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Determination of Metal Content in Soil and Wheat Plant by Inductively Coupled Plasma Mass Spectrometry

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Abstract

This study aims to investigate the level of soil pollution and the grade of accumulation of metals and heavy metals by wheat plants from the soil in different parts of the crop: root, stem, leaf, spike and grain. Sampling campaigns took place in February, April and July when wheat plants were at different growth stages. A number of eight soil samples and eight wheat plant samples were collected. The sampled wheat plant was taken at the same time and from the same place as the soil. Concentrations of Al (aluminium), Cr (chromium), Mn (manganese), Fe (iron), Ni (nickel), Co (cobalt), Cu (copper), Zn (zinc), Sr (strontium), Cd (cadmium) and Pb (lead) were determined by inductively coupled plasma mass spectrometry. Bioconcentration and translocation factors were calculated for the samples analysed.

Keywords

Soil, Wheat Plant, Heavy Metals, Translocation Factor

1. Introduction

Soil is the vehicle for plant growth and provides crops with the nutrients needed for vital activities [1].

However, intensive agriculture, unregulated industrial farming, irrational use of fertilizers and urbanization often result in increased plant nutrients, heavy metals and pollutants that cause environmental pollution which affects crop yield and quality [2]. Farmers routinely apply excess manure, synthetic fertilizers, and other agrochemicals to improve crop yields without considering the recommended dosages. This can lead to offsite transport and leaching of nutrients, heavy metals, and other pollutants, which can contaminate soils and surface, subsurface, and ground waters [3].

Heavy metals are a major environmental concern due to their toxicity and detrimental effects on living organisms and ecosystems [4]. Heavy metal contamination of soils is one of the main factors contributing to soil quality decline and loss of biodiversity, which is also associated with plant contamination, as metals accumulate in the surface layer of soils and then enter the trophic chain [5]. Metals contamination of soils is one of the main factors contributing to soil quality decline and loss of biodiversity as metals accumulate in the surface layer of soils and then enter the trophic chain [6]. It is also worth mentioning that metal contamination of soils is rarely evident in the short term, but is characterised by dangerous ecological effects delayed in time [7]. Studying the mobility process of metals in soil is one of the most important ways to identify the ability of these contaminants to move between the solid and liquid phases in soil and later into the trophic chain [7]. Studying the mobility process of metals in soil is an important way to identify the ability of these contaminants to move between the solid and liquid phases in soil and later into the trophic chain [7]. The mobility of metals depends on the chemical form in which they are present in soil, as well as on the physical and chemical properties of the soil (pH, redox condition, electric conductivity, content of the organic matter, granulometric composition) [6] [8]. The most mobile elements are those of anthropogenic origin occurring in ionic and carbonate form. In contrast, metals associated with silicates and primary minerals have significantly reduced mobility [7]. The analysis of the ecological risks associated with metal contamination of soils has received much attention in the literature to date [7] [9] [10]. Soil has a significant impact on human health and quality of life [11]. Soil pollution can adversely affect human health.

Plants have varying abilities to accumulate elements, and their characteristics depend on factors such as plant species, growth stage, and environmental conditions. When we use these types of plants, we will introduce a number of elements into the body that include both essential nutrients and potentially toxic metals [12]. Bioconcentration factor is an important parameter in phytoremediation determining information on the metal uptake, its mobilization into the plant tissues, and storage in the shoot parts [13].

Bioconcentration factor (BCF) was calculated using the ratio of metals content in roots ($mg \cdot kg^{-1}$ dry weight) to its content in the rhizosphere soil ($mg \cdot kg^{-1}$ dry weight) and provides information on the plant species' ability to uptake and concentrate heavy metals from the soil [14]. A value of BCF greater than 1 indicates accumulation in roots biomass, while a BCF lower than 1 indicates no accumulation. The translocation factor (TF) was calculated as the ratio of metal content in leaves ($mg \cdot kg^{-1}$ dry weight) to that in roots ($mg \cdot kg^{-1}$ dry weight). This factor indicates the plant's capacity to transport heavy metals from roots to leaves, stems or flowers [15]. A value of TF greater than 1 indicates that metals are translocated from the roots to the aerial part of the plant [16].

Cereal product composition has a key role in the human diet. Cereals, especially wheat, may have a high nutritional value for human life, accounting for roughly 1/4 of annual cereal production, wheat supplying 70% of the world's food. Wheat is a principle food of the human daily die [17]. Wheat is an important source of dietary protein and has the potential to be exploited to replace animal protein-based products [18].

Because wheat is a staple food of the human daily diet [17], this led to the determination of the metal content in wheat plant.

The locality of Dragomiresti has 68% of the surface is arable soil and it is very important to calculate the content of metals in the soil. The soil and the wheat plant of the study area were used to determine the content of metals in the soil and in different parts of the wheat. The purpose of this study was to: (1) determine the content of heavy metals in farmland soil and wheat; (2) assess the degree of heavy metal pollution in farmland soil; (3) explore the accumulation and migration of metals in wheat.

2. Materials and Methods

The samples analysed in the study come from Geangoeşti, Dragomireşti Jud. Dâmbovița.

Dragomireşti area is located in the south-west of Targoviste Municipality at a distance of 10 km from two old capitals of the Romanian Country, namely Targoviste and Câmpulung, and they are situated at an altitude of 300 meters above sea level.

The village is crossed from west to east by the Dâmbovița River, and the component villages are on either side of the river. On the right side as the river flows, we have the villages of Decideni, Râncaciov and Ungureni, and on the left Dragomireşti, Geangoeşti and Mogoșești. This locality is located on the border between hills and plains in this area, the relief is mixed, here we find both plain and hilly landforms, but in a smaller proportion.

In terms of its position to the major landforms, Dragomireşti village can be placed on the line separating the hills from the plains, namely Ploiești-Târgoviște-Pitești. The settlement is crossed from east to west by two hills running parallel to the Dâmbovița River flowing between them. At certain points, the hills also form the boundary of the locality, and their heights descend visibly in this geographical setting.

Sampling was done by actually walking the wheat plot in rows and taking soil and plant samples with a distance of 2 meters between each sample. Soil and plant samples were collected at the same time and from the same place. Sampling was carried out in February and April and wheat sampling in July.

The metal content of the samples was determined using inductively coupled plasma mass spectrometry (ICP-MS).

The soil sample (0.4 g) was placed in the digestion vessel, followed by 2.5 mL of 67% high purity nitric acid and 7.5 mL of 37% high purity hydrochloric acid. After shaking (20 min) and digestion (25 min and 1800˚C), the vessels were cooled to room temperature, and then each solution was transferred to volumetric flasks (50 mL).

The location on the map of the lot from which the samples were collected is shown in **Figure 1**.

Figure 1. Location of the lot in Dragomirește (Source: ancpi.geoportal.ro).

3. Results and Discussion

3.1. Determination of Metals in the Crop Soil

Six soil samples were analysed at the first collection and another six samples at the second collection. The values of the metal concentrations obtained are shown in **Tables 1-2**. The highest concentration was obtained for aluminium and the lowest was for cadmium

Table 1. Descriptive statistics of metals concentration in Geangoesti area (mg/kg). First sampling.

Metal[a)]	Min[b)]	Max[c)]	Mean[d)]	Median[e)]	Std. dev.[f)]	RSD[g)]
Al	15111.18	24130.06	19335.16	18800.42	2817.91	0.074 - 0.106
Cr	43.82	61.77	51.39	51.24	5.49	0.072 - 0.106
Mn	905.60	1154.12	1007.37	999.77	85.72	0.087 - 0.107
Fe	9078.60	11913.95	11012.87	11279.03	922.27	0.069 - 0.101
Ni	379.27	475.71	419.14	416.65	33.40	0.078 - 0.107
Co	15.34	18.96	17.18	16.98	1.51	0.072 - 0.105
Cu	34.96	47.97	41.47	41.92	5.06	0.072 - 0.106
Zn	112.34	158.35	135.75	139.00	17.19	0.086 - 0.108
Sr	32.10	57.94	44.25	43.83	8.42	0.083 - 0.104
Cd	0.86	1.09	1.01	1.03	0.08	0.068 - 0.078
Pb	23.61	32.93	29.20	29.81	3.38	0.066 - 0.100

where: [a)]metals: Al-aluminiu, Cr-crom, Mn-mangan, Fe-fier, Ni-nichel, Co-cobalt, Cu-cupru, Zn-zinc, Sr-strontiu, Cd-cadmiu, Pb-plumb; [b)]Min: the lowest value obtained; [c)]Max: The highest value obtained; [d)]Mean: is determined by summing a set of numbers and then dividing the total by the count of those numbers; [e)]Median: be defined as the middle number of a group of numbers; [f)]Std. dev: The function estimates the standard deviation based on a sample; [g)]RSD: The Relative Standard Deviation is a measure of the standard deviation of the sample from the sample mean for a given data set.

Analysing the values in **Table 1** and **Table 2**, it can be seen that at the second sampling, the concentration of metals decreases, except for the concentration of aluminium which increases at the second sampling. The second sampling was carried out after the agricultural plot had been fertilised.

Table 2. Descriptive statistics of metal concentration in the Geangoesti area (mg/kg). Second sampling.

Metal	Min	Max	Mean	Median	Std. dev.	RSD
Al	14842.63	27303.48	19203.03	18644.47	3906.04	0.086 - 0.142
Cr	36.76	66.93	49.94	50.25	9.32	0.090 - 0.142
Mn	719.48	1164.02	974.64	1018.77	156.96	0.090 - 0.144
Fe	7351.70	11383.48	9616.60	9906.96	1430.28	0.088 - 0.133
Ni	291.52	485.86	400.45	417.89	64.55	0.090 - 0.143
Co	11.85	19.55	16.47	17.38	2.75	0.088 - 0.140
Cu	25.09	50.16	39.02	41.85	8.39	0.089 - 0.140
Zn	85.65	145.92	120.47	126.24	19.82	0.089 - 0.146
Sr	28.63	75.81	42.46	42.24	15.12	0.090 - 0.140
Cd	0.65	1.07	0.86	0.89	0.13	0.070 - 0.098
Pb	16.54	32.10	24.67	25.99	5.03	0.088 - 0.135

To verify the level of heavy metals pollution of the soil, the values obtained by ICP-MS (Inductively coupled plasma mass spectrometry) were compared with the values presented in the ORDER no. 756 of 3 November 1997 [19] for the approval of the Regulation on the evaluation of environmental pollution. It was found (**Figure 2**) that the concentration of heavy metals is higher than the normal concentration presented in the order but the concentration of any metal did not exceed the alert threshold for sensitive uses.

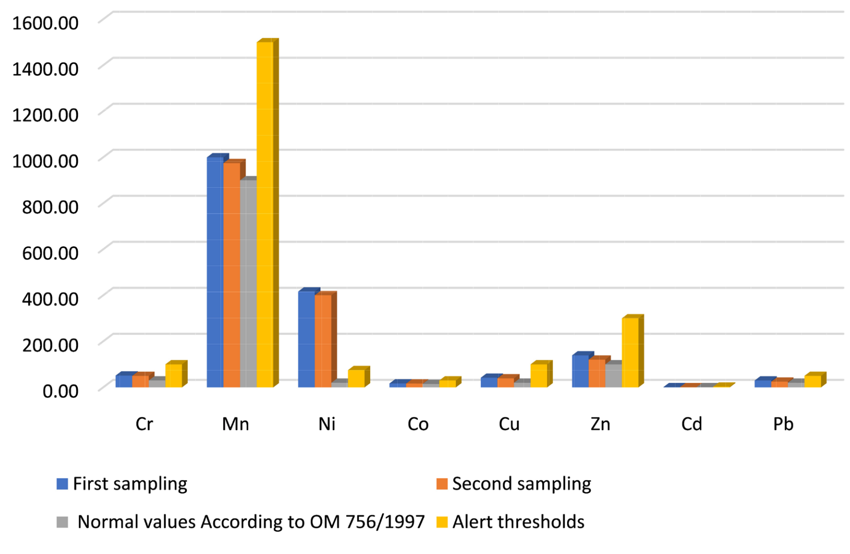


Figure 2. Heavy metal concentrations in soil (mg/kg).

The correlation matrix was performed and the values obtained are presented in Table 3.

Table 3. Matrix of correlation coefficients for metals.

	Al	Cr	Mn	Fe	Ni	Co	Cu	Zn	Sr	Cd	Pb
Al	1										
Cr	**0.9285**	1									
Mn	0.7633	**0.9404**	1								
Fe	0.1650	0.4374	0.6502	1							
Ni	0.7132	**0.9198**	**0.9751**	0.6682	1						
Co	0.5390	0.7934	**0.9063**	0.7341	**0.9587**	1					
Cu	0.2862	0.6124	0.7788	0.7988	0.8646	**0.9224**	1				
Zn	0.5423	0.7584	0.8963	0.7303	0.8932	**0.9409**	0.7835	1			
Sr	0.7507	0.8931	0.8557	0.4806	**0.9271**	0.8753	0.7571	0.7635	1		
Cd	0.4468	0.6939	0.8604	**0.9131**	0.8674	**0.9154**	0.8463	**0.9417**	0.7108	1	
Pb	0.2892	0.6010	0.7962	**0.9171**	0.8486	**0.9254**	**0.9586**	0.8630	0.6992	**0.9497**	1

There is a very strong correlation between Cr-Al, Mn-Cr, Ni-Cr, Ni-Mn, Co-Mn, Cd-Fe, Pb-Fe, Co-Ni, Sr-Ni, Cu-Co, Zn-Co, Cd-Co, Pb-Co, Pb-Cu, Cd-Zn, Pb-Cd a strong correlation between Sr-Cr, Zn-Mn, Sr-Mn, Cd-Mn, Cd-Ni, Pb-Ni, Sr-Co, Cd-Cu, Pb-Zn and a moderate correlation between Mn-Al, Sr-Al, Co-Cr, Zn-Cr, Cu-Mn, Pb-Mn, Co-Fe, Cu-Fe, Zn-Fe, Zn-Cu, Sr-Cu, Sr-Zn and Cd-Sr. The lowest correlation coefficient values are obtained for Fe-Al, Cu-Al and Pb-Al.

3.2. Bioconcentration Factor

To assess the ability of wheat to accumulate metals from the soil and to translocate them from the root to the aerial part of the plant, the following coefficients were calculated: bioconcentration factor (BCF) and translocation factor (TF).

If $BCF > 1$: plant accumulates metals.

If $BCF < 1$: the plant does not absorb metals from the soil.

$$BCF = \frac{CR}{GS} \tag{1}$$

where

BCF	bioconcentration factor
CR	root metal concentration
GS	soil metal concentration

In February, when the first samples were collected, the plant was 20 - 25 cm tall and had 3 - 4 leaves. As the plant was small, only the soil-plant bioconcentration factor was calculated.

Table 4. Soil-plant bioconcentration factor. First sampling.

Sample name	Metals										
	Al	Cr	Mn	Fe	Ni	Co	Cu	Zn	Sr	Cd	Pb
G1/S	0.154	0.230	0.406	0.119	0.139	0.499	0.682	0.152	1.243	1.043	0.564
G4/S	0.216	0.288	0.492	0.132	0.163	0.409	0.716	0.173	1.375	0.786	0.341
G6/S	0.370	0.448	0.605	0.240	0.245	0.665	0.862	0.229	1.547	1.068	0.556
G7/S	0.006	0.021	0.050	0.004	0.007	0.180	0.103	0.064	0.347	0.542	0.270
G10/S	0.250	0.300	0.444	0.132	0.165	0.543	0.659	0.177	1.896	0.985	0.631
G11/S	0.212	0.214	0.478	0.154	0.163	0.310	0.571	0.135	0.920	0.540	0.303
G13/S	0.079	0.119	0.341	0.060	0.088	0.151	0.539	0.179	1.287	0.590	0.205
G16/S	0.261	0.343	0.517	0.176	0.198	0.367	0.787	0.218	1.204	0.783	0.345

Analysing the values of metal concentrations in **Table 4**, it can be seen that sample G1/S (BCF = 1.043) and sample G6/S (BCF = 1.068) accumulate cadmium. Strontium is assimilated by samples G1/S, G4/S, G6/S, G10/S, G13/S end G16/S. For the other metals, the bioconcentration factor value is less than 1, which means that the plant does not accumulate heavy metals.

The second sampling was done in April, when the plant was developed and the spike was formed. The following were calculated for these samples: root-soil bioconcentration factor and translocation factor in the soil-plant system.

Soil-root bioconcentration factor was calculated at the second sampling, the results are presented in **Table 5**.

Table 5. Soil-root bioconcentration factor. Second sampling.

Sample name	Metals										
	Al	Cr	Mn	Fe	Ni	Co	Cu	Zn	Sr	Cd	Pb
R1/S1	1.926	1.753	2.782	1.347	1.451	2.600	2.230	0.601	3.106	2.959	2.745
R4/S4	1.143	0.973	1.605	0.710	0.751	1.686	1.832	0.511	2.254	3.036	1.773
R6/S6	1.781	1.415	2.009	1.074	1.085	2.099	1.904	0.465	2.428	2.323	2.102
G10/S10	1.493	1.242	1.761	0.924	0.921	1.650	1.327	0.408	1.963	2.002	1.615
G11/S11	1.568	1.361	2.300	1.121	1.215	1.938	1.680	0.511	2.448	2.414	1.971
G13/S13	1.143	1.760	2.338	1.083	1.260	2.055	1.462	0.511	2.842	2.233	1.873

The calculated bioconcentration factor for Al (aluminium), Cr (chromium), Mn (manganese), Fe (iron), Ni (nickel), Co (cobalt), Cu (copper), Sr (strontium), Cd (cadmium), Pb (lead), Zn (zinc) are greater than 1, which means that the plant accumulates the above-named metals.

- ✓ For Al the bioconcentration factor value is between 1.143 and 1.143. The plant accumulates Al
- ✓ For Cr the bioconcentration factor value is between 0.973 and 1.760. Sample G1.4 R/S1.4 does not accumulate Cr.
- ✓ For Mn the bioconcentration factor value is between 1.605 and 2.338. The plant accumulates Mn.

- ✓ For Fe the bioconcentration factor value is between 0.710 and 1.347. Sample G1.4 R/S1.4 (0.751) and G1.10/S1.10 (0.924) do not accumulate Fe.
- ✓ For Ni the bioconcentration factor is between 0.751 and 1.451. Sample G1.4 R/S1.4 (0.751) and G1.10/S1.10 (0.921) do not accumulate Ni.
- ✓ For Co the bioconcentration factor is between 1.650 and 2.600. The plant accumulates Co.
- ✓ For Cu the bioconcentration factor value is between 1.327 and 2.230. The plant accumulates Cu.
- ✓ For Sr the bioconcentration factor is between 1.963 and 3.106. The plant accumulates Sr.
- ✓ For Cd the bioconcentration factor value is between 2.002 and 3.036. The plant accumulates Cd.
- ✓ For Pb the bioconcentration factor value is between 1.615 and 2.745. The plant accumulates Pb.
- ✓ For Zn the bioconcentration factor value is between 0.408 and 0.601. The plant does not accumulate Zn.

The highest value of the bioconcentration factor was recorded for Sr (3.106) and the lowest value for Zn (0.408).

3.3. Translocation Factor (Tf) of Metals

Metals concentrations detected in the root and in different parts of the wheat were used to calculate the translocation factor, the translocation factor was calculated as the ratio of the total metal concentration in the stem, leaves, spike and the metal content in the roots.

$$\text{Tf r-t} = \frac{Ct}{Cr} \tag{2}$$

where

Tf	the root-stem translocation factor
Ct	the metal concentration in the stem
Cr	the metal concentration in the root

$$\text{Tf t-f} = \frac{Cf}{Ct} \tag{3}$$

where

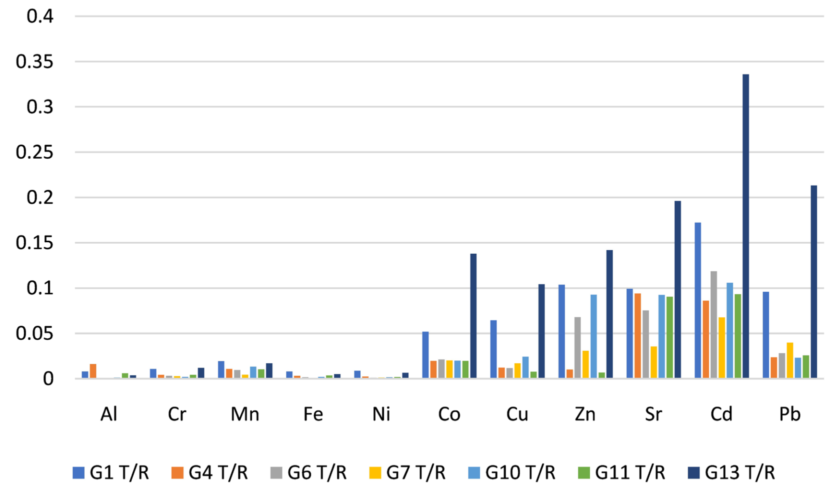
Tf t-f	the stem-to-root translocation factor
Cf	the metal concentration in leaves
Cr	the metal concentration in the stem

$$\text{Tf t-s} = \frac{Cs}{Ct} \tag{4}$$

where

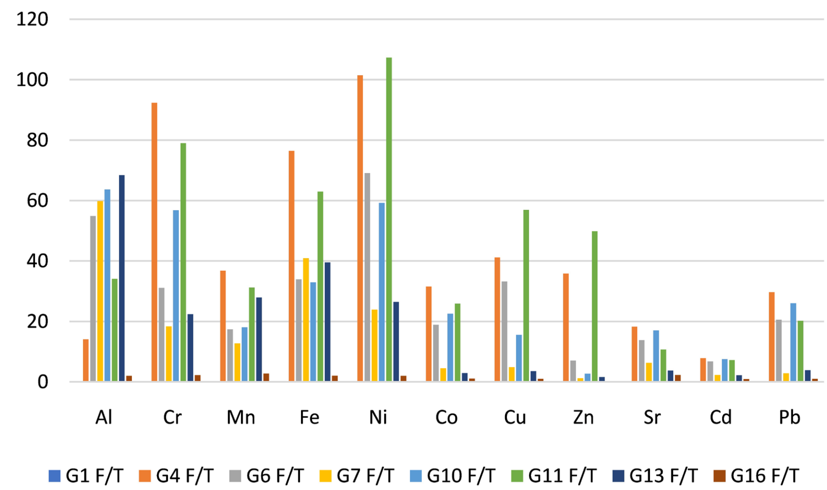
Tf t-s	the stem-spike translocation factor
Cs	the metal concentration in the spike
Ct	the metal concentration in the stem

From Figure 3 it can be noticed that the plant does not translocate metals to the stem, the TF value is less than 1. The highest TF values were recorded for Sr and Cd.



Where G1T/R; G4T/R; G6T/R; G7T/R; G10T/R; G11T/R; G13T/R is the name of the samples

Figure 3. Stem-to-root translocation factor.



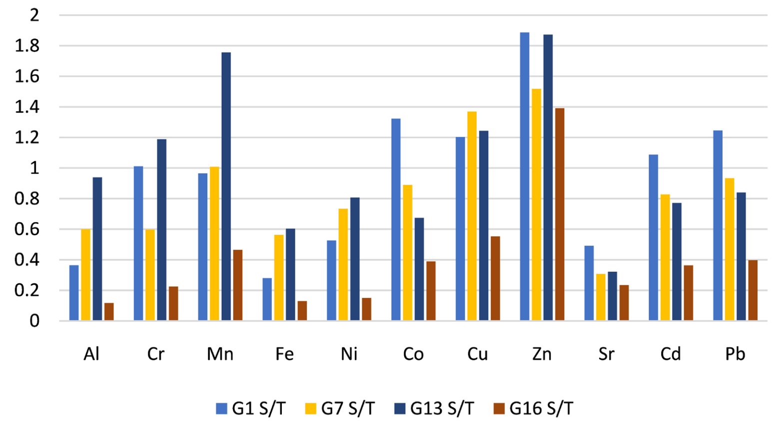
Where G1 F/T; G4 F/T; G6 F/T; G7 F/T; G10 F/T; G11 F/T; G13 F/T; G16 F/T is the name of the samples

Figure 4. Strain-flaxen translocation factor.

Figure 4 shows that the plant translocates aluminum, chromium, manganese, iron, nickel, cobalt, copper, zinc, strontium, cadmium and lead from stem to leaf. The calculated transfer factor values indicate easier translocation for aluminium, chromium, iron and nickel and heavier for cobalt, copper, zinc strontium cadmium and lead.

Figure 5 shows that the metals aluminum, iron, nickel and strontium are not translocated to the spike.

While chromium is translocated only by sample G13S/T; manganese is translocated by sample G13S/T; zinc is translocated by samples G1 S/T, G7 S/T, G13S/T; cadmium is translocated by sample G1 S/T and lead is translocated by sample G1 S/T.



Where G1 S/T; G7 S/T; G13 S/T; G16 S/T is the name of the samples.

Figure 5. Strain-spike translocation factor.

3.4. Evaluation of Metals in Wheat Grains

Figure 6 shows the concentrations of metals found in wheat grains. The order of the values of the metal concentrations in the grain wheat is as follows: Mn > Al > Pb > Fe > Zn > Cu > Sr > Ni > Co > Cr > Cd.

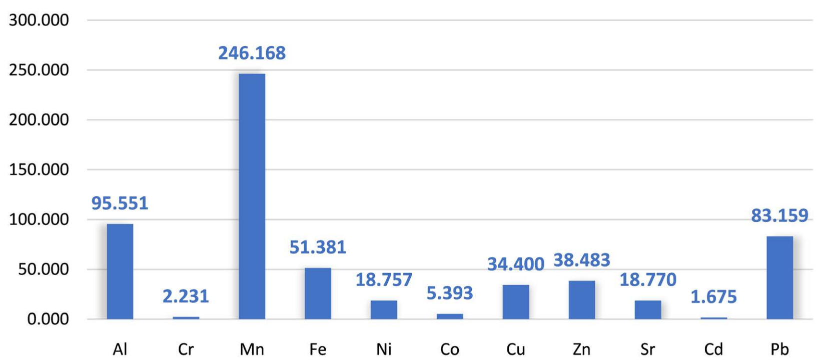


Figure 6. Metals in wheat grains (mg/kg).

Table 6. Arsenic and heavy metals in wheat grains.

mg/kg	Metals[a)]					
	Cu	Zn	Pb	As	Cd	Hg
sample of the wheat	34.400	38.483	83.159	not detected	not detected	not detected
Order No 975 Maximum limits for arsenic and heavy metals in wheat	5	15	1	0.2	0.2	0.05

[a)]Cu-copper, Zn-zinc, Pb-lead, As-arsen, Cd-cadmium, Hg-mercury.

Analysing the values in Table 6 it can be seen that the concentration of Cu, Zn and Pb is higher than the maximum limit presented in Order No 975. The metals As (arsenic), Cd (cadmium), Hg (mercury) were not detected in the analysed wheat sample.

4. Conclusion

Analysis of the 16 soil samples showed that the heavy metal concentrations did not exceed the alert threshold for the sensitive use type. The calculated transfer coefficients for the metals aluminium, chromium, manganese, iron, nickel, cobalt, copper, strontium, cadmium, lead, and zinc are greater than 1, which means that the plant accumulates these metals. The highest value of the bioconcentration factor was recorded for Sr (3.106) and the lowest value for Zn (0.408). Calculating the stem-root translocation factor, it was observed that its value is less than 1, which means that the plant does not translocate metals to the stem. The plant translocates aluminum, chromium, manganese, iron, nickel, cobalt, copper, zinc, strontium, cadmium and lead from stem to leaf. The calculated transfer factor values indicate easier translocation for aluminum, chromium, iron and nickel and harder for cobalt, copper, zinc strontium cadmium and lead. The metals aluminum, iron, nickel and strontium were found not to translocate to the spic. Eleven metals were detected in wheat grains. The order of metal concentrations is as follows: Mn > Al > Pb > Fe > Zn > Cu > Sr > Ni > Co > Cr > Cd. In the wheat flour obtained from the analyzed batch, it was found that the concentration of Cu, Zn and Pb exceeded the maximum permissible limit.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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