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ASSESSMENT OF THE QUALITY OF THE DANUBE DELTA AQUATIC ECOSYSTEM – SURFACE WATER AND SEDIMENT

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Abstract. The main objective of this paper is to provide data on the Danube delta aquatic ecosystem quality (surface water and sediment). The samples were collected during the period May-October of the two consecutive years: 2005 and 2006 from Uzlina and Murighiol locations situated on Sf. Gheorghe branch. High concentration of PAH's compounds were recorded in Murighiol sediments and represents a significant pollution of the quality of sediment. The comparative analysis (physicochemical determinations) of the quality characteristics of the Danube delta aquatic ecosystem in these two years emphasised a dynamic character of the water quality that can determine an evolution into the unfavourable stages, which lead to worsen life conditions for aquatic organisms.

Keywords: the Danube delta, water, sediment, physicochemical characterisation, pollution, PAH.

AIMS AND BACKGROUND

The territory of the Danube Delta Biosphere Reservation has a total surface of almost 580 000 ha and includes the Danube delta, the complex Razim–Sinoe, the seaboard Danube till Cotul Pisicii and the lake Saraturi–Murighiol. From this surface, more than 50% (312 440 ha) are represented by aquatic and terrestrial natural ecosystems included in the universal patrimony.

The locations from where were taken the samples of surface water and sediment are situated on Sf. Gheorghe branch, one in the Murighiol and another in the

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Uzlina location. In Murighiol, the samples were collected from a canal, nearly the Murighiol lake. In this location, usually are presented a lot of vessel and here is a point where it makes filling with fuel. In many cases, on the surface of the Danube water, were presented oil spots. Also, in this location, wastewaters from houses situated in the neighbourhood are released in the Danube.

On the Uzlina location, the samples were collected from the touring complex Cormoran. In this place there are many naval transportation (speed boat, motorboat, skiff, fisher). The Cormoran complex presents a treatment station for domestic wastewaters. After treatment, the water is dump in the Danube river. The place from where the samples were collected is situated on an jetty.

The major sources of pollution in the Danube Delta Biosphere Reservation are represented by the economical agents from the nearby area, and also by the naval transportation. The release of heavy metals into the aquatic environment is known to cause detrimental effects to the environment. Most part of heavy metals become associated with particles and as a result of setting time, accumulate in bottom sediments of the receiving water. The fixation mechanisms of heavy metals represent important parameter for the risk assessment of the pollution level in the ecosystem. Mobility and availability are highly dependent on the way and the strength of the fixation of the heavy metals by the sediments components.

In this context, in a previous study carried out in the laboratory were established the extraction techniques for heavy metals from sediments[1]. The results of this study concluded that the optimum method applied to Cu, Mn, Zn, Fe, Pb, Cd, Cr, Ni and Hg extraction and determination is the French standard NF X31-120/1992 (Ref. 2).

The results of the study concerning the physicochemical quality state of the deltaic ecosystems from the Uzlina and Murighiol locations, carried out in the period April-October 2003 and 2004 (Ref. 3) showed an oxygen deficit and the presence of some chemical parameters (heavy metals, phenol compounds, mineral oil, *o*-phosphates, lindane) in concentration which exceeds the reference values[3].

The Norm No 161/2006 (Ref. 4) (transposed from Directive 2000/60/EC) introduced a classification of surface water quality with the purpose to establish ecological conditions of water bodies. The classification consists of five ecological conditions for surface water: very good (I), good (II), moderate (III), poor (IV) and bad (V). The quality parameters for chemical and physicochemical characterisation include: temperature condition, oxygen condition, nutrients, pollution with dangerous substances, mineralisation and pH condition. The Norm provides two categories of chemical conditions for all water, sediment and biota systems:

– good chemical conditions: imply that all quality parameters for the indicators are situated in the limits imposed by quality standards;

– bad chemical conditions: imply that parameters exceed the limits.

Taken into account that the dynamic character of the water quality can determine an evolution into the unfavourable stages which will lead to worsen life conditions for aquatic organism, the investigations were carried on in the next years 2005 and 2006 and the results of both investigation were compared. The results of the analyses provide relevant information on the ecosystem quality, with the focus on the physicochemical characteristics and an overview of the pollution level evolution in time.

EXPERIMENTAL

SAMPLING

Water and sediment samples were taken in each month from May to October (years 2005 and 2006) from the Murighiol and Uzlina locations (see the map of the Danube delta in Fig. 1).

Fig. 1. Map of the Danube delta: the Uzlina and Murighiol locations on Sf. Gheorghe branch

The sampling and the preservation of the samples were done in accordance with the recommendations of the specific international guides[5–7].

All water samples were collected and stored in glass bottles. At each sampling location, 10 l of water samples were collected. The sediment samples were taken from 2–3 m within the Danube river and for sampling was used a Van Veen Bottom sampler. All samples were kept in cooling boxes, at 4°C, during transportation, and the analyses were performed immediately after receiving the samples in the laboratory.

ANALYSIS

Detailed description of samples and analytical analysis performed is presented in Table 1.

Table 1. Summary of the samples collected from the Danube delta in the period May-October 2005 and 2006

Sample type	Location (L) and analysis (A) description
Surface water	L: Uzlina A: Group 1-8
Sediment	L: Uzlina A: Group 1, 2, 3 (only Lindane), 7 and 9
Surface water	L: Murighiol A: Group 1-8
Sediment	L: Murighiol A: Group 1, 2, 3 (only Lindane), 7 and 9

The following selected target compounds were analysed:

- Group 1: Polynuclear aromatic hydrocarbons – fluoranthene, benzo[*b*]fluoranthene, benzo[*k*]fluoranthene, benzo[*a*]pyrene, benz[*a*]anthracene, benzo[*g*,*h*,*i*]perylene, indeno[1,2,3-*cd*]pyrene, naphthalene, phenanthrene, anthracene, chrysene, pyrene (HLPC);
- Group 2: Heavy metals – arsenic, cadmium, copper, chromium, iron, lead, manganese, mercury, nickel, zink (atomic absorption spectrometry in flame and AAS with cold vapour technique for Hg);
- Group 3: Pesticide – organochlorine pesticides, triazine pesticides, organophosphorous pesticides (GC-ECD, GC-NPD)
- Group 4: Nutrients (water samples only) – ammonium, nitrate, nitrite, total nitrogen, *o*-phosphate, total phosphorus (spectrometry UV-vis.);
- Group 5: Salinity (water samples only) – conductivity, residue filterable, chloride, sulphate, calcium, magnesium, sodium;
- Group 6: Oxygen condition (water samples only) – COD, BOD, dissolved oxygen;
- Group 7: Other dangerous substances – mineral oil (IR spectroscopy), polychlorobiphenyls PCB (GC-ECD);
- Group 8: Other relevant parameters (water samples only): surfactants anionic, phenol index (spectrometry UV-vis);

• Group 9: Mobile forms of heavy metals (sediment samples only): cadmium, copper, chromium, iron, lead, manganese, mercury, nickel, zink (atomic absorption spectrometry in flame and AAS with cold vapour technique for Hg).

ANALYTICAL METHODOLOGY FOR ORGANIC POLLUTANTS

Water samples

For the extraction of PAH's compounds, 1 l of surface water was extracted with 50 ml of methyl chloride (Merck quality). The determination of all 12 compounds was performed with Agilent 1100, HPLC equipment and fluorescence detection.

Mineral oil was extracted from 1 l of surface water with 50 ml of carbon tetrachloride (Merck quality). The FT-IR System Perkin Elmer Spectrum BX II was used for quantitative determination of the total content of mineral oil.

Pesticides compounds: different solvents were used for extraction function of the class of pesticide: *n*-hexane for organochlorine pesticides, methyl chloride for organophosphorus and for chloroform triazine pesticides. The quantitative determination of pesticides was performed using the GC System Agilent Technologies type 6890 N, with: ECD detector (for organochlorine pesticides) and NPD (for triazine and organophosphorous pesticides).

Polychlorobiphenyls (PCB): 1l of surface water was extracted with 40 ml of *n*-hexane. The analytical procedure was performed with Network GC System Agilent Technologies type 6890 N, using ECD detector.

Sediment samples

For sediment samples determinations were performed with the same equipment used for water samples.

PAH's compounds were extracted from wet sediment with 100 ml of acetone and 50 ml of petroleum ether.

Mineral oil was extracted from dry sediment (before extraction procedure, the sediment was mixed with anhydrous sodium sulphates) with 50 ml of carbon tetrachloride.

Organochlorine pesticide (*Lindane*): the dry sediment (same procedure for drying like mineral oils) was extracted with 40 ml of *n*-hexane.

ANALYSIS OF HEAVY METALS

Water samples

250 ml of surface water was digested with 6 ml of hydrochoric acid (37%, Merck quality) and 2 ml of nitric acid (65%, Merck quality) on a hot electric plate. The quantitative determination of metals (cadmium, copper, iron, lead, manganese, nickel, zink) was performed with an Unicam Sollar type 929 atomic absorption

spectrometer with a mixture of air and acetylene for flame combustion. For chromium determination was used the flame mixture acetylene–nitrogen protoxide. Mercury determination was performed with could vapour technique after 24 h of oxidation of organic matter with potassium permanganate. Arsenic determination was done using a hydride generator coupled with AAS System.

Calcium, magnesium and sodium were analysed directly from the water samples, and for determination was used atomic absorption spectrometry with flame (air – acetylene for Mg and acetylene – nitrogen protoxide for Ca) and atomic emission spectrometry with air – acetylene flame for Na.

Sediment samples

Dry sediment (at room temperature) was digested with a mixture of aqua regia (21 ml of hydrochoric acid 37% and 7 ml of nitric acid 65%). The total concentration of metals (Cd, Cu, Cr, Fe, Pb, Mn, Ni and Zn) was determinated in liquid solution.

For mercury, the digestion procedure was performed in a Microwave Laboratory System Ethos Millestone on dry sediment mixed with 7 ml of nitric acid.

For arsenic determination it was necessary to add 10 ml of nitric acid and 15 ml of sulphuric acid (96%, Merck quality) on dry sediment.

ANALYSIS OF INORGANIC COMPOUNDS

For the quantification of inorganic parameters (ammonium, nitrate, nitrite, total nitrogen, *o*-phosphate, total phosphorus) was used a Perkin Elmer Lambda 25 UV-vis. spectrometer and specific equipment for pretreatment of the water samples.

Same equipment was used for determination of anionic surfactants and phenol index.

MOBILE FORM OF METALLIC ELEMENTS

The working conditions consisted in: 40 g of pretreated sediment were mixed with 40 ml of buffer solution of 1 M ammonium acetate and 0.01 M EDTA (pH = 7.2 ± 0.2). The obtained suspension was shaken 2 h with 40 rot./min. The resulted solution were filtered and after that analysed by AAS for metals determination.

RESULTS AND DISCUSSION

A large database was obtained after two years of physicochemical analyses of surface waters and sediments. The evolution of the concentration found in the water samples during the last two years and the comparison of these results with the reference values[4] imposed by the Norm 161/2006 are presented in Figs 2-7 for Murighiol and in Figs 8-11 for Uzlina.

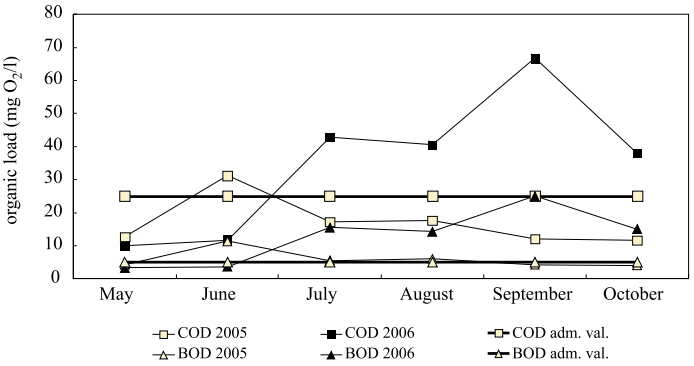

Fig. 2. Murighiol – organic load

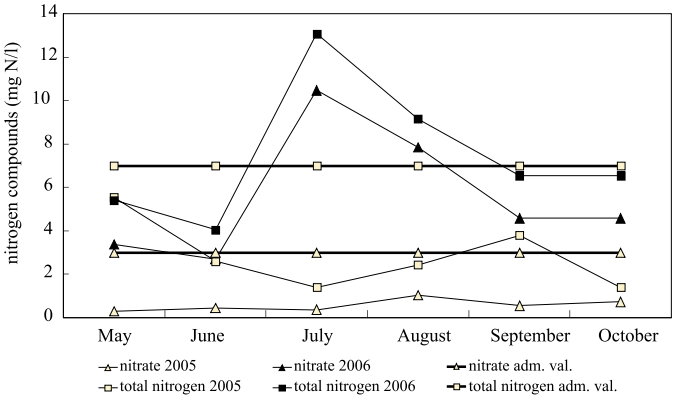

Fig. 3. Murighiol – nitrogen compounds

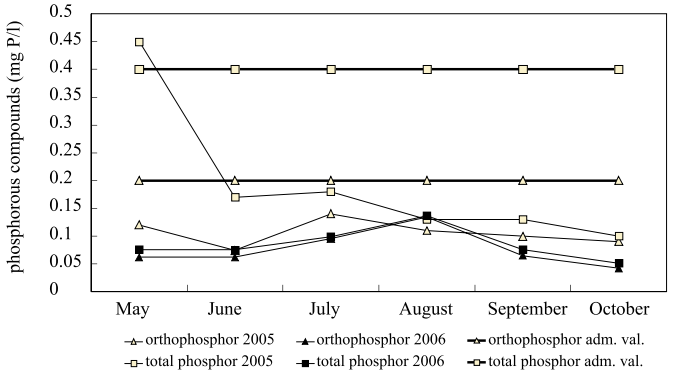

Fig. 4. Murighiol – phosphorous compounds

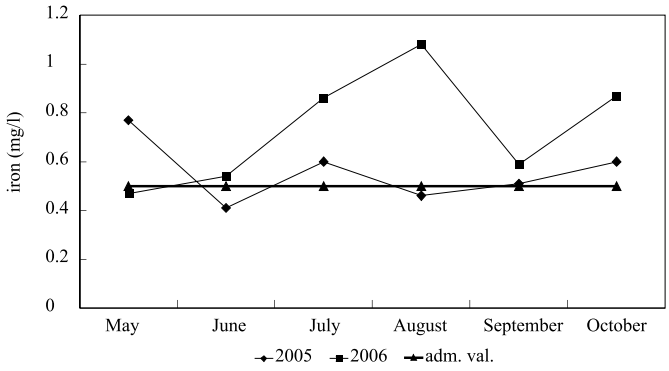

Fig. 5. Murighiol – iron

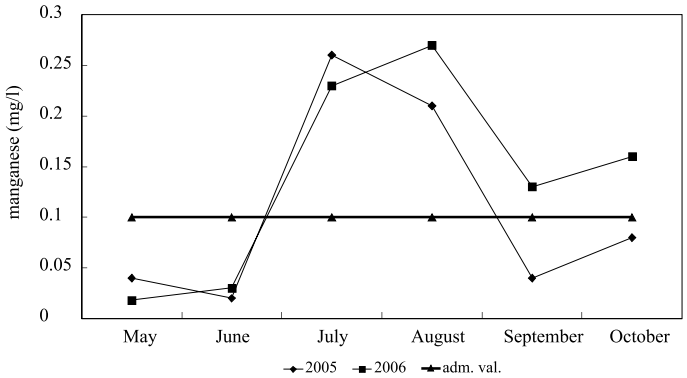

Fig. 6. Murighiol – manganese

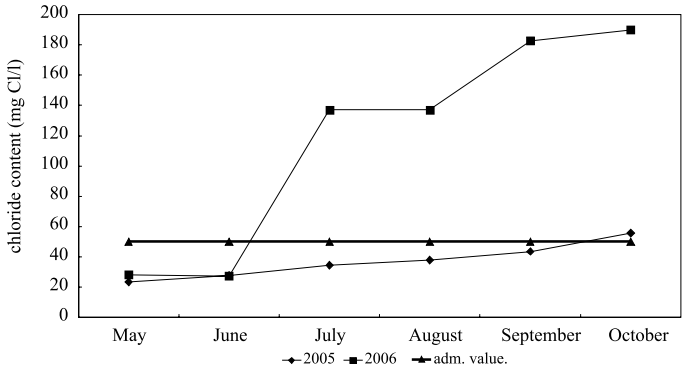

Fig. 7. Murighiol – chloride

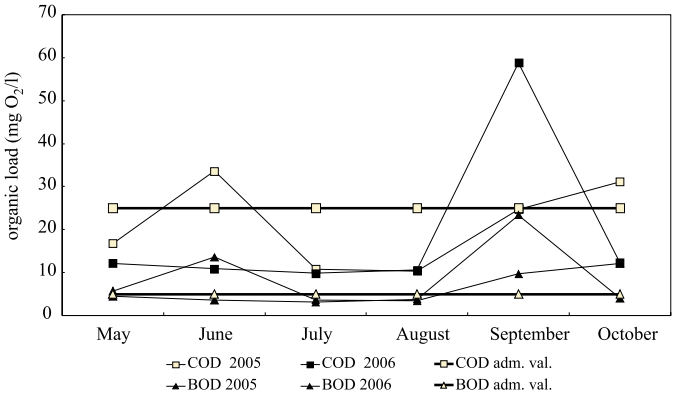

Fig. 8. Uzlina – organic load

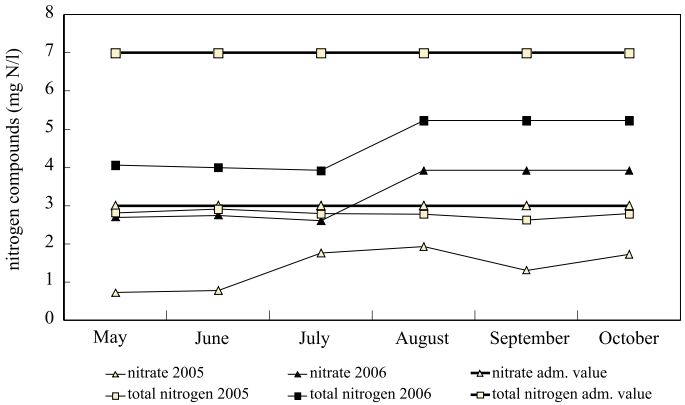

Fig. 9. Uzlina – nitrogen compounds

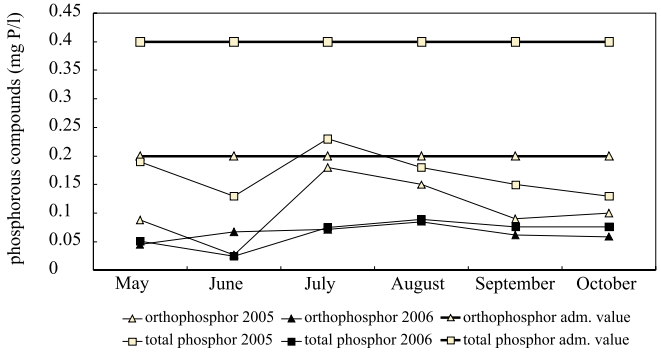

Fig. 10. Uzlina – phosphorous compounds

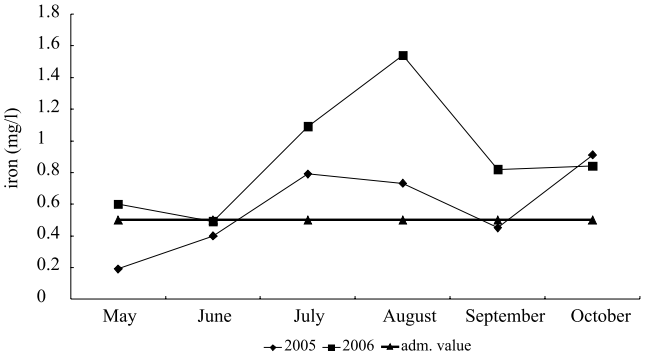

Fig. 11. Uzlina – iron

The values obtained were compared with second class quality for surface water. The following physicochemical changes took place in the water samples investigated in 2006, in comparison with 2005.

In Murighiol

• The water bodies presents a high organic load (based on COD and BOD concentration) from July till October; the values were higher than admissible limits (class II quality), thus in September was registered the highest value at BOD (27 mg O_2/l, class V) and COD (67 mg O_2/l, class III);

• Nitrogen compounds (represented by total nitrogen and nitrate ions) were also higher in 2006 than 2005, but only in July and August the values of total nitrogen were over admissible value; the nitrate concentration was higher than admissible value for class II of surface water (3 mg N/l) in almost all investigated months;

• The total concentration of phosphor was higher than the reference value only in May; all the other values, including *ortho*-phosphorous and phosphoric compounds are situated in both years under admissible values;

• The heavy metals concentrations were below the reference values, with exception of iron and manganese; for iron concentration were recorded in 2006 high values from July to October, higher than admissible value and higher than values detected in 2005; for manganese concentration, the situation was similar, the values from July to October being higher than the limit value (0.1 mg/l);

• An unexpected increase of the chloride ion concentration was observed in July and persisted at this high level also in the next months (140-200 mg/l); this increase is also reflected by the higher values of the conductivity and the filterable residue in the same period;

• The dissolved oxygen is lower than admissible value in all investigated period, the same situation is recorded in 2005; this fact causes detrimental effect on aquatic organisms, because the content of dissolved oxygen is an essential factor for alive organisms;

• Other pollutants detected in surface water in 2006 were PAH's compounds (benz(a)antracene and fenantrene); the concentration of these pollutants was higher than admissible values in 3 different months;

• Phenol compounds exceed the reference value during the whole period investigated in 2005 and 2006; this fact influences ecological state of water bodies.

In Uzlina

• Quality of the surface water from the Uzlina location is better than that of the water collected from Murighiol, due to the fact than Uzlina is situated on an open field and the flow of water is much higher. Here water has a good traffic and in an uninterrupted flowing, not blocked on the canal like those in the Murighiol location;

• For organic load (BOD and COD concentration), the highest concentrations recorded in both years were in September 2006 (23.4 mg O_2/l for BOD, class V and 58.8 mg O_2/l for COD, class III), the other values of 2006 being under the limits;

• Nitrate concentrations presented an increase from August to October and the values did not correspond to the class II according to the national norm; all total nitrogen values are under normal limit (7 mg N/l);

• The concentration of phosphorous compounds is lower in 2006 than in 2005 and is under the limits;

• Iron concentrations are higher than admissible value in 2006 in all investigated period and reflecting a pollution influence;

• Decrease of phenol compounds concentration was observed in 2006, but not within the tolerable limit for aquatic life ecological protection (101 μg/l – the highest value in 2005; 13 μg/l – the highest value in 2006).

The results of the sediments analyses (Tables 2 and 3) show a relatively uniform distribution during the investigated period in both locations.

In the Murighiol location, the sediment samples present:

• total heavy metals content (Zn, Cd, Cr, Ni, Pb, Hg and As) under reference values for the sediment quality according to Romanian Norm 161/2006;

• the major problem in Murighiol location appeared from the high concentration of total PAH content (between 4.5 and 13.5 mg/kg dry matter (dm), much more than 1 mg/kg dm, which represents the limit value); this problem may be older, because in the last legislation for sediment quality was proposed for measurement only benz[*a*]antracene, not all 11 compounds presented in new Norm 161/2006. For this reason, in the preceding investigations (2005), only benz[*a*]antracene was analysed;

• for copper were recorded higher values than the limit (40 mg/kg dm); in the Norm 161/2006, the limit value for Cu is 5 times lower than in the last legislation; in all investigated months, the copper concentration is in the range 29-67 mg/kg dm;

Table 2. Comparison analyses of Murighiol sediments (total concentration)

No	Parameter (mg/kg dm)	May		June		July		August		September		October		Order MMGA 161/2006
		2005	2006	2005	2006	2005	2006	2005	2006	2005	2006	2005	2006	
1	copper	42.7	55.4	67	45.3	43.1	52.3	41.6	34.4	40	28.6	46.7	41.6	40
2	zink	123	105	143	109	113	157	116	120	108	60.8	111	83.7	150
3	cadmium	< 0.5	<0.5	0.69	<0.5	1.3	<0.5	1.1	<0.5	1.5	<0.5	<0.5	<0.5	0.8
4	chromium	32.3	21.7	38.9	22.2	25.7	36.2	28.1	24.2	35.9	17.6	54.6	24.1	100
5	lead	30.3	25.7	26.2	30	30.6	27.9	36.2	16.7	29.4	19.4	35.3	21.2	85
6	mercury	0.13	<0.1	0.16	<0.1	<0.1	<0.1	<0.1	<0.1	0.17	<0.1	<0.1	<0.1	0.3
7	arsenium	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	29
8	nickel	–	33.4	–	31.5	–	41.4	–	27.6	–	19.0	–	29.1	35
9	Lindane	–	0.009	–	0.013	–	0.012	–	0.017	–	0.014	–	0.015	0.00005
10	PAH total	–	6.36	–	7.84	–	10.89	–	13.53	–	7.44	–	4.54	1
11	benz[*a*] antracene	0.05	0.38	0.03	0.44	< 0.02	0.16	< 0.02	0.43	0.056	0.41	< 0.02	0.2	–

Table 3. Comparison analyses of Uzlina sediments (total concentration)

No	Parameter (mg/kg dm)	May		June		July		August		September		October		Order MMGA
		2005	2006	2005	2006	2005	2006	2005	2006	2005	2006	2005	2006	
1	copper	81.2	58.8	69	55.9	80.5	53.8	76.8	55.6	63.2	55.4	46.7	56.5	40
2	zink	191	175	162	158	184	173	172	169	137	130	155	134	150
3	cadmium	1.05	<0.5	0.58	<0.5	1.5	<0.5	1.4	<0.5	0.67	<0.5	<0.5	<0.5	0.8
4	chromium	52.3	48.9	43.1	42.4	46.7	38.2	47.2	39	43.8	37.1	53.4	40.7	100
5	lead	35.1	46	28.9	28.9	36.7	18.8	33.3	21	34.8	30.7	27.7	29.2	85
6	mercury	0.15	<0.1	0.18	<0.1	<0.1	<0.1	<0.1	<0.1	0.18	<0.1	<0.1	<0.1	0.3
7	arsenium	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	29
8	nickel	–	56.9	–	56.3	–	46.1	–	50.4	–	49.3	–	51.1	35
9	Lindane	–	0.01	–	0.011	–	0.011	–	0.014	–	0.013	–	0.01	0.00005
10	PAH total	–	1.26	–	0.56	–	7.66	–	4.27	–	0.35	–	1.00	1
11	benz[*a*] antracene	0.039	<0.01	0.04	<0.01	< 0.02	0.05	< 0.02	0.03	<0.02	0.01	< 0.02	0.02	–

• Lindane concentrations are over the limit value (a very low concentration imposed by the Norm 161/2006, 0.00005 mg/kg dm) in 2006 and show a specific organic pollution.

In the Uzlina location, the quality of the sediments presents:

• concentration of copper between 45 and 80 mg/kg dm, values higher than admissible limit;

• structure of the sediment with high concentration of zinc (more than 150 mg/kg dm), with the risk for pollution with mobile forms of zinc;

• total concentrations of other metallic elements, such as Cr, Pb, Hg, As are under the limits value for the quality of sediment from surface water;

• the concentrations of cadmium in 2006 are lower and are within the normal level; in 2005, in 3 different months was recorded concentration of Cd compounds higher than limit value (0.8 mg/kg dm);

• geo-morphological structure of the sediment presents nickel compounds in concentrations situated between 46 to 67 mg/kg d.m. which are higher than the limit (35 mg/kg dm);

• for the PAH's compounds were recorded values over the proscribed limit in May, July and August, but the concentration are not so high than in Murighiol location;

• Lindane was present in all the investigated months in concentrations between 0.01 to 0.014 mg/kg dm, values higher than the limit (0.00005 mg/kg dm).

The study of availability of heavy metals (Cd, Cu, Cr, Zn, Ni, Pb, Mn, Fe and Hg) to migrate from sediments into aquatic bodies (Figs 12 and 13) emphasises the following aspects:

• the total concentration of chromium (Cr^{2+} + Cr^{3+}) in sediment (the Uzlina and Murighiol locations) within the scheduled investigation was between 18 and 49 mg/kg; the results of the investigations point out that this element is not in exchangeable forms, being bound by crystalline iron oxides (poorly and strongly), organic matter and in residual forms;

• the percentage of mobility for all elements was in general lower in 2006 (exception for Cu and Pb in the Uzlina location) and also the concentration of mobile forms of Zn and Mn in both locations;

• concentrations of mobile copper were higher in 2006 than in 2005 in both locations; same situation was detected for lead in the Uzlina location; this fact is due to increase of mobility percentage; the total concentrations of the metallic element for Cu were lower in 2006 than 2005, but the mobility percentage was higher; for Pb, the total concentration was situated in the same range or was lower, but the higher amounts of mobile Pb were detected because that element was easier available than in 2006;

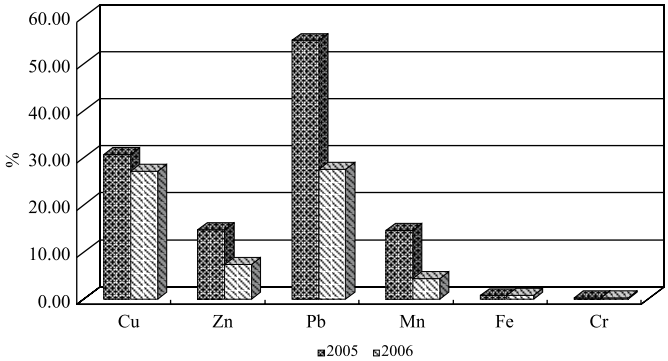

Fig. 12. Average of mobility percentage of heavy metals from the Murighiol location in the period My-October 2005 and 2006

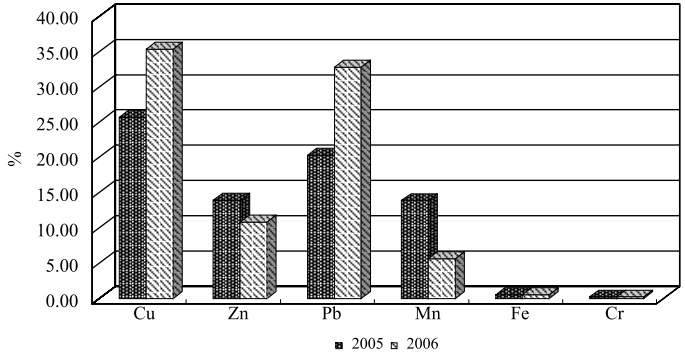

Fig. 13. Average of mobility percentage of heavy metals from the Uzlina location in the period May-October 2005 and 2006

• metallic species of cadmium and mercury were not found in mobile state inside the sediment bodies;

• even if nickel concentration was recorded over the limit value in sediments from Uzlina, this element presents low concentration of mobile Ni (1– mg/kg dm) and does not represent a pollution factor in water bodies;

• mobile forms of iron were situated in 2006 in the same range of concentration as in 2005 in both locations.

CONCLUSIONS

The measured values were momentary and showed the character of the contamination levels, relevant for the location and the investigation period.

The pollutants found in concentration over the reference limits put in evidence a potential pollution of water, alteration of water quality and diminutions of biological activity.

The water courses presented a deficit in dissolved oxygen and under saturation, not satisfying the prescribed regulation (7 mg/l O_2), concerning the conditions for protection of aquatic organisms.

High concentration of organic load (basis on COD and BOD concentration), phenol compounds, some PAH's compounds and metallic elements (iron and manganese) were detected in both locations, which do not correspond to favourable conditions for the water organisms development.

The major problems consist in detection of high concentration of PAH's total compounds (between 4.5 and 13.5 mg/kg dm in the Murighiol location and in the range 0.35–7.7 mg/kg dm in Uzlina) in sediments structure. These compounds are persistent substances and for this reason it is possible that pollution may be older than the investigated period from 2006.

The quality of waters and sediments collected from the Murighiol location is strongly affected by organic pollutants and correspond to bad chemical condition, because some important parameters (PAH compounds, dissolved oxygen, organic load) overtop the limits.

In the Uzlina location, the quality of water and sediment is better than in Murighiol and it is situated more close to the good chemical condition than to bad chemical condition. The concentrations recorded over limits are momentary and do not reflect the whole period of investigation. Even if the total concentration of heavy metals exceeded limit values (for Cu, Zn and Ni), the study of mobility shows that mobile elements can not affect alive organisms from water bodies. In this location, the auto-purification and biological activities of the river take place in good condition.

In the period April-October 2003 and 2004 were investigated water and sediment samples collected from the Uzlina and Murighiol locations.

In 2005 and 2006, the collection of samples from the two locations was not possible in April, because of the big floods, which conducted to a very high level of water flow.

The variation of parameters during all investigated period in the Uzlina and Murighiol locations was strongly influenced by the climate conditions (drought, flood).

In 2003 and 2004, in spring, when it was a dry weather, without rains, were recorded highest values of:

- organic load (BOD, 118 mg O_2/l; COD, 295 mg O_2/l) in Murighiol, June 2004;
- iron (8 mg/l) in Murighiol, June 2003;
- manganese (0.64 mg/l) in Murighiol, June 2003.

In spring-summer 2005 and 2006, a better quality of the surface water was recorded in principal, as a result of strong dilution of water. But in autumn it was

noticed a return to normal flow of water, which modified the quality of surface water in a negative way.

During all the period 2003-2006, the quality of sediment sustained slowly changes. The temporary high volume of water carried big quantity of alluviums and ensured the mixture of sediments, which explains the presence of Cd and Hg in the sediments structure in 2005 (low values, without problem for the environment).

These conclusions show a dynamic variation of physicochemical characteristics of water courses in investigated locations and it is necessary to continue the study in the next year.

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