Researches on Heavy Metals Determination from Water and Soil in Galati County, Romania

CARMEN PENELOPI PAPADATU[1], MARIAN BORDEI[1], GHEORGHE ROMANESCU[2,3], ION SANDU[2,4*]

[1] Dunarea de Jos University of Galati, Faculty of Engineering, 111 Domneasca Str.,800008, Galati, Romania

[2] Alexandru Ioan Cuza University of Iasi, ARHEOINVEST Interdisciplynary Platform, 22 Carol I Blvd., Corp G, 700506, Iasi, Romania

[3] Alexandru Ioan Cuza University of Iasi, Faculty of Geography and Geology, 22 Carol I Blvd., Corp G, 700506, Iasi, Romania

[4] Romanian Inventors Forum, 3 Sf. Petru Movila St., Bl. L11, Sc. A., III/3, 700089, Iasi, Romania

This work presents a monitoring study accomplished on water and some soil samples collected from some points of Danube River and from other two locations situated in Galati County, from Romania. Water samples were collected from four points in Danube River, one point in Prut River (Oancea city) and from one point in Vadeni village (water from fountain). The heavy metals concentrations were determined using the Atomic Absorbtion Spectroscopy (AAS) Technique, together with some physical parameters. The soil samples were studied using two complementary techniques: X-Ray Fluorescence (XRF) and Scanning Electron Microscopy (SEM). It explores the possibility of the existence of a correlation between the concentration of heavy metals from surface waters and the concentration of heavy metals in soil in order to assess the contamination as a result of anthropogenic activities.

Keywords: heavy metal determination; water and soil monitoring; anthropogenic contamination; AAS, SEM-EDX and XRF techniques

The total complex of ecosystem is known as the biosphere, because it comprises the surface layer of the planet capable of supporting life. People must take in consideration the rules which govern the health and functioning of the biosphere because, if these rules are not respected, the life must disappear.

The plants and animals able to exist in any area will form a biotic community. Essentially, an ecosystem is a biotic community in interaction with its physical environment of sunlight, atmosphere, water and soil or rock. In this ecosystem for each species exists as a population, the growth or it decline which are affected by the capacity of the system to provide the requirements of life [1].

Water is an obvious limiting factor for plants and animals. The plants are classified in relation to their tolerance for dry, medium or wet conditions as: *xerophytes, mesophytes and hydrophytes*. Most land plants are in the intermediate, *mesophytic* range; desert plants are usually *xerophytes; hydrophytes grow in water or require an abundance of water in the soil.* All plants require water to support their active growth and metabolism [2].

Exposure to environmental toxicants can have profound effects on normal growth and development. Many environmental toxicants as the heavy metals from soil and water can alter the reproductive function and they can have effects on the central nervous system and behaviour. The link between these reproductive and neurologic phenomena has not been systematically investigated yet but we know that the heavy metals existents in water and soil have bad consequences on human body, plants and animals. The neuro-endocrine (hypothalamic-pituitary-gonadal) system, which integrates inputs and outputs from the nervous and reproductive systems, is functionally and anatomically situated to mediate the effects of environmental toxicants, particularly those that are endocrine-disrupting chemicals (EDCs), in case of developmental processes [1].

* email: ion.sandu@uaic.ro

Over 90% of Cd, Pb, Mn, Ni, Cu and Zn contents present in freshwater and sediments originates from human activities [3], associate with suspended particulate matter which settle and are accumulated in the bottom sediment. In literature was shown that heavy metals concentrations in aqueous phase decreased while they increased in the solid phase (as material in suspension and/or sediment) and in biota, conclusion which is useful in performing of assessment of water quality and the heavy metals pollution impact on coastal ecosystems [4-6].

In this paper it is presented a study accomplished on water and soil samples. This work analyzed the concentration of heavy metals from surface waters and concentration of heavy metals (Cu, Zn) in soil in order to assess the contamination as a result of anthropogenic activities. Main sources of pollution affecting the complex come from industrial area (mainly Metallurgical industry) of the Galati city, from the core Port and from the Danube (practically is the end area after 9 countries and many industrial and agricultural pollution sources) [3].The water samples were collected from three points in Danube River, *one point in Prut River (Oancea city) and from one point in Vadeni village (water from fountain).* The hydrographical basin of the Prut River is located in the north – east of the Danube basin with a catchment area of 27500 km^2 being developed on the territory of the following three countries: Ukraine, Romania and Moldavia.

In Romania, the area of the Prut River basin has 10967 km^2. There are many sources of water along the Prut River and Danube River [7-10]. In figure 1 was presented a map of the hydrographic basin of the Prut River and partially, we can observe the Danube River corresponding to Galati area [11].

Beside the water analysis, it was accomplished a chemical and structural analysis of soil. The soil samples were collected from different depths for analyses using X-Ray Fluorescence (XFR) and Scanning Electron Microscopy (SEM) Technique In table 1 were presented some

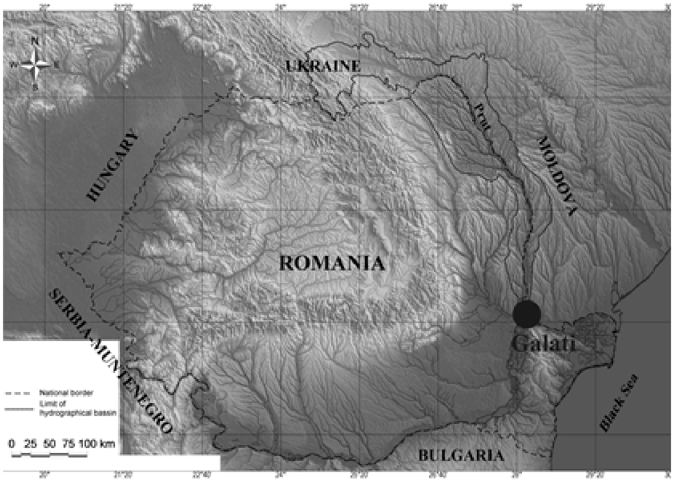

Fig. 1. The hydrographic basin of Prut River and Danube River

permissible values for heavy metals in water and indicators [12-15]. This study was realized during the following period: 04.01.2014 - 07.01.2014.

Experimental part

Materials and methods

For the water samples we considered for this study sampling sites (stations) such as: (*S1*) - Prut (Oancea 45°00' N and 28°07' E) and the measures were done in two days: in 04.18.2014 and in 05.21.2014; (*S2*) - the mouth spill of Siret River in Danube (the Bridge Galati-Braila) and the measures were done in two different days: 05.20.2014 and 06.30.2014; (*S3*) - Danube River (Braila Cliff) and the measures were done in the days: 05.20.2014 and 06.30.2014; (*S4*) - Danube River (Galati Cliff, near the ore carrier Port) and the measures were done in the days: 05.20.2014 and 06.30.2014; (*S5*) - Vadeni Village in Galati County (water fountain) and the measures were done in 04.20.2014 – before the floods.

Galati is the county seat. Oancea village is documented in 1521 during the rule of Stefan Voda. It is situated in the Eastern part of Romania, on the right bank of the Prut River, at 57 Km north of Galati town and 7 Km West of the town called Cahul in Moldavia Republic.

Methods used for water analysis

The monitoring process of water quality was made in accordance to the National Integrate System of Monitoring of Romanian Waters. The samples procedure is according to APHA. At each sampling time, the following chemical and physical parameters were analyzed: water debit, temperature, *p*H, Cu^{2+}, Zn^{2+} etc. The physical values were obtained on situ by means of a multiparameter type WTW Multi350i. The concentrations of the heavy metals (for example, Cu^{2+}, Zn^{2+}) were determined using the AAS (Atomic Absorption Spectroscopy) method. The analyses were carried out at the laboratory of the Materials and Environment Quality Centre from Dunarea de Jos University – Faculty of Engineering.

Methods used for soil analysis.

The soil samples were studied using two techniques: X-Ray Fluorescence (XRF) and Scanning Electron Microscopy (SEM). The collected soil samples were dried and all the roots and rocks were removed. For the SEM analysis the samples were sieved and pressed to a carbon tape.

Results and discussions

Experimental results have enabled a short monitoring. Seasonal averages of physical parameters for Prut River point (*S1*) ranged the records between 90.18 and 351.5 m^3/s for flow water; 5.55 and 21.18°C for temperature and 7.8-8.1 for *p*H value. The water debit in the studied zone for *S1* had an average value approximately 100 m^3/s. The debit was relatively constant. The seasonal variation of the temperature was within the normal range.

In figures 2 and 3 were presented some heavy Metals (Cu^{2+}, Zn^{2+}) concentrations from water, for zone *S1* (Prut River – Oancea zone). For this zone, in May, the concentration of Cu^{2+} was higher than the value from April. The value was 0.22 mg/L in April and it was 0.27 mg/L in May. The concentration of Zn^{2+} from water was 0.21 mg/L in April and it was 0.22 mg/L in May, after floods. In this case, in summer period was observed that Zn^{2+} concentration decreased.

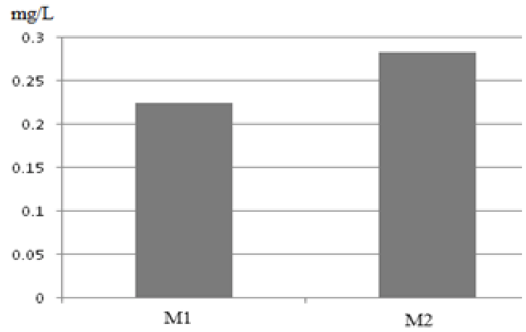

Fig. 2. Cu^{2+} mean concentration from the water in *S1* zone: M1 represents the measured value at 04.18.2014; M2 represents the measured value at 05.21.2014

Table 1
PERMISSIBLE VALUES FOR HEAVY METALS IN WATER AND INDICATORS

Indicators/heavy metals in water	Permissible values	Special permissible values (exceptional)	Analysis Methods Standards /STAS
Copper (Cu^{2+}), mg/L, max	0.005	0.1 (0.2)	[16-19]
Iron (Fe^{2+}+Fe^{3+}), mg/L	0.1	0.3	[20-21]
Phosphates (PO_4^{3-}), mg/L	0.1	0.5	[22]
Sulphates (SO_4^{2-}), mg/L	250	400	[23]
Zinc (Zn^{2+}), μg/L	5000	7000	[24]
Nitrates (NO_3^-), mg/L	45	-	[25]
Cadmium (Cd^{2+}), mg/L	0.005	-	[26, 27]
Mercury (Hg^{2+}), mg/L	0.001	-	[28]
Plumb (Pb^{2+}), mg/L	0.01	-	[29]
Chromium (Cr^{3+}), mg/L	0.1		[6, 30-33]

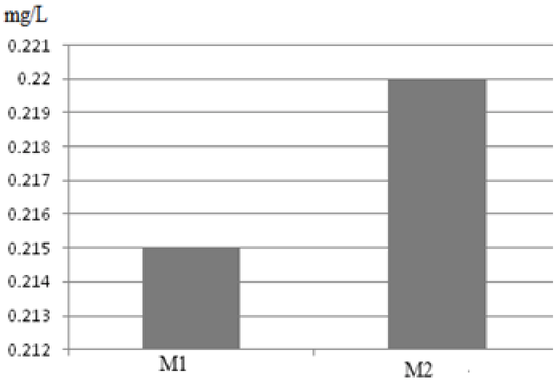

Fig. 3. Zn $^{2+}$ mean concentration from the water in *S1* zone: M1 represents the measuring values at 04.18.2014; M2 represents the measuring values at 05.21.2014, after floods

For this zone (*S1*), at analyzing the samples dated 04.18.2014, besides the heavy metals, were more determined the following optional: nitrites (0.0082 mg/L), nitrates (9.40 mg/L), phenols (3.60 mg/L), sulfates (211 mg/L). At analyzing the samples dated 05.21.2014, besides the heavy metals, were more determined the following optional: nitrites (0.124 mg/L), nitrates (9.51 mg/L), phenols (3.86 mg/L), sulfates (235 mg/L). The last results were obtained after spring floods.

Taking account the *S2* zone, it was made the *p*H determination dated 06.30.2014. The value measured in this case, was 7.17. For the sample dated 05.20.2014, the *p*H value was 8.33. The concentration of Cu^{2+} and Zn^{2+} from water, in *S2* zone, were presented in figures 4 and 5.

For this zone (*S2*), at analyzing the samples dated 05.20.2014, besides the heavy metals, were more determined the following optional: nitrites (0.17 mg/L), nitrates (13.1 mg/L). At analyzing the samples dated 06.30.2014, besides the heavy metals, were more determined the following optional: nitrites (0.16 mg/L), nitrates (13 mg/L). The concentrations correspond to normal limits. In this case, in summer period was observed a decreasing of heavy metals concentrations, most likely caused by the fixation of pollutants in sediment and vegetation, followed by a higher increasing tendency on Danube River, possibly caused by the input of industrial water. The results obtained for *S3* zone are presented in figure 6 and 7.

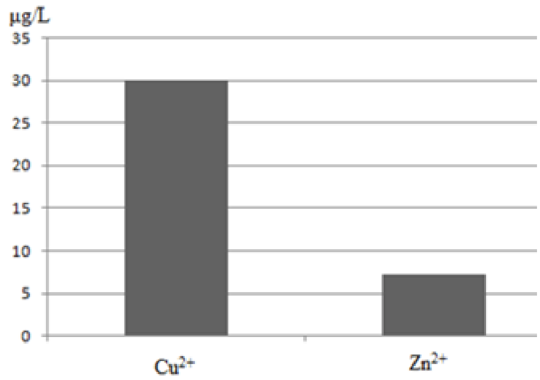

Fig. 4. Cu^{2+} and Zn^{2+} concentrations from the water in *S2* zone (samples dated 05.20.2014)

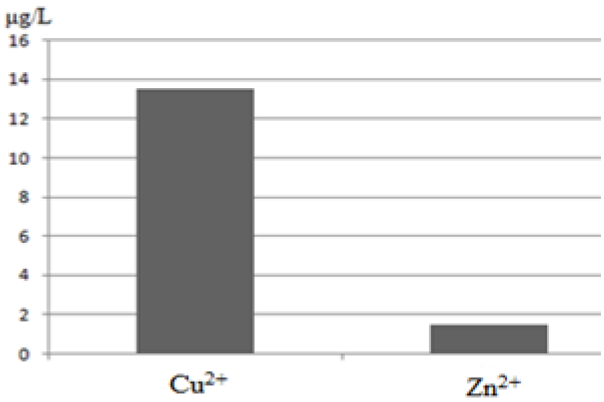

Fig. 5. Cu^{2+} and Zn^{2+} concentrations from the water in *S2* zone (samples dated 06.30.2014)

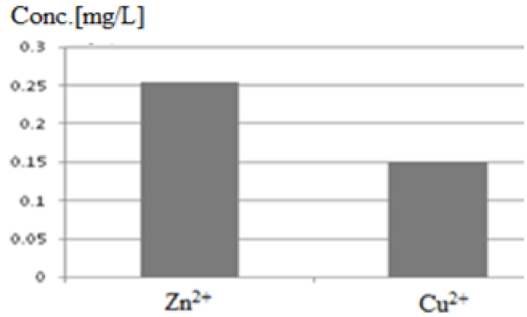

Fig. 6. Cu^{2+} and Zn^{2+} concentrations from the water in *S3* zone (samples dated 05.20.2014)

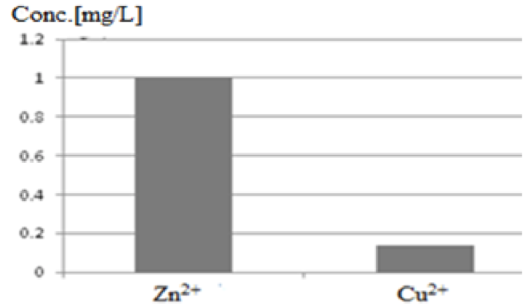

Fig. 7. Cu^{2+} and Zn^{2+} concentrations from the water in *S3* zone (samples dated 06.30.2014)

For this zone (*S3*), at analyzing the samples of water from the River (on the coast) dated 05.20.2014, besides the heavy metals, were more determined the following optional: nitrites (0.14 mg/L), nitrates (9.10 mg/L), phenols (3.7 mg/L). At analyzing the samples dated 06.30.2014, besides the heavy metals, was more determined the following optional: (0.15 mg/L) and nitrates (9 mg/L). The pH value was 8.03, in normal limits. Cooper registered variations between 0.1 mg/L and 0.15 mg/L. In this case, it is observed that the maximum experimental value is almost to alert threshold value. The results obtained for *S4* zone are presented in figures 8 and 9.

For *S4* zone, at analyzing the samples collected near the ore carrier Port dated 05.20.2014, besides the heavy metals, were more determined the following optional: nitrites (0.150 mg/L), nitrates (9.20 mg/L). The *p*H value was 6.87. For the samples dated 06.30.2014 the *p*H values was 8.07 (after floods). The samples were collected directly from the coast of the River, near the ore carrier Port, without

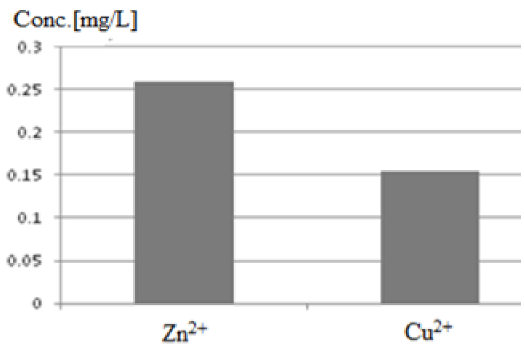

Fig. 8. Cu^{2+} and Zn^{2+} concentrations from water in *S4* zone (samples dated 05.20.2014)

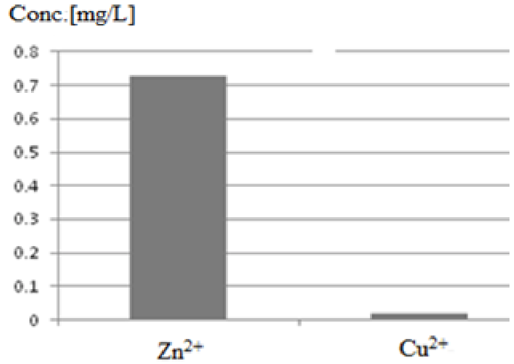

Fig. 9. Cu^{2+} and Zn^{2+} concentrations from water in *S4* zone (samples dated 06.30.2014, after floods)

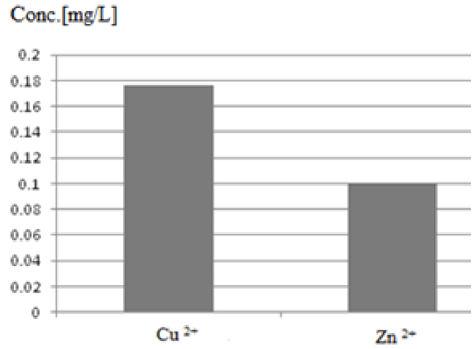

Fig.10. Heavy metals concentrations obtained from fountain (draw well) water sample (*S5* zone)

chemical processing. It can be observed that the concentration of Cu^{2+} from the water is higher, especially after floods. In figure 10 were presented the concentrations of Cu^{2+} and Zn^{2+} from fountain water (Vadeni Village) corresponding to the samples dated 04.18.2014. Copper registered variations between 0.005 mg/L and 0.150 mg/L. In this case, it is observed that the maximum experimental value is close to alert threshold value.

Besides the heavy metals, were more determined the following optional: nitrites (0.07 mg/L), nitrates (27.50 mg/L). High concentrations of copper were found in water because Cooper registered almost 0.18 mg/L. In this case, it is observed that the maximum experimental value corresponds to alert threshold (Quality Class IV-polluted). The concentration of Zinc from the fountain water was included in normal limits.

There are differences between the concentrations of heavy metals (Cu^{2+} and Zn^{2+}) from fountain water and the concentrations of these heavy metals from river (Groundwater v.s. surface water). These differences can be explained taking into account the human activities which determine the air pollution, for example. The quality classes of the drinking water [34-37] and the limit values for heavy metals were presented in table 2.

Copper is the most important mineral involved in the process of photosynthesis and nitrogen assimilation in plants affect, in the composition of protein complexes, such as hemocyanin, cytochrome oxidase and lactase tyrosinase [34, 35]. In literature, in unpolluted freshwater the normal copper content in solution is 0.5 to 2 mg/L and very polluted is 500-2000 mg/L [34-36]. For Copper in drinking water, we can accept the value of 2 mg/L, if the distribution pipes are from Copper. In table 3 were presented the admissible limits according to Ord.756/1997 [33] for less sensitive soil. The results obtained for the soil samples from Prut (Oancea) zone were presented in table 4. The samples were collected from different depths.

In figures 11, 12, 13 and 14 were presented the variation of the heavy metals concentration in soil, depending by the depth.

Chromium (Cr) element has all the concentrations over the legal normal threshold and under the legal alert threshold. The Cr sources are the pigment industry, stainless steel and other alloys [35]. This element in soil can be Cr(III) which is a stabile element and Cr(IV) which is very toxic for living organisms.

The normal concentration in soil for copper – Cu(II) - is 15 mg/kg. But Cooper registered variations between 19.24 mm/kg (1/5 sample) and 24.54 mg/kg (2/10 sample).In this case, it is observed that the maximum experimental value is over the legal normal thresholds but is under the legal alert threshold. Zn(II) registered variations between 51.08 mg/kg (3/15 sample) and 72.56 mg/kg (2/10

Table 2

THE QUALITY CLASSES FOR WATER ACCORDING TO [38]

Quality class Ord.MAPM 1.146/2002	pH	Fe^{2+} (mg/L)	Mn^{2+} (mg/L)	Pb^{2+} (µg/L)	Cr^{3+} (µg/L)	Ni^{2+} (µg/L)	Zn^{2+} (µg/L)	Cd^{2+} (µg/L)	Cu^{2+} (µg/L)
I	6.5 -8.5	fond	fond	fond	fond	fond	Fond	fond	Fond
II		0.1	0.05	5	50	50	100	1	20
III		0.3	0.1	10	100	100	200	2	40
IV		1.0	0.3	25	250	250	500	5	100
V		> 1.0	> 0.3	> 25	> 250	> 250	> 500	> 5	> 100

Table 3

ADMISSIBLE LIMITS ACCORDING TO [33] FOR LESS SENSITIVE SOIL

According to Ord.756/1997 [31]	Ni(II) [mg/kg]	Cr(III) [mg/kg]	Cu(II) [mg/kg]	Zn(II) [mg/kg]	Mn(II) [mg/kg]
Normal value	20	30	15	100	-
Alert threshold	200	300	100	300	-
Intervention level	500	600	250	700	-

Table 4

ELEMENTS CONCENTRATIONS FOR SOIL SAMPLES (EXPERIMENTAL)

Sample code/ depth[cm]	Ni(II) [mg/kg]	Cr(III) [mg/kg]	Cu(II) [mg/kg]	Zn(II) [mg/kg]	Mn(II) [mg/kg]
1/5	37.06	57.10	19.24	52.29	415.33
2/10	68.04	97.89	24.54	72.56	851.45
3/15	48.60	54.55	21.28	51.08	464.69

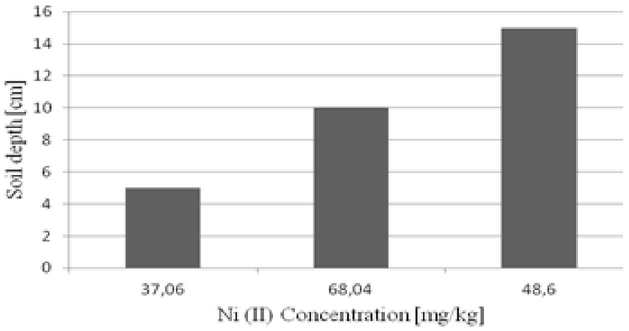

Fig. 11. Ni(II) concentration in soil, depending by depth

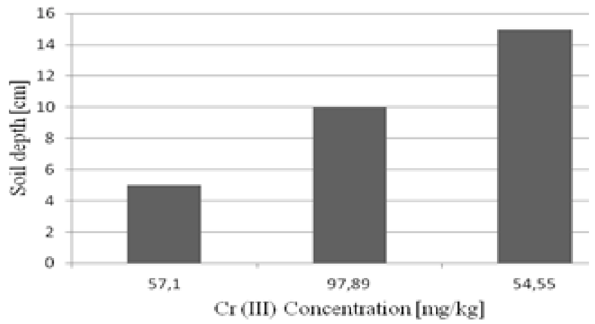

Fig. 12. Cr(III) concentration in soil, depending by depth

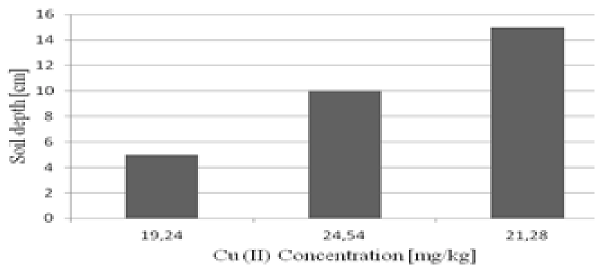

Fig. 13. Cu(II) concentration in soil, depending by depth

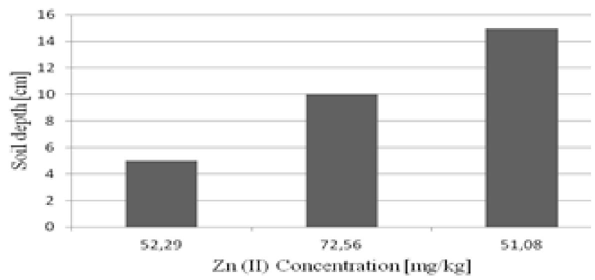

Fig. 14. Zn(II) concentration in soil, depending by depth

sample). In this case, it is observed that the measured values are smaller than the maximum permissible value of 100 mg/kg corresponding for legal normal level, as in literature [35, 39, 40]. In soil, Cu(II), Cr(III) and Ni(II) registered higher concentrations. In both cases (river water and soil), copper has concentrations over the legal normal thresholds. Compared with the concentrations from river water, appeared differences between the experimental values. These differences can be explained due to the existence of the anthropogenic activities such as: industrial water spilled partial treated, corrosive gas and acid rain, determined by human activities. In figure 15 was presented a SEM image where we can identify a soil structure based on clays and less quart.

There can be observed large particles (5-15μm) and discrete particles (0.5 – 2μm). These particles have different morphology, such as platy, nodular and spherical. The dimension of the aggregate is uniform and suggests a covering with a humic material because there are not evident pores.

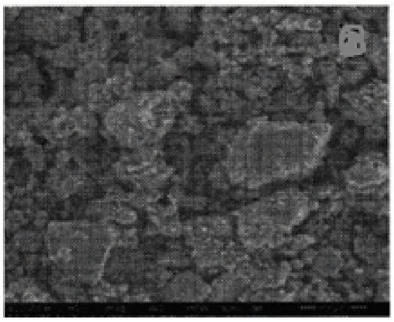

Fig. 15. SEM image soil (sample 1/5, 1000X)

Conclusions

There are differences between the concentration of heavy metals in the waters of the rivers, the concentration of the heavy metals from soil and from fountain water because the anthropogenic activity (acid rain, industrial overflows) existence. Taking into account the admissible limits for heavy metals in water, the analysed samples proved that the water is in weighted average polluted (III – rd class of pollution). During the considered period, the degree of pollution ranges from class II to class IV (polluted). The pollution intensity is maximum in the summer period (Class III-IV), in the case of Cu^{2+} in water. The quality of the water of Danube River situated at the confluence with Prut River corresponds to the Third (III) Class of pollution. This water quality corresponds to the weighted average polluted limits.

It is important to accomplish a study which reunites results for water and soil to understand the environmental pollution in the Galati County region. The heavy metals concentrations were found in normal values in spring period, before the floods, in soil and for Zinc (Zn^{2+}) in water. The constant monitoring of the Danube River, Prut River and other water sources is necessary because the water is an environment that sustains an ecosystem which depends on the quality of the water and soil. Composition of river water differs from the composition of water from the aquifer since intervenes the air pollution. The study revealed that the Cu^{2+} content in water and sediment samples from the two studied rivers (except Zn^{2+} in water samples) exceeded the maximum permissible concentration for quality class II (regulated by WFD and transposed into Romanian legislation by Order No. 161/ 2006), and it was especially high, this water being affected by anthropogenic load and the metallurgical industry. It is also important to take into account the important influence of the Danube River concentrations on heavy metals (loaded with pollutants from 9 countries situated upstream) and the necessity to have global measures in order to avoid at least the increase of the Danube pollution in the future.

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