges in *class 3* (significant enrichment), *class 2* (moderate enrichment) and *class 1* (deficiency to minimal enrichment). The EFZn values are in the moderate enrichment with Zn (*class 2*) and deficiency to minimal enrichment (class 1) ranges. Ni enrichment varies from a significant enrichment (*class 3*) to a moderate enrichment (*class 2*) and deficiency to minimal enrichment (*class 1*). From the Cu enrichment point of view, the soils are classified in *classes 1 and 2*.

The order of heavy metals enrichment of soils by Ca is: EFCr> EFNi> EFCu> EFZn> EFCo> EFPb> EFCd.

EF calculated according to Mn highlighted the following: EFCd registers the lowest results, all samples being in the range of deficiency-minimal enrichment (*class 1*). After EFPb and EFCo, the soil has the same order of the enrichment degree, which falls in the classes: moderate enrichment (*class 2*) and deficiency to minimal enrichment (*class 1*). Regarding the values for EFCr, the results showed a high (*class 4*), significant (*class 3*), moderate (*class 2*) and deficiency to minimal enrichment (class 1) enrichment of the soil. According to EFZn, zinc accumulated less (*class 2 and class 1*).

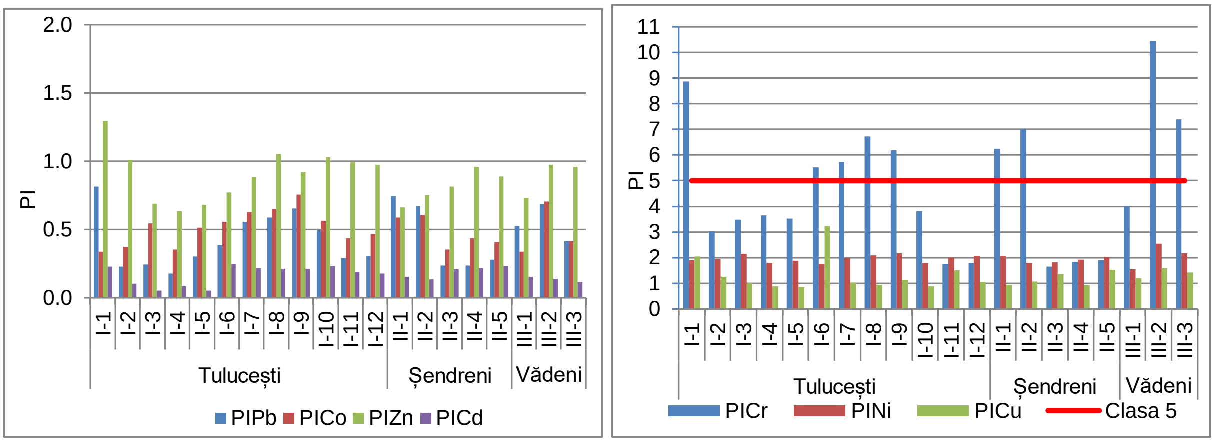
The soil has a significant (*class 3*) and a moderate (*class 2*) enrichment with Ni. The Cu enrichment is evident, the EFcu values being in the range of *class 3*. Punctually, the soil also has a medium enrichment (*class 2*) and deficiency to minimal enrichment (*class 1*).

From the viewpoint of EF evaluation according to Mn, the order of soil enrichment with metals is: EFCr> EFNi> EFCu> EFZn> EFCo> EFPb> EFCd.

The three situations' results illustrate that the EF of the soil samples from the territories of Tulucesti, Vadeni and Sendreni do not show significant differences. However, for some sampling points, the results lead to a different classification. The values obtained by reference to Al and Mn are closer for the elements Cr and Ni, as opposed to the values calculated as a function of Ca, which generates a lower enrichment classification. For Pb, Co, Cd, Zn, and Cu, the values calculated with Al and Ca are similar, in contrast to those obtained with Mn.

Soil pollution assessment by the Pollution Index (PI)

Soil pollution with heavy metals and trace elements was also assessed according to the Individual Pollution Index (IP), which shows which of the analyzed elements generates the highest level of contamination. Some complex indices calculation relies on this index (PIsum, PINemerow, PLI, PIN). Figures 7.3 and 7.4 show the PI and PISum values for the 0-30 cm agricultural layer.



(a) (b)

Figure 7.3 Average Pollution Index (PI) variation on the 0-30 cm depth of agricultural soils taken from the territories of Tulucesti, Sendreni, Galati county and Vadeni, Braila county: a) $PI_{Pb,\ Co,\ Zn,\ Cd}$, b) $PI_{Cr,\ Ni,\ Cu}$

The PICr results show that the 0-30 cm layer of agricultural soils is very heavily polluted (class 5), heavily polluted (class 4) and moderately polluted (class 3). Regarding Ni pollution, the values of the Pollution Index show low (class 2) and moderate (class 3) soil pollution. In the case of Cu, there is low soil contamination (class 2) and strong pollution (class 4). PIZn values classify the soil as unpolluted (class 1) and low polluted (class 2). Soils are unpolluted with Pb Co and Cd (class 1). The results obtained for PI follow the same trend of classification as EF: PI_{Cr}> PI_{Ni}> PI_{Cu}> PI_{Zn}> PI_{Co}> PI_{Pb}> PI_{Cd}.

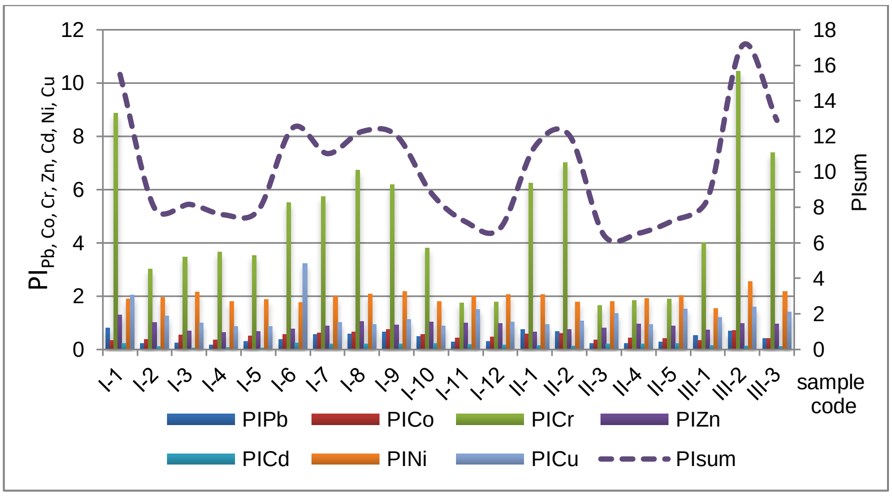


Figure 7.4 Distribution of Individual Pollution Index (PI) values in the 0-30 cm layer of agricultural soils and the variation of PI_{sum} by PI values

7.2. Soil degree of pollution assessment through complex pollution indices

Complex pollution indices assessed the overall level of soil contamination with heavy metals and its associated risk.

Soil pollution assessment by The New Pollution Index (PIN), Nemerow Pollution Index ($PI_{Nemerow}$) and Pollution Load Index (PLI)

The PINemerow index was calculated according to the PI values and divided the soil into three pollution classes. According to this index, on the 0-30 cm depth, the following classes of soil contamination are: heavily polluted soil (class 5) for I-1, III-2 sites, moderately polluted soil (class 4) for I-6, I-7, I-8, I-9, II-1 sites, slightly polluted soil (class 3) for I-2, I-3, I-4, I-5, I-10, III-1 sites, warning limit (class 2) for I-11, I-12, II-3, II-4, II-5 sites.

The PLI index, calculated as the geometric mean of the PI values, indicates that the soil is polluted in the I-1, I-6, I-8, I-9 and III-2 locations, while the other sampling sites are uncontaminated.

The assessment of soil contamination was also performed according to the PIN index by the formula mentioned in subchapter 2.3.3 of the thesis in extenso. The results showed that the soil is slightly contaminated (Class 3) in I-1, I-6, I-7, I-8, and I-9 sites, while the I-2, I-3, I-4, I-5, I-10, I-11, I-12, II-3, II-4, II-5 and III-1 locations are at the warning limit of contamination (Class 2). The variation of the average PIN values in the 0-30 cm layer, illustrated in Figure 7.5, shows that chromium has the highest contribution to the soil classification according to this index.

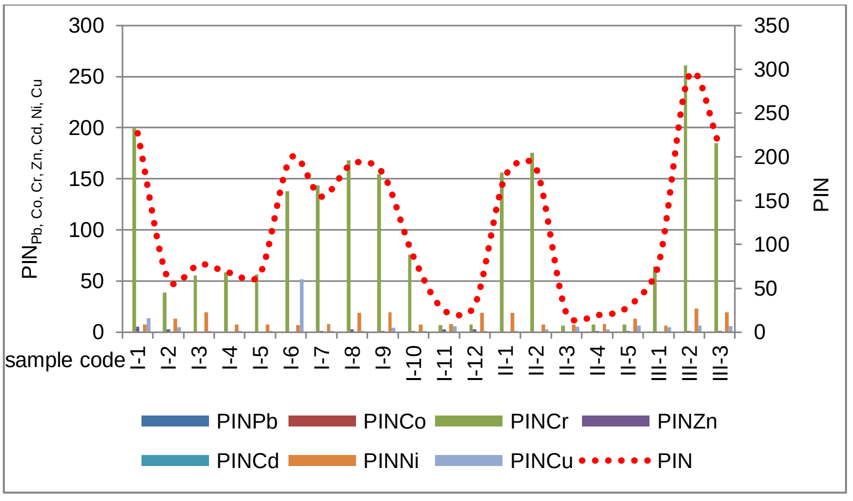


Figure 7.5 The New Pollution Index (PIN) distribution of values in the 0-30 cm layer of soils collected from Tulucesti, Sendreni, Galati county and Vadeni, Braila county

Soil pollution assessment by the Potential Ecological Risk Index (PERI)

The potential ecological risk was assessed according to the complex PERI index, which consists of the toxic or potentially toxic elements ecological risk factors (Er) sum. Er was calculated according to subchapter 2.3.3 of the thesis in extenso. The results showed that the level of heavy metals in the studied soils induces a low ecological risk. Er values are distributed as follows: ErNi> ErCr> ErCu> ErCd> ErPb> ErZn. The ecological risk increases with the concentration of the element in the soil and the toxicity caused by it. Figure 7.6 show the variation of PERI according to Er.

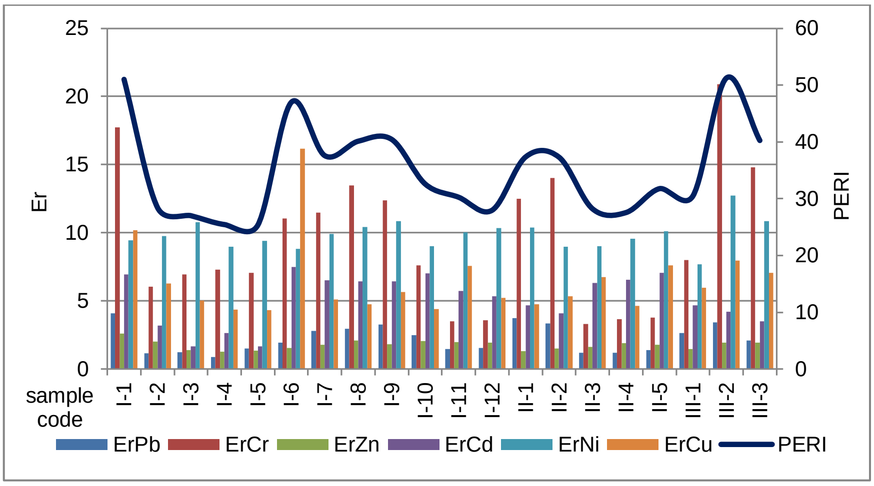


Figure 7.6 Variation of the average Potential Ecological Risk Index (PERI) depending on the Potential Ecological Risk Factor (Er) in the 0-30 cm layer of the agricultural soils taken from the territories of Tulucesti, Sendreni, Galati county and Vadeni, Braila county

Soil pollution assessment by the Contamination Severity Index (CSI)

Another complex index is the CSI, which provides information on the overall intensity of pollution according to the following factors: specific metal weight statistically calculated by Pejman, A. et al., 2015, ERL and ERM, according to subchapter 2.3.3 (thesis in extenso). According to this index, the soil is in class 2 of contamination (very low severity) for I-2 ... I-7, I-10 ... I-12, II-1 ... II-5, and III-1 locations, and in class 3 (low severity) for I-1, I-8, I-9, III-2, and III-3 sites. Figure 7.7 show the variation of this index.

The MERMQ index is connected with the CSI index and relies on the sum of the ratios between the concentration of the element in the soil and the ERM value of that element, according to subchapter 2.3.3 (thesis in extenso). This index provides an overview of the risk associated with anthropogenic heavy metal pollution and determines the percentage probability of metal toxicity. The results show that, in the studied area, the level of metals accumulated in the arable soils induces a medium risk, with a 21 % probability of toxicity.

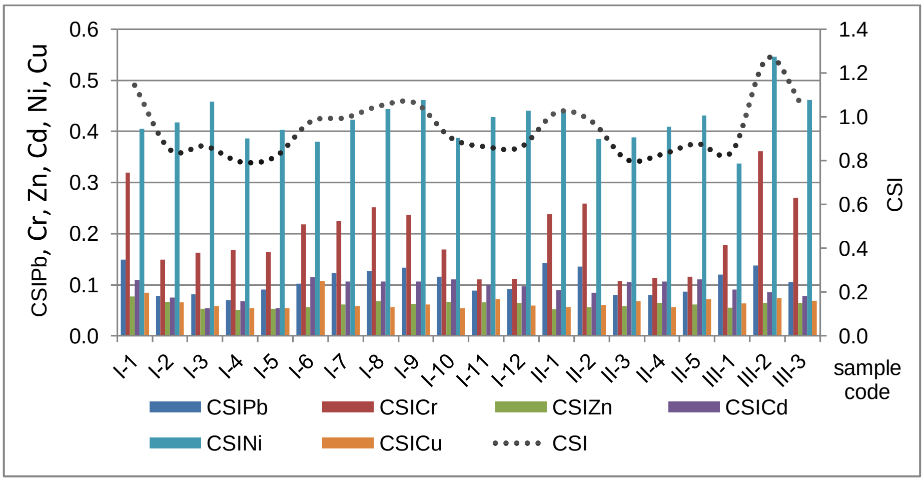


Figure 7.7 Variation of the average Contamination Severity Index (CSI) in the 0-30 cm layer of agricultural soils taken from the territories of Tulucesti, Sendreni, Galati county and Vadeni, Braila county

7.3. Partial conclusions

Soil pollution with chemical elements from anthropogenic activities is a topical issue, all the more so as it can pose major risks to food safety and human health.

The assessment of soil contamination with heavy metal was performed by calculating simple and complex pollution indices. The results highlighted the enrichment of the natural geochemical background with anthropogenic elements. Through these indices, it was possible to spatially analyze the level of contamination, both as a territorial distribution and on soil layers.

In conclusion, depending on the concentrations of heavy metals in the soil and the coefficients introduced in the calculation, the pollution indices classify the soil from unpolluted to very high polluted.

8. HEAVY METALS BIOACCUMULATION THROUGH THE SOIL-PLANT SYSTEM

8.1. Heavy metals pollution assessment of wheat

As maize, wheat is one of the most cultivated agricultural plants in the world (216 million ha, 757 million tons in 2017-2019 [Erenstein, O. et al., 2021]), so the study of nutrients, toxic and potentially toxic elements bioaccumulation, especially in the edible parts of the plant, is of great interest. Wheat cultivation and the fertilizers or phytosanitary treatments employment requires caution, particularly in polluted agricultural land so as in the industrial, mining, or high-traffic areas, because the potentially toxic elements tend to bioaccumulate in caryopsis too.

Under the presented ecopedological conditions, wheat is not a bioaccumulator of Cd, Co, Cr, Ni, Pb and Zn (Figure 8.1, 8.2). A particular situation is for the Cu bioaccumulation factor. In Tulucesti territory, the wheat does not show obvious bioaccumulation, except for the caryopsis related to the I-1-c (Tulucesti) site, for which BF has a value of 1.2443. Evident bioaccumulation is in the caryopsis from II-1-c and III-1-c locations and in the leaves from the III-1-f site (Figure 8.3). The deposition of toxic air particles or cooper phytosanitary treatment use could explain the cooper bioaccumulation in these sites. At the same time, zinc bioaccumulation is evident in the caryopsis from III-4-c and III-5-c locations.

The metals bioaccumulation in the wheat tissues generally follows the order leaves>caryopsis for Cd, Co, Cr, Ni and Pb, and caryopsis>leaves for Cu and Zn.

The elements bioconcentration in the wheat leaves decreases in the order: Zn> Cu> Cd> Pb> Ni> Co> Cr. In the case of caryopsis, the order of bioaccumulation is: Cu> Zn> Cd> Pb> Ni> Co> Cr.

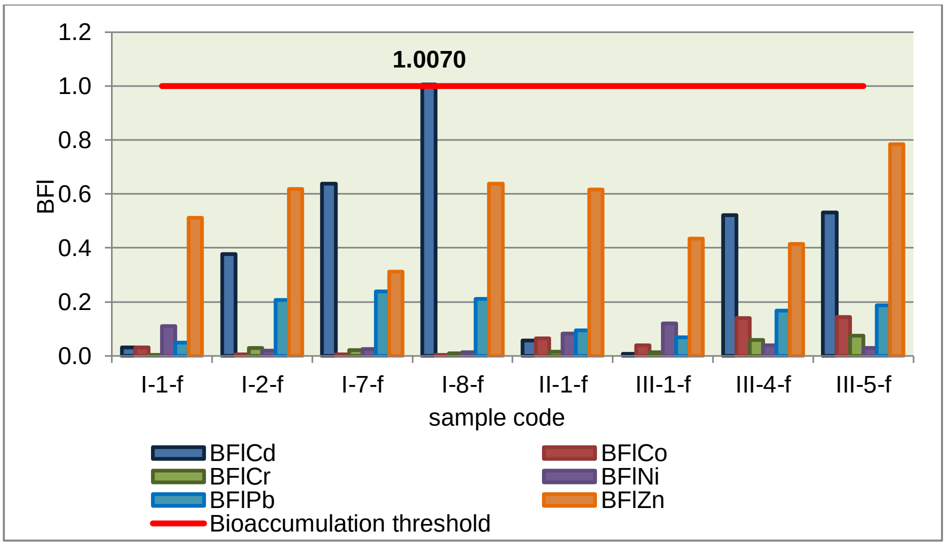


Figure 8.1 Bioaccumulation factor of Cd, Cr, Co, Ni, Pb and Zn in wheat leaves

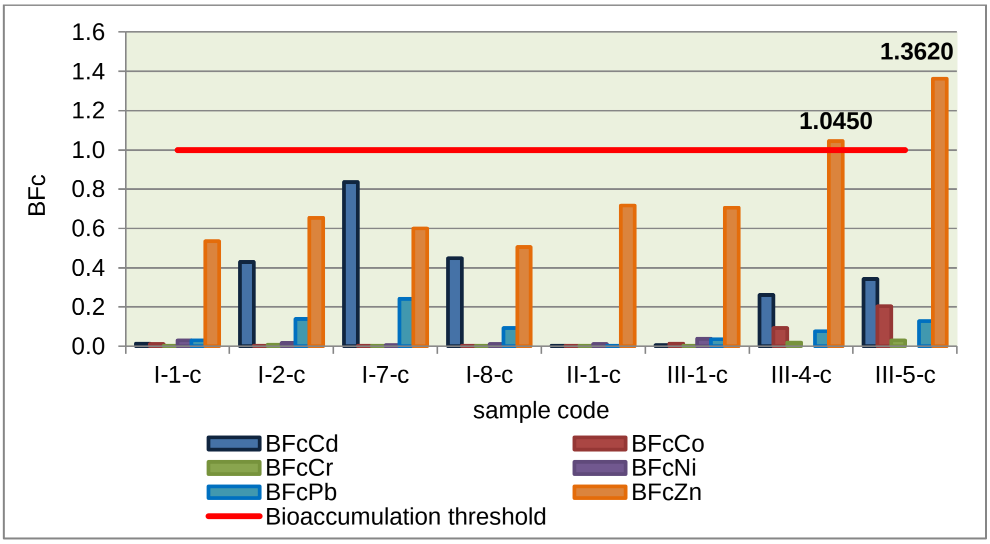


Figure 8.2 Bioaccumulation factor of Cd, Cr, Co, Ni, Pb and Zn in wheat caryopsis

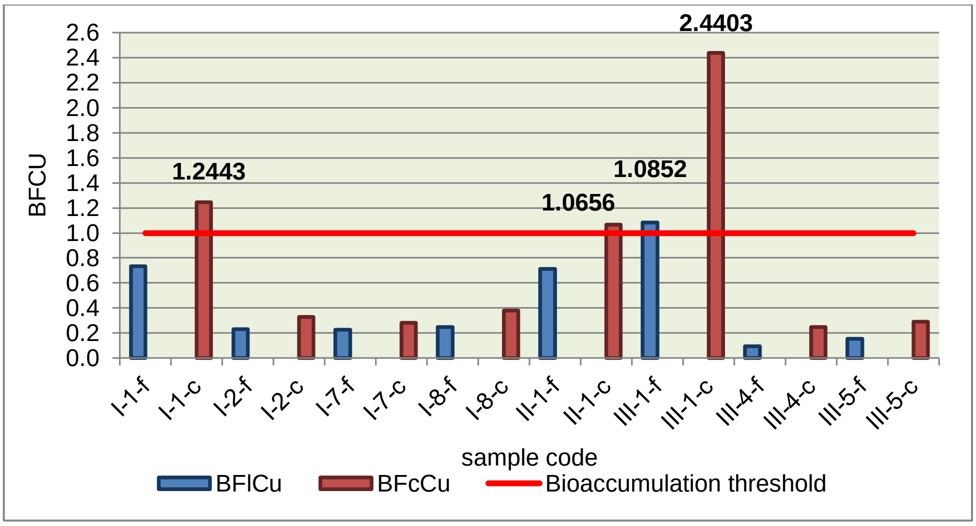


Figure 8.3 Bioaccumulation factor of Cu in wheat leaves and caryopsis

8.2. Heavy metals pollution assessment of maize

Maize has caught the attention of researchers as it is one of the plants with a phytoextraction potential for certain metals. Worldwide, 197 million ha were cultivated with this plant in 2017-2019, from which the harvest was 1137 million tons [Erenstein, O. et al., 2021]. It shows that it is one of the most consumed cereals. Although studies have shown that maize accumulates heavy metals in the root, stem and leaves, careful monitoring of crops is necessary since the whole plant is used for animal feed [Ion, V., 2010].

The heavy metals bioaccumulation factor for maize leaves and caryopsis, which is below 1, illustrates the low phytoextraction capacity of this plant in the ecological conditions it was grown (Figures 8.4, 8.5, 8.6).

The metals bioaccumulation maize tissues generally follow the order: leaves>caryopsis. The values of metals bioaccumulation factor for maize leaves decrease in the order: Cu>Zn>Cd>Pb>Co>Cr>Ni. In the case of caryopsis, the order of bioaccumulation is: Zn>Cd>Pb>Cu>Co Cr>Ni.

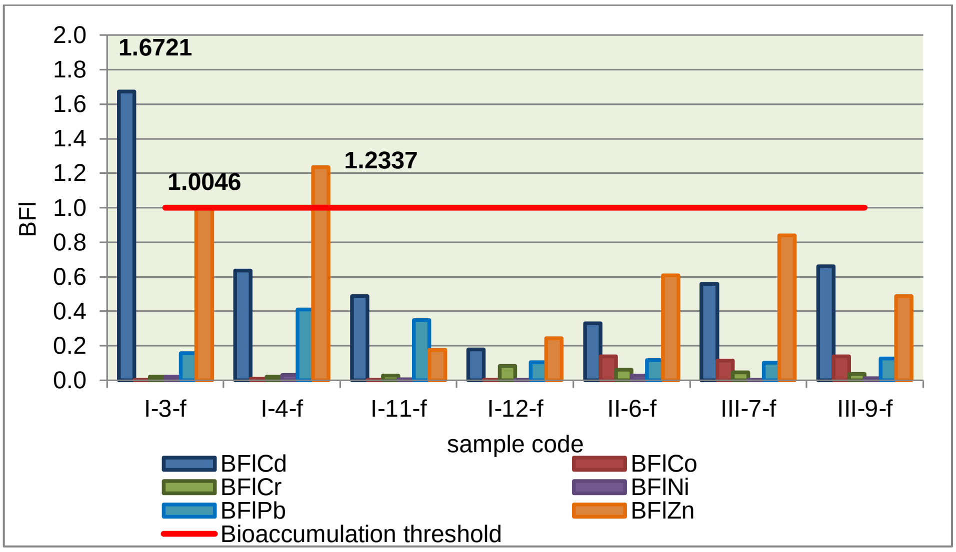


Figure 8.4 Bioaccumulation factor of Cd, Cr, Co, Ni, Pb and Zn in maize leaves

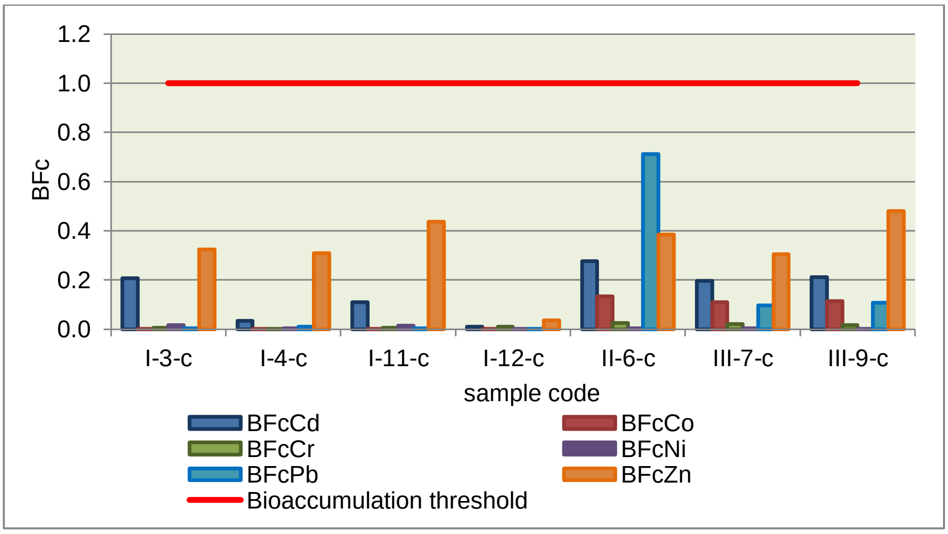


Figure 8.5 Bioaccumulation factor of Cd, Cr, Co, Ni, Pb and Zn in maize caryopsis

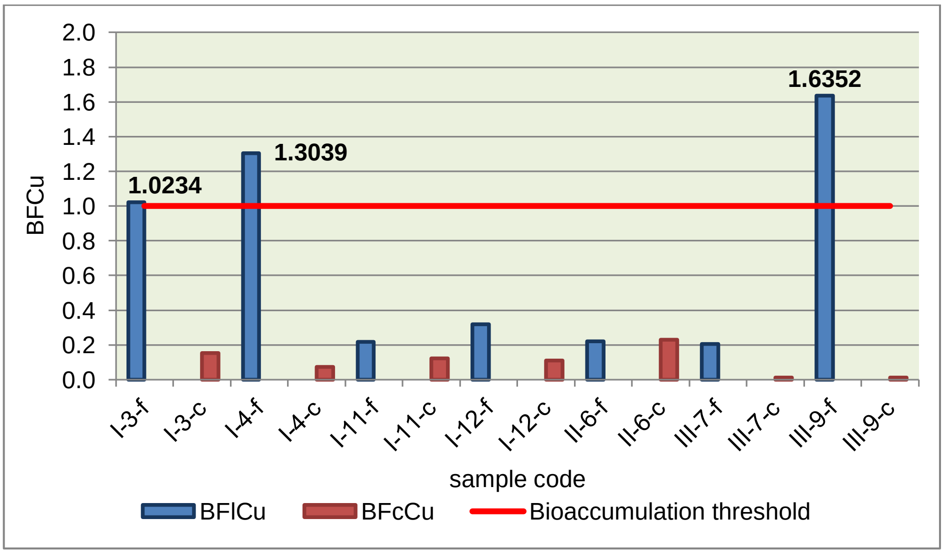


Figure 8.6 Bioaccumulation factor of Cu in maize leaves and caryopsis

8.3. Heavy metals pollution assessment of sunflower

Sunflower is an appreciated plant, both in terms of nutritional qualities [Khan, S. et al., 2015] and for the production of biofuel [Antolin, G. et al., 2002], [Tutunea, D . et al., 2018]. Many studies have shown its phytoextraction capacity mainly in the root and the stem.

The metals bioaccumulation factors for the sunflower sections show a disseminated trend of Cu, Cd and Zn accumulation in leaves and achenes (Figures 8.7, 8.8, 8.9, 8.10). Overall, FB of leaves decreases as follows: Cu<Cd<Zn<Pb<Co<Ni<Cr, and of caryopsis, in the order: Cu<Cd<Zn<Ni<Pb<Cr<Co..

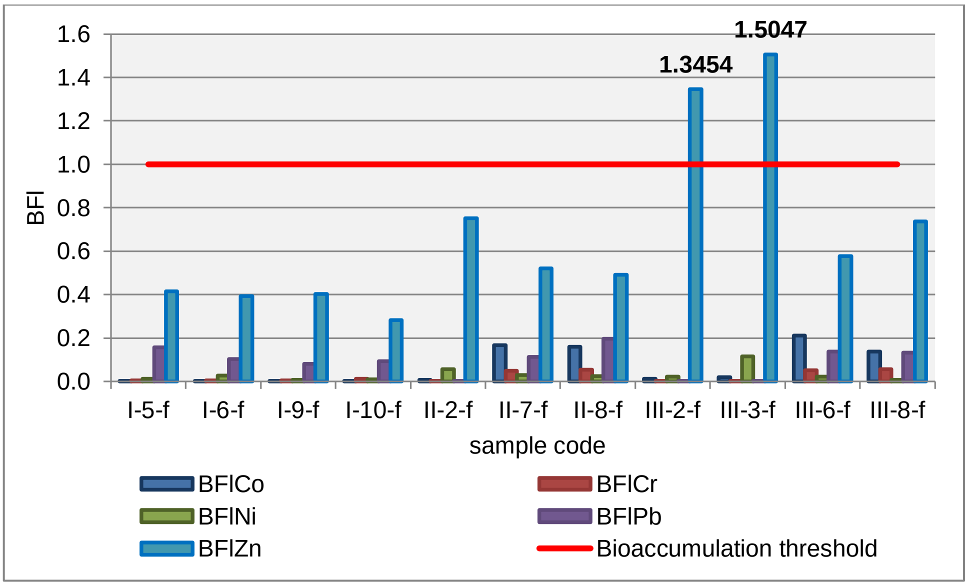


Figure 8.7 Bioaccumulation factor of Cr, Co, Ni, Pb and Zn in sunflower leaves

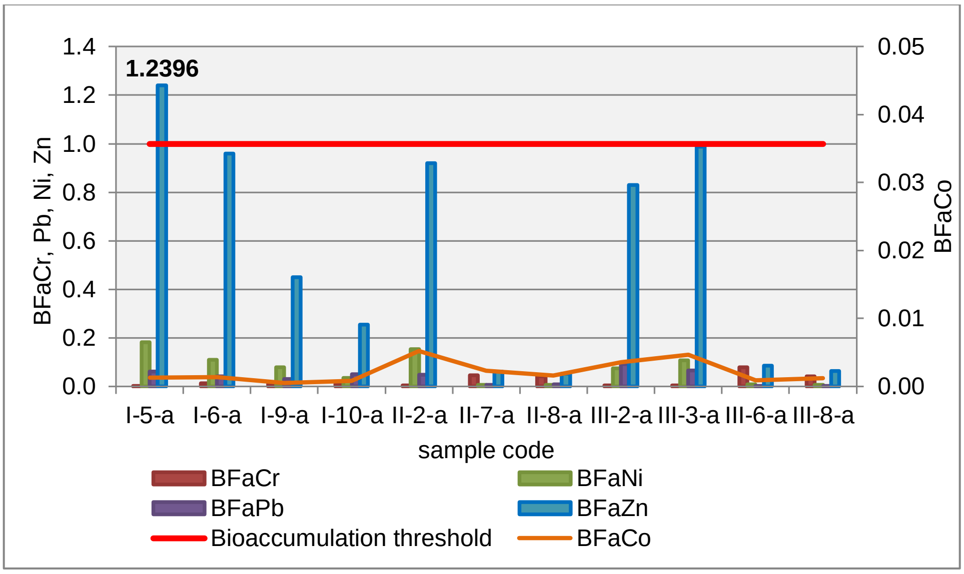


Figure 8.8 Bioaccumulation factor of Cr, Co, Ni, Pb and Zn in sunflower achenes

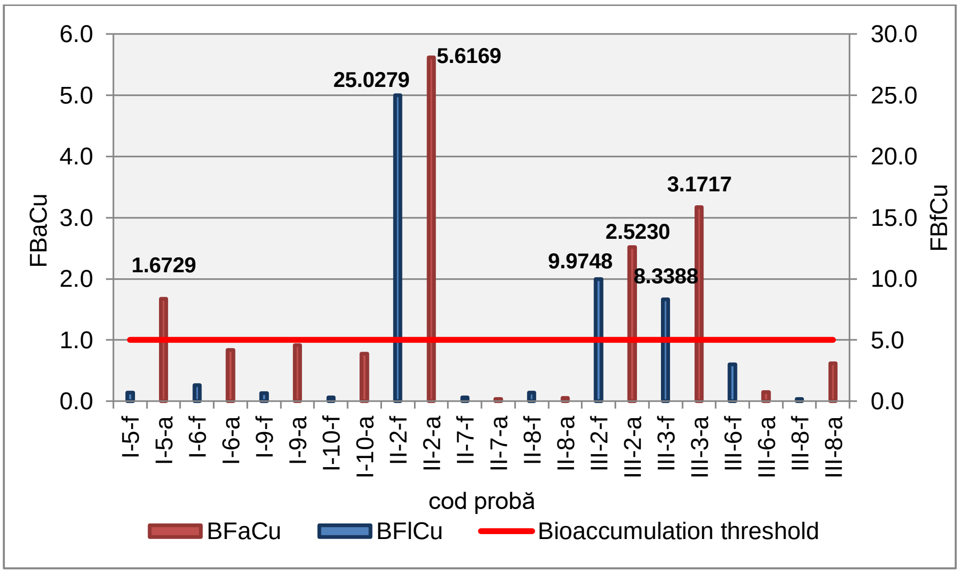


Figure 8.9 Bioaccumulation factor of Cu in sunflower leaves and achenes

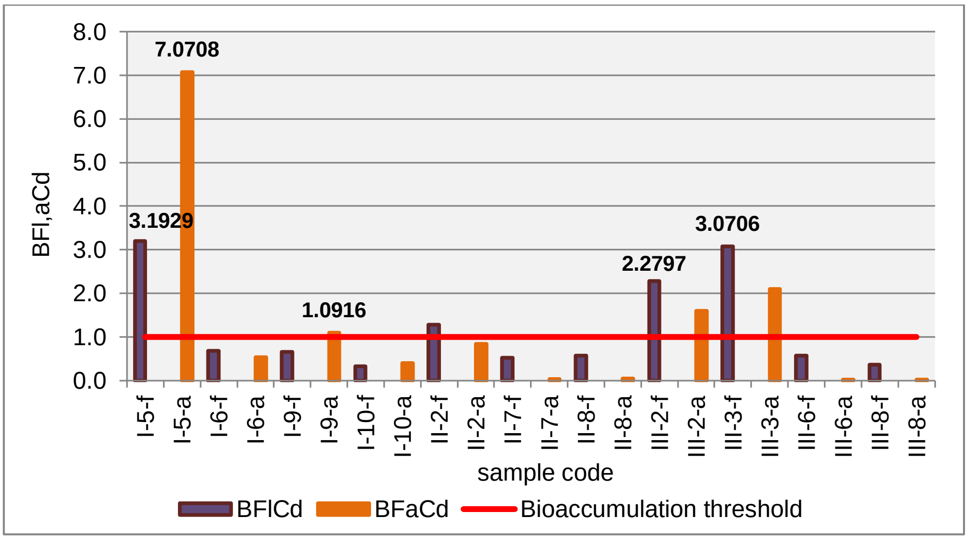


Figure 8.10 Bioaccumulation factor of Cd in sunflower leaves and achenes

8.4. Partial conclusions

The bioaccumulation factors of heavy metals in wheat, maize and sunflower were calculated by the elemental concentrations determined using the AAS HR-CS and ICP-MS methods. The results highlighted a tendency of higher heavy metals accumulation in the leaves than in caryopsis/achenes. The agricultural plant species showed a selectivity by their morphological structure and local conditions in terms of micro and macro elements bioconcentration.

9. FINAL CONCLUSIONS. PERSONAL CONTRIBUTIONS AND FUTURE RESEARCH DIRECTIONS

Final conclusions

The doctoral thesis ***"Research on iron and steel works industry impact on soil edaphic and vegetal potential in the adjacent areas "*** aimed to the complex analysis of the soil and the most cultivated plants (wheat, maize and sunflower), by combining classical methods of investigation with advanced spectroscopic and nuclear techniques for environmental analysis.

The complementary techniques allowed the interdisciplinary assessment of the agricultural soil contamination with micro, macroelements and trace elements, up to a depth of 30 cm, and the quantification of these elements in plants.

This paper contributes to the soil and vegetal state database of agricultural lands in the administrative-territorial units of Tulucesti and Sendreni in Galati county and Vadeni in Braila county, located in the vicinity of the Galati Iron and Steel Works.

The soil assessment required the investigation of the primary macroelements (nitrogen, phosphorus, potassium) and of other soil parameters that influence the mobility of nutrients, toxic or potentially toxic elements (pH, the content of organic matter, $CaCO_3$ and soluble salts, and the parameters involved in the cation exchange properties). Furthermore, the physical and hydro-physical parameters of the soil (bulk density, particle size composition, total porosity, aeration porosity, field water capacity, wilting coefficient, total water capacity, drainage capacity, and easily accessible water capacity) were evaluated.

The complementary advanced spectroscopic (ATR-FT-IR, HR-CS AAS, ICP-MS) and nuclear (PIGE, PIXE, RBS) techniques allowed the multi-elemental analysis of the samples taken from the territories adjacent to the industrial complex.

The atomic absorption spectrometry (AAS) method, in flame and on a graphite furnace, quantified the concentration of elements with a specific role in biochemical processes, which can pose risks to human health (Al, Ca, Cd, Co, Cu, Cr, Mn, Ni, Zn, Pb). The results showed an accumulation of Cr, Cu and Ni in concentrations that exceed the normal value regulated in Romania, which indicates the anthropogenic impact on agricultural soils. As regards the content of these elements in cultivated plants, the soil-plant connection and the selectivity of bioaccumulation by species and plant section are found.

The analysis performed by the ATR-FT-IR technique allowed the identification of the characteristic functional groups of the minerals in the soil and plants. The ATR-FT-IR spectra revealed the presence of clay (montmorillonite and kaolinite) and non-clay minerals (quartz, feldspar, calcite, dolomite and gypsum), which influence the absorption and availability of nutrients and pollutants. Functional groups of the organic compounds such as cellulose, lignins, sugars, proteins, and starch were found.

The ATR-FT-IR method results were supplemented by those of the SEM-EDX technique, which provided a detailed analysis of the mineralogical composition and micro and macro elements concentration in the environmental samples microsurfaces. The SEM method allowed the micromorphological and structural analysis of soil and plant samples. SEM micrographs at various magnifications showed the presence of clay and non-clay minerals, and oxides in the soil matrix, marked by grains, flattened rounded and shaped edges structures. SEM micrographs of plants provided information on structural elements in the tissues, which fulfil physiological functions (cell walls, cell membranes), and organic compounds (lipids, starch, proteins). Therefore, the information generated by the SEM technique confirms the results obtained by the ATR-FT-IR spectra. The SEM-EDX method made it possible to reveal the semi-quantitative concentration of the elements in the samples. By this method, C, N, O, Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Gd, Co, Ni, Cu, Zn, Ga, and Hg were identified both in soil and plant samples. The EDX spectra and the maps of the elemental distribution highlighted the presence of O and Si in the soil, which associate with the other chemical elements to form complex compounds in the soil matrix. Wheat, maize and sunflower tissue's main elements are C and O, usually present in the organic matrix.

The IBA methods (PIGE and PIXE) allowed the complementary determination of the chemical elements in the agricultural soils and plant sections. PIXE analysis consisted of the irradiation of the samples in a vacuum and with an external beam, which allowed the identification of the elements with $Z>20$ (Ca, Ti, Cr, Mn, Fe, Ni, Zn), and some elements with $Z<20$ (Na, Mg, Al, Si, P, S). PIGE technique completed this method by the quantification of the light elements (F, Al, Na, Mg, Si, Cl, K). Furthermore, through all the advanced analytical methods, the PIGE technique is the only one that detected the F element, toxic even in low concentrations.

ICP-MS method offered the information on the isotopic composition of the soils and plants, which is the first scientific contribution to the isotopic signature database of the research region. The isotope ratio proved the anthropogenic impact of the neighbouring industrial activity and other contamination sources (traffic emissions, use of fertilizers and phytosanitary products, storage of industrial waste, et cetera).

The quantification of soil pollution with heavy metals involved the simple and complex pollution indices assessment, which showed the enrichment with anthropogenic elements of the natural geochemical background. In this way, it was possible to spatially analyze the level of contamination, both as a territorial distribution and on soil layers.

The study of toxic or potentially toxic elements accumulation in the leaves and caryopsis/achenes of wheat, maize and sunflower through the bioaccumulation factor accomplished the complex soil pollution analysis. This thesis contributes to the first data regarding the transfer of metals from soil to plants cultivated in the Galati-Braila region.

Personal contributions and future research directions

Personal contributions

The doctoral thesis ***"Research on iron and steel works industry impact on soil edaphic and vegetal potential in the adjacent areas"*** objective was the complex analysis of soil to its physical, hydro-physical and chemical properties, as well as in terms of mineralogical composition.

This study gives the first information on ATR-FT-IR and SEM-EDX mineralogical and microstructural analysis of wheat, maize and sunflower cultivated in Sendreni, Tulucesti and Vadeni, Galati-Braila region.

Furthermore, it provides the first data on the isotopic signature of agricultural soils, as well as of crop plants (wheat, maize, sunflower) from Sendreni and Tulucesti territory, Galati county and Vadeni, Braila county, through the stable isotopes ratio of Cd, Co, Cu, Fe, Ni, Pb, and Zn.

Advanced complementary spectroscopic and nuclear methods (HR-CS AAS, PIXE, PIGE, RBS) were applied to assess the micro, macroelements and trace element levels in the leaves, caryopsis and achenes of wheat, maize and sunflower.

This study is the first scientific contribution regarding the micro and macro elements' bioaccumulation capacity in the wheat, maize and sunflower tissues from the agricultural areas adjacent to the Galati Iron and Steel Work.

Future research directions

The main future research directions will focus on the following issues:

- Study of soil and crops contamination with other categories of pollutants not included in this study (aromatic and polyaromatic hydrocarbons, petroleum hydrocarbons, organochlorine organic compounds, pesticides, et cetera);
- Extension of the study to other plant species cultivated near Galati Iron and Steel Works, intended for human and animal consumption;
- The assessment of the accumulation of the toxic elements in the medicinal plants of the Galati-Braila area's natural flora;
- Extending the research to other categories of agricultural land (pasture, hayfield, vineyard, orchard);
- Evaluation of the groundwater and surface water quality, intended for human and animal consumption and used for irrigation works;
- Study of heavy metals concentrations in other plant sections (root, stem) of wheat, maize and sunflower (ongoing activity);
- Employment of the nuclear analysis techniques (PIGE, PIXE, RBS) for the study of other categories of environmental samples;
- Assessing the degree of population exposure to pollutants in the vicinity of the steel plant industry by the carcinogenic risk indices (ongoing activity);
- Extending the study of agroecosystem pollution on the entire surface of Galati and Braila counties;
- Continuation of the study of macro and microelements stable isotopes in soil and plants, as well as the initiation of research on the isotopic signature of the pollution sources in the Galati-Braila area;

- Gamma-spectrometric study of the activity of the natural radionuclide present in soil, plants and water;
- Creating a county-wide database on soil contamination with toxic elements;
- Achievement of geochemical atlas of Galati and Braila counties.

APPENDICES

Appendix 2.1a Delimitation of soil depth sampling on Tulucesti territory, Galati county: (a) sample I-1a/b, (b) sample I-2a/b, (c) sample I-3a/b, (d) sample I-4a/b, (e) sample I-5a/b, (f) sample I-6a/b, (g) sample I-7a/b, (h) sample I-8a/b, (i) sample I-9a/b, (j) sample I-10a/b, (k) sample I-11a/b, (l) sample I-12a/b (source: Moraru S.S.)


(a)


(b)


(c)


(d)


(e)


(f)


(g)


(h)


(i)


(j)


(k)


(l)

Appendix 2.1b Delimitation of soil depth sampling on Sendreni territory, Galati county: (a) sample II-1a/b, (b) sample II-2a/b, (c) sample II-3a/b, (d) sample II-4a/b, (e) sample II-5a/b, (f) sample II-6a/b, (g) sample II-7a/b, (h) sample II-8a/b (source: Moraru S.S.)


(a)


(b)


(c)


(d)


(e)


(f)


(g)


(h)

Appendix 2.1c Delimitation of soil depth sampling on Vadeni territory, Braila county: (a) sample III-1a/b, (b) sample III-2a/b, (c) sample III-3a/b, (d) sample III-4a/b, (e) sample III-5a/b, (f) sample III-6a/b, (g) sample III-7a/b, (h) sample III-8a/b, (i) sample III-9a/b

(source: Moraru S.S.)

(a)

(b)

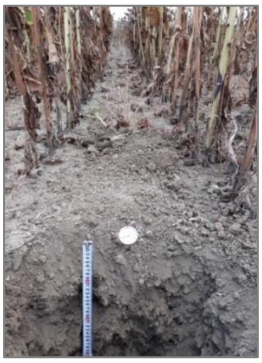
(c)

(d)

(e)

(f)

(g)

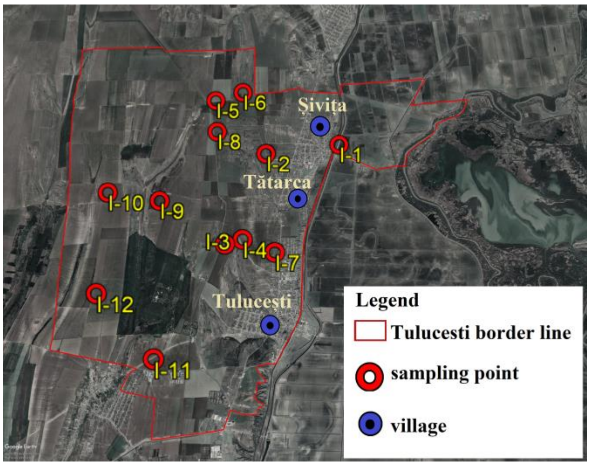


(h)

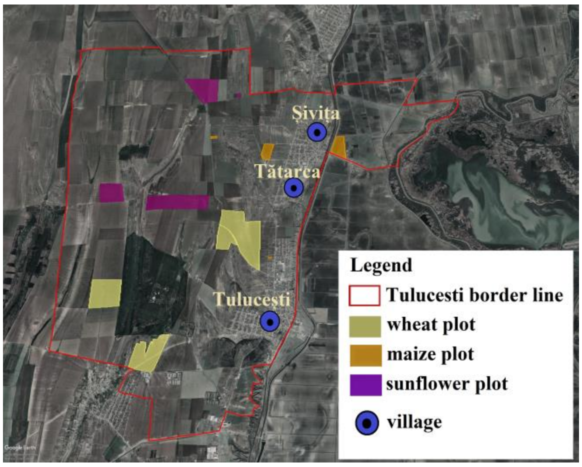
(i)

Appendix 2.2a Sampling lots location in Tulucesti territory, Galati county: (a) identification of plant samples, (b) agricultural culture

(source: processed after Google Earth Pro)



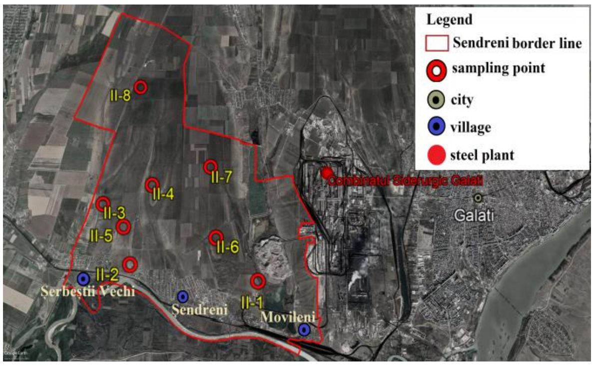
(a)



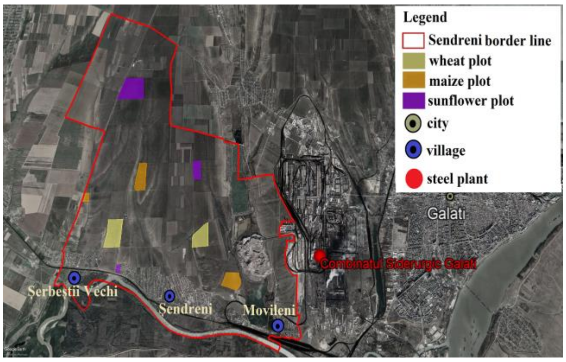
(b)

Appendix 2.2b Sampling lots location in Sendreni territory, Galati county: (a) identification of plant samples, (b) agricultural culture

(source: processed after Google Earth Pro)



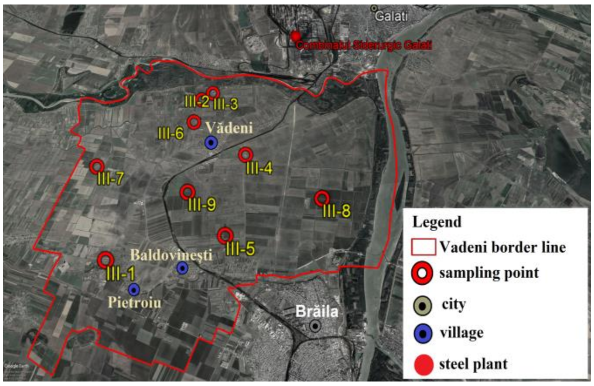
(a)



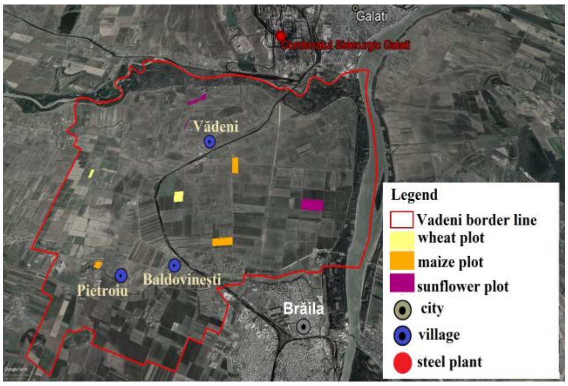
(b)

Appendix 2.2c Sampling lots location in Vadeni territory, Braila county: (a) identification of plant samples, (b) agricultural culture

(source: processed after Google Earth Pro)

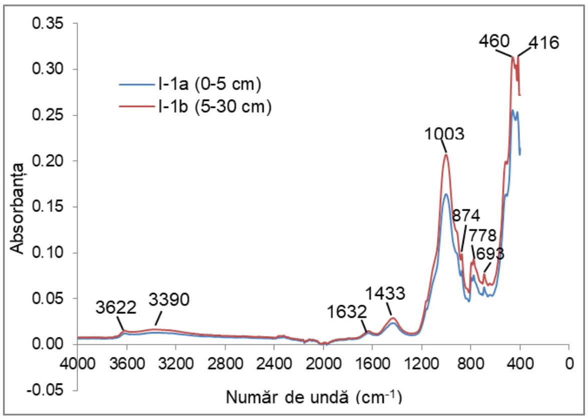


(a)

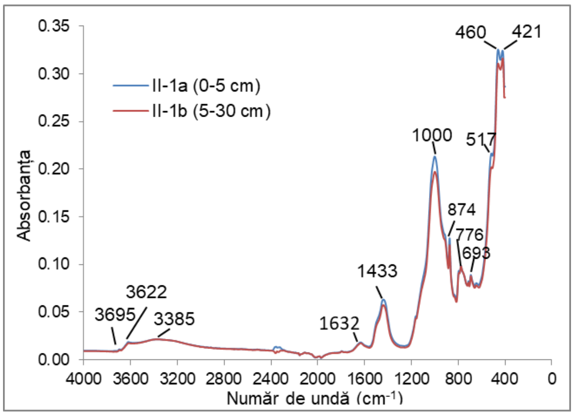


(b)

Appendix 6.1 Characteristic ATR-FT-IR spectra of soil samples taken from the territories: (a) Tulucesti (I-1a, I-1b), (b) Sendreni (II-1a, II-1b - b’; II-2a, II-2b - b”) și (c) Vadeni (III-1a, III-1b - c’; III-2a, III-2b - c”, III-3a, III-3b - c’”)



(a)



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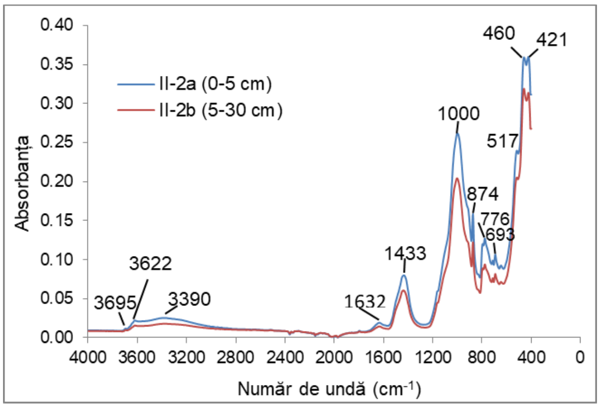
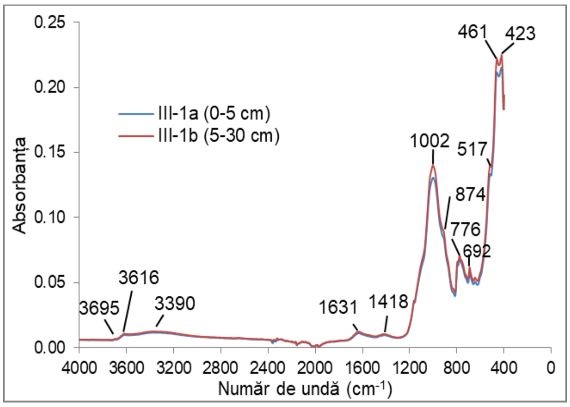
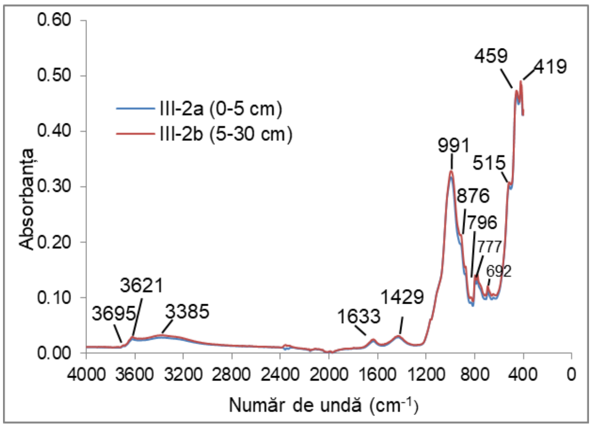


(b)”



(c)’



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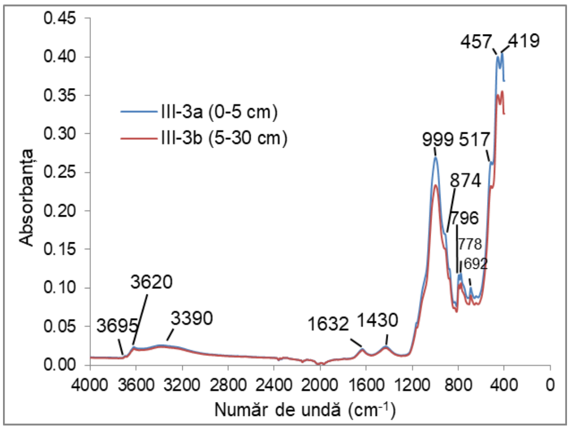

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Moraru, **S.S.**, Simionică, A., Simionică, R.L., Manea, M.E., *Reconstrucția ecologică a solurilor aferente pășunilor din bazinul Chinejei, în segmentul Frumușița-Foltești, județul Galați*, Pedopeisaje naturale și antropizate din estul României. Ghidul celei de-a XXIX-a ediții a simpozionului "Factori și procese pedogenetice din zona temperată", coord. Rusu C., Bulgariu D., Vasiliniuc I., Editura Universității "Al. I. Cuza" din Iași, p. 190-212 (2019), ISBN 978-606-714-549-6, ISSN 1582-4616.

Arbanas (Moraru), **S.-S.**, Ene, A., *Nutrient stocks study in agroecosystems located near the steel industry, Galati, Romania*, Annals of "Dunarea de Jos" University of Galati, Mathematics, Physics, Theoretical Mechanics, FASCICLE II, YEAR XI (XLIII), No. 2, p. 82-93 (2020), Galati University Press, ISSN 2067-2071. https://www.gup.ugal.ro/ugaljournals /index.php/math/article/view/4045

SCIENTIFIC PAPERS PRESENTED AT CONFERENCES

A. National conferences

Sorina Simona Arbanaș (Moraru), Antoaneta Ene, Steluța Gosav, *Investigarea compoziției mineralogice a solurilor agricole din aria de influență a Combinatului Siderurgic Galați prin Spectroscopie ATR-FTIR*, Conferință științifică cu participarea elevilor "Fizica medicală: simbioză între Fizică, Medicină și Mediu", Galați, Secțiunea 2: Mediul înconjurător: teorie, instrumente, aplicații, 28 februarie 2019, Universitatea " Dunărea de Jos" din Galați, Facultatea de Științe și Mediu, prezentare orală, p. 3 din Program și p. 43 din Rezumatele conferinței.
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B. International conferences

Moraru Simona, Moraru Ionel, Popescu Adina, Contoman Maria, *Pedological Study Regarding the Ecological Reconstruction of Pastures Soils from the Lower Chineja Basin, Galaţi County*, Simpozionul de Agricultură și Industrie Alimentară, Iași, Universitatea de Științe Agricole și Medicină Veterinară "Ion Ionescu de la Brad" din Iași, Facultatea de Agricultură, 19-20 octombrie 2017, First Section: Water and Soil, poster, p. 11 din Program. http://www.uaiasi.ro/simpozion/_simpozion2017/fisiere/Program--2017.pdf

Sorina-Simona Arbanaș (Moraru), Alina Simionică, Antoaneta Ene, *Agricultural Potential of Chernozems Near the Iron And Steel Integrated Works of Galati, in the Perimeter of the Territorial Administrative Unit of Sendreni, Galati County*, 6th Edition of Scientific Conference of Doctoral Schools SCDS-UDJG -Perspectives and challenges in doctoral research, Galați, Section 2: Advanced investigation methods in environment and biohealth, Universitatea "Dunărea de Jos" din Galați, Romania, 7-8 iunie 2018, prezentare orală OP.2.1, p. 11 din Program, p. 51 din Book of Abstracts.
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***Awarded presentation - First prize - Scientific Conference of the Doctoral School of "Dunărea de Jos" University of Galati, 6th edition,* "Dunărea de Jos" University of Galați, Romania, 7-8th of June 2018.**

Sorina-Simona Arbanaș (Moraru), Antoaneta Ene, Alina Simionică, *Pedological Study of Pasture Soils in the Southern Part of Galati County for Ecological Reconstruction*, 6th Edition of

Scientific Conference of Doctoral Schools SCDS-UDJG -Perspectives and challenges in doctoral research, Galați, Section 2: Advanced investigation methods in environment and biohealth, Universitatea "Dunărea de Jos" din Galați, Romania, 7-8 iunie 2018, poster PP.2.17, p. 27 din Program, p. 171 din Book of Abstracts.
http://www.cssd-udjg.ugal.ro/files/2018/05_Program_detaliat_al_conferintei_2018.pdf
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***Awarded presentation - Honourable mention - Scientific Conference of the Doctoral School of "Dunărea de Jos" University of Galati, 6th edition,* "Dunărea de Jos" University of Galați, Romania, 7-8th of June 2018.**

Sorina-Simona Arbanaş (Moraru), Alina Simionică, Răzvan-Laurenţiu Simionică, Mirela-Elena Manea, Georgiana-Elena Lupu, *Stadiul actual al cunoaşterii fertilităţii solurilor din terenurile cu folosinţa arabil, în unitatea de terasă a comunei Tuluceşti, judeţul Galaţi*, a XXVIII-a ediție a Simpozionului cu participare internațională "Factori și procese pedogenetice din zona temperată" cu tema: Pedopeisaje din teatre de război, Universitatea " Al. I. Cuza" din Iași, Facultatea de Geografie și Geologie; Academia Română, Filiala din Iași-Colectivul de Geografie, Iași, România, 21-23 septembrie 2018, poster, p. 16 din Program, p. 227 din Ghid.

Sorina-Simona Moraru, Antoaneta Ene, Alina Simionică, *Hydro-Physical Characteristics of Calcaro-Calcic Chernozems in the Malina River Basin, Galati County, Romania*, 18th International Balkan Workshop on Applied Physics and Materials Science, Section 3: Nuclear and sub-Nuclear Physics and Applications, Constanța, 10-13 iulie 2018, Universitatea "Ovidius" din Constanța, poster, p. 160 din Book of Abstracts.http://ibwap.ro/wp-content/uploads/2018/07/IBWAP-2018-BOOK-of-ABSTRACTS.pdf

Antoaneta Ene, Claudia Stihi, Ana Pantelică, Marina V. Frontasyeva, **Sorina-Simona Moraru**, *Nuclear and Related Techniques Used for the Investigation of Soil Pollution and Metal Transfer in Plants in Industrial Areas*, 18th International Balkan Workshop on Applied Physics and Materials Science, Section 3: Nuclear and sub-Nuclear Physics and Applications, Constanța, 10-13 iulie 2018, Universitatea "Ovidius" din Constanța, poster, p. 120 din Book of Abstracts.
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Sorina Simona Arbanaș (Moraru), Antoaneta Ene, *Investigation of Hydro-Physical Characteristics of Agricultural Soils in Galati Region, Romania*, UGAL International Conference Multidisciplinary HUB for the Higher Education Internationalization by Means of Innovative Interaction with the Labour Market and Society, Galați, Universitatea " Dunărea de Jos" din Galați, ACADEMIC project, CNFIS - FDI - 2018 - 0054, Session: Progress in Science, Engineering, and Management of Agriculture and Food Bio-resources, 26-27 octombrie 2018, Galați, România, poster.
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Moraru S.S., Ene A., Gosav S., Moraru D.I., Sloata F., *Relation Between Land Use, Industrial Activity and Metal Accumulation in Plants*, 32nd Task Force Meeting of the UNECE ICP Vegetation, Târgoviște, Session: Moss survey, 18-21 februarie 2019, Universitatea " Valahia" din Târgoviște, România, poster, p. 8 și 81 din Programme & Abstracts. https://icpvegetation.ceh.ac.uk/sites/default/files/Book_of_Abstract_TFM_2019_final.pdf

Ene A., Stihi C., Frontasyeva M., **Moraru S.S.**, Pantelica A., *Nuclear and Atomic Techniques Used for Heavy Metal Pollution Investigations in Agroecosystems*, 32nd Task Force Meeting of the UNECE ICP Vegetation, Târgoviște, Session: Moss survey, 18-21 februarie 2019, Universitatea "Valahia" din Târgoviște, România, poster, p. 8 și 75 din Programme & Abstracts.
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Sorina-Simona Arbanas (Moraru), Antoaneta Ene , Steluta Gosav, Dana Iulia Moraru, *Soil Properties Assessment in the Frame of Heavy Metals Contamination Due to Industrial Activities in Galati Area, Romania,* 7th Edition of Scientific Conference of Doctoral Schools SCDS-UDJG - Perspectives and challenges in doctoral research, Galați, Section 2: Advanced investigation methods in environment and biohealth, Universitatea "Dunărea de Jos" din Galați, Romania, 13-14 iunie 2019, prezentare orală OP.2.4, p. 12 din Program, p. 64 din Book of Abstracts.
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***Awarded presentation - First prize - Scientific Conference of the Doctoral School of "Dunărea de Jos" University of Galati, 7th edition,* "Dunărea de Jos" University of Galați, Romania, 13-14th of June 2019.**

Sorina-Simona Arbanas (Moraru), Antoaneta Ene, Dana Iulia Moraru, *Contamination Level of Triticum Vulgare L. Cultivated on Soils around a Metallurgical Area in Galati, Romania*, 7th Edition of Scientific Conference of Doctoral Schools SCDS-UDJG -Perspectives and challenges in doctoral research, Galați, Section 2: Advanced investigation methods in environment and biohealth, Universitatea "Dunărea de Jos" din Galați, Romania, 13-14 iunie 2019, poster PP.2.2, p. 31 din Program, p. 213 din Book of Abstracts. http://www.cssd-udjg.ugal.ro/index.php/2019/programme-2019 http://www.cssd-udjg.ugal.ro/index.php/2019/abstracts-2019

Antoaneta Ene, Ana Pantelică**, Sorina-Simona Arbanaș (Moraru)**, Violeta Pintilie, Florin Sloată, Florina Cristiana Căpriță, Mihai Straticiuc, Dragoș Mirea, Andreea Şerban, Claudia Stihi, Marina Frontasyeva, Oleg Bogdevich, Elena Culighin, *Development of analysis methodology using Proton Induced X-ray Emission (PIXE) as a complementary technique to determine trace elements in environmental matrices*, 7th Edition of Scientific Conference of Doctoral Schools SCDS-UDJG - Perspectives and challenges in doctoral research, Galați, Section 2: Advanced investigation methods in environment and biohealth, Universitatea "Dunărea de Jos" din Galați, Romania, 13-14 iunie 2019, poster PP.2.4, p. 31 din Program, p. 215 din Book of Abstracts.
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Antoaneta Ene, **Sorina-Simona Moraru**, Dana Iulia Moraru, *Assessment of bioaccumulation of heavy metals in sunflower cultivated in the agricultural area next to steel industry*, 19th International Multidisciplinary Scientific Conference on Earth & Planetary Science - SGEM Geoconference 2019, Albena, Bulgaria, Section: Soils, 28 iunie-07 iulie 2019, poster, p. 47 din Plenary Programme.

Sorina-Simona Moraru, Antoaneta Ene, Steluta Gosav, *Study of the correlativity between parameters and mineralogy of contaminated agricultural soils*, 19th International Multidisciplinary Scientific Conference on Earth & Planetary Science - SGEM Geoconference 2019, Albena, Bulgaria, Section: Soils, 28 iunie-07 iulie 2019, poster, p. 47 din Plenary Programme.

Sorina Simona Arbanaș (Moraru), Antoaneta Ene, Steluta Gosav, Dana Iulia Moraru, *Intensive Agricultural Practices and Industrial Activities Influence on Soil Fertility of Agroecosystems from Prut and Siret Lowlands, SE Romania*, MONITOX International Symposium Deltas and Wetlands, Tulcea, Joint Operational Programme Black Sea Basin 2014-2020, code BSB165, Section 3 - Environmental Technologies, Restoration and Management of Aquatic Ecosystems, 15-17 septembrie 2019, Tulcea, România, poster S3.16, p. 7 din Program, p. 80 din Book of Abstracts.
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Antoaneta Ene, Oleg Bogdevich, Elena Zubcov, Yuriy Denga, Thomas Spanos, Ana Pantelică, Marina Frontasyeva, Claudia Stihi, Liliana Teodorof, Adrian Burada, Cristina Despina, Dana Iulia Moraru, Elena Culighin, Alina Sion, Vasile Bașliu, Alina Cioromila, **Sorina Simona Moraru**, Florin Sloată, *Nuclear and Atomic Techniques Used for the Quantification and Mapping of Heavy Metals and Trace Elements in Soils*, MONITOX International Symposium Deltas and Wetlands, Tulcea, Joint Operational Programme Black Sea Basin 2014-2020, code BSB165, Section 1 - Monitoring of Toxicants in Rivers - Deltas - Seas Ecosystems in the Black Sea Basin, 15-17 septembrie 2019, Tulcea, România, poster S1.07, p. 4 din Program, p. 24 din Book of Abstracts.
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Sorina-Simona Arbanaș (Moraru), Antoaneta Ene, Steluta Gosav, *Mineralogic Composition Assessment of Soils from Covurlui and Braila Plains by ATR-FTIR Technique*, Simpozionul Internațional "Prioritățile chimiei pentru o dezvoltare durabilă", PRIOCHEM XV, Section 4: Students section, Institutul Național de Cercetare și Dezvoltare pentru Chimie și Petrochimie, București, 30 octombrie-1 noiembrie 2019, București, România, poster, p. 242 din Abstract Book. https://icechim.ro/wp-content/uploads/summary2019-proceedings.pdf

Sorina-Simona Arbanas (Moraru), Antoaneta Ene, *Nutrient stocks study in agroecosystems located near the steel industry, Galati, Romania,* 8th Edition of Scientific Conference of Doctoral Schools SCDS-UDJG-Perspectives and challenges in doctoral research, Galați, Section 2: Advanced investigation methods in environment and biohealth, Universitatea "Dunărea de Jos" din Galați, Romania, 18-19 iunie 2020, prezentare orală OP.2.8, p. 10 din Program, p. 73 din Book of Abstracts.
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***Awarded presentation - First prize - Scientific Conference of the Doctoral School of "Dunărea de Jos" University of Galati, 8th edition,* "Dunărea de Jos" University of Galați, Romania, 18-19th of June 2020.**

Antoaneta Ene**, Sorina-Simona Arbanas (Moraru)**, Steluta Gosav, Florin Sloata, Magdalena Aflori, Vasile Basliu, Alina Cantaragiu, *Assessment of mineralogical composition of cultivated soils impacted by iron and steel industry using combined advanced techniques,* 8th Edition of Scientific Conference of Doctoral Schools SCDS-UDJG -Perspectives and challenges in doctoral research, Galați, Section 2: Advanced investigation methods in environment and biohealth, Universitatea "Dunărea de Jos" din Galați, Romania, 18-19 iunie 2020, prezentare orală OP.2.9, p. 10 din Program, p. 74 din Book of Abstracts. http://www.cssd-udjg.ugal.ro/index.php/programme-2020
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Antoaneta Ene, **Sorina Simona Arbanas (Moraru)**, Florin Sloata, *Review of assessment methodology used for soil and sedements pollution by heavy metals based on single and multiple complex indexes*, International Conference „Environmental Toxicants in Freshwater and Marine Ecosystems in the Black Sea Basin", Kavala, Greece, Joint Operational Programme Black Sea Basin 2014-2020, code BSB165, Greece, 8-11 septembrie 2020, poster P.B.6, p. 5 din Conference program și p. 41 din Abstract Book.
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Antoaneta Ene, Ana Pantelica, Claudia Stihi, **Sorina Simona Moraru**, Alina Ceoromila, *Assessment of quality of Danube River sediments in Romania, ecological and health risk*, International Conference „Environmental Challenges in the Black Sea Basin: Impact on Human Health", Galati, Romania, Section 4: Health and environment education, innovative solutions to improve scientific information dissemination, 23-26 septembrie 2020, poster P.A.21, p. 4 din Conference program, p. 44 din Abstract Book.
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Sorina-Simona Moraru, Antoaneta Ene, *Nitrogen, phosphorus and potassium investigation in topsoil layers from temperate agroecosystems, SE Romania*, International Conference „Environmental Challenges in the Black Sea Basin: Impact on Human Health", Galati, Romania, Section 3: Environmental technologies, remediation and management of riverine, deltaic and coastal ecosystems, 23-26 septembrie 2020, poster P.A.17, p. 3 din Conference program, p. 38 din Abstract Book.
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Sorina-Simona Arbanaș (Moraru), Antoaneta Ene, *Agricultural potential of soils in Sendreni and Tulucesti areas, Galati county,* 9th Edition of Scientific Conference of Doctoral Schools SCDS-UDJG-Perspectives and challenges in doctoral research, Galați, Section 2: Advanced investigation methods in environment and biohealth, Universitatea "Dunărea de Jos" din Galați, Romania, 10-11 iunie 2021, poster PP.2.3, p. 32 din Program, p. 233 din Book of Abstracts.
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http://www.cssd-udjg.ugal.ro/index.php/abstracts-2022

PARTICIPATION IN NATIONAL AND INTERNATIONAL PROJECTS

1. **RESEARCH GRANT JINR-Romania no. 21/2018**, Development of laboratory infrastructure for applications of nuclear and magnetic techniques on characterization of agricultural soils and content of potentially toxic elements, Theme no. 03-4-1128-2017/2019, Order IUCN 321/21.05.2018, Director Romania: Ene Antoaneta (UDJ Galati); student doctorand, membru; 2018-2019.
2. **RESEARCH Project JINR-Romania no. 111/2018**, Assessment of industrial impact on agroecosystems and human health risk in Romania using nuclear and related analytical techniques, Theme no. 03-4-1128-2017/2019, Order IUCN 322/21.05.2018, Director Romania: Ene Antoaneta (UDJ Galati); student doctorand, membru; 2018-2019.
3. **RESEARCH Project JINR-Romania no. 112/2018**, Assessment of air and soil quality in Romania studied by NAA and related analytical techniques, Theme no. 03-4-1128-2017/2019, Order IUCN 322/21.05.2018, Project Director: Stihi Claudia (Valahia University of Targoviște), Ene Antoaneta (Project Responsable, UDJ Galati); student doctorand, membru; 2018-2019.
4. **Project JINR-Romania no. 63/2019**, Assessment of industrial impact on agroecosystems and human health risk in Romania using nuclear and related analytical techniques, JINR Theme no. 03-4-1128-2017/2019, Order JINR 397/27.05.2019, Leaders from Romania: Ene Antoaneta (Project Director, UDJ Galati), Project Responsable: Stihi Claudia (Valahia University of Targoviște); student doctorand, membru; 2018-2019.
5. **Project JINR-Romania nr. 64/2019**, Assessment of air and soil quality using biomonitoring, neutron activation analysis and related analytical techniques, JINR Theme no. 03-4-1128-2017/2019, Order JINR 397/27.05.2019, Leaders from Romania: Stihi Claudia (Project Director, Valahia University of Targoviste), Ene Antoaneta (Project Responsible partner P1, UDJ Galati); student doctorand, membru; 2018-2019.
6. **RESEARCH GRANT JINR-Romania no. 26/2019**, Development of laboratory infrastructure for applications of nuclear and related techniques on the characterization of agricultural soils andtransfer of potentially toxic elements in plants, Theme no. 03-4-1128-2017/2019, Director Romania: Ene Antoaneta (UDJ Galati); student doctorand, membru; 2018-2019.
7. **Project Joint Operational Programme Black Sea Basin 2014-2020**, code BSB27, Black Sea Basin interdisciplinary cooperation network for sustainable joint monitoring of environmental toxicants migration, improved evaluation of ecological state and human health impact of harmful substances, and public exposure prevention (MONITOX) (2018-2021), Grant contract 105070/14.09.2018, Project Manager (Leader Partner) Ene Antoaneta (UDJG); student doctorand, voluntar, 2018-2021.
8. **Project Joint Operational Programme Black Sea Basin 2014-2020**, code BSB165, Creating a system of innovative transboundary monitoring of the transformations of the Black Sea river ecosystems under the impact of hydropower development and climate change (HydroEcoNex), (2018-2021), Grant contract 105067/14.09.2018, Project coordinator (Partner PP3) Ene Antoaneta (Dunarea de Jos University of Galati); (implementation period: 21 September 2018-20 March 2021); student doctorand, voluntar, 2018-2021.
9. **Proiectul Excelență, performanță și competitivitate în activități CDI la Universitatea "Dunărea de Jos" din Galați, EXPERT (ID 345)**; Programul 1-Dezvoltarea sistemului național de cercetare-dezvoltare, Subprogram 1.2-Performanță instituțională-Proiecte de finanțare a excelenței în CDI; Cod proiect:14PFE/17.10.2018; Coordonator: Prof.dr. ing. Bahrim Gabriela-Elena; student doctorand, grup țintă, 2019-2020.
10. **Proiect: Burse pentru educația antreprenorială în rândul doctoranzilor și cercetătorilor postdoctorat (BeAntreprenor!)**, Cod MySMIS: 124539, Programul Operațional Capital Uman, Axa prioritară 6-Educație și competențe; Director UDJG: Moraru Luminița; student doctorand, grup țintă, septembrie 2019- ianuarie 2021.
11. **Project JINR-Romania no. 68/2020**, Air pollution assessment by neutron activation analysis and related atomic methods using biological indicators, cod temă 03-4-1128-2017/2022,

Order IUCN 269/20.05.2020, Director: Ene Antoaneta (UDJ Galati); student doctorand, membru; 2019-2020.

12. **Project JINR-Romania no. 71/2020**, Neutron activation analysis and related analytical techniques for assessment of sediment quality in the Danube River and its deltaic areas, cod temă 03-4-1128-2017/2022, Order IUCN 269/20.05.2020, Director: Ene Antoaneta (UDJ Galati); student doctorand, membru; 2019-2020.

13. **RESEARCH GRANT JINR-Romania no. 32/2020**, Development of laboratory infrastructure for applications of nuclear and related techniques for the assessment of soil and sediment quality (metals, radionuclides, microplastics) in Danube and Black Sea region, Theme no. 03-4-1128-2017/2022, Director Romania: Ene Antoaneta (UDJ Galati); student doctorand, membru; 2019-2020.

Axa prioritară 6 - Educație și competențe

Titlul proiectului: Burse pentru educația antreprenorială în rândul doctoranzilor și cercetătorilor postdoctorat (BeAntreprenor!)

Contract nr. 51680/09.07.2019 POCU/380/6/13 - Cod SMIS: 124539