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ANALYSIS AND ASSESSMENT OF HEAVY METALS CONCENTRATIONS IN NEMUNAS RIVER BOTTOM SEDIMENTS AT ALYTUS CITY TERRITORY

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Abstract. Concentrations and spatial distribution of heavy metals on the left and right banks of the river Nemunas bottom sediments are analyzed in this article. The research methodology of X-ray fluorescence spectrometry for bottom sediments and operating principles of XL2 spectrometer used for analysis are overviewed. The results of analysis are presented and compared with LAND 20-2005 requirements as well as studies that were carried out previously. The influence of Alytus city for Nemunas river sediments quality is assessed. Dischargers formed additional samples which were taken and included to the list of ordinary samples. The trend of heavy metals (Cd, Cu, Cr) concentrations showed the growth of pollution downstream the urban area. The estimated Zd (total pollution) values clearly indicated higher contamination by heavy metals on the left bank of Nemunas River. Extensive surveys of river sediments allow assessing the extent of anthropogenic impact, which can be harmful to the river ecosystem and human health.

Keywords: x-ray fluorescence spectrometry, heavy metals, sediments, water pollution.

Introduction

The present day river bottom sediments are complex mechanical, mineral and chemical composition heterogeneous polydispersed system which is sensitive to the change of surrounding environment physical and chemical conditions. Typically river sediments are formed from the mineral part of the river banks surrounding landscape, various chemical and mechanical maturity subsoil and rock soil constituents (Jankauskaitė *et al.* 2008). In addition, dead and unevenly decomposed biomass, manganese, iron and other elements hydroxides participates in sediments formation. In recent decades, the particularly important factors that determined sediments macro- and microelement composition has become of human productive and economic activity (Chalmers *et al.* 2006).

Heavy metals (HM) transport, access to river and accumulation in sediments are determined by natural and anthropogenic factors as a result of the self-purification of river water (Taraškevičius, Zinkutė 2011). HM which have low solubility and biodegradation characteristics are more accumulated in sediments. Usually in unaffected environments, the concentration of most of the metals in rivers is very low and is mostly derived from weathering of rock and soil (Reza, Singh 2010). In addition to natural factors concentrations of heavy metals in river sediments depend on the type of anthropogenic effect, intensity and duration. Urban environment is a complicated system, because 70–90% of all the soil lies under the streets, pedestrian paths, buildings and therefore, its natural environmental recovery takes a lot of time, a natural energy and material circulation are destroyed (Vasarevičius *et al.* 2010). The highest accumulation of metals occurs in low flow period and the lowest accumulation of metals occurs during the high flow period, suggesting that the concentration of heavy metals in bed sediments is brought about by changes of water flow (Bartoli *et al.* 2011; Li *et al.* 2012).

Contaminated sediments have negative impact to the bottom and bottom-dwelling organisms. Such environment leads to the loss or extinction of molluscs, fish and other hydrobionts (Farkas *et al.* 2007; Lourino-Cabana *et al.* 2011). In addition, the sediments that are heavily contaminated with toxic substances cause not only diseases or extinct of aquatic wildlife but are also dangerous to animals and humans that are consuming fish. Even if trace metals are initially emitted in their elemental forms, usually considered as non-bioavailable, their ultimate fate

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remains unknown. Therefore, urban runoffs not only degrade the environmental quality of aquatic ecosystems, but also lead to increasing contaminants in aquatic organisms through bioaccumulation and biomagnification, potentially causing elevated trace metal concentration in the food chain (Yujun *et al.* 2011; Sen, Varol 2011). Thus, the primary purposes of sediment quality guidelines are to protect the aquatic biota from the deleterious effects associated with sediment-bound contaminants, to rank and/or prioritize contaminated areas or chemicals of concern for further investigation (Honglei *et al.* 2007; Fu *et al.* 2014).

The analysis of HM concentrations and determination of allowable exceedance limits oblige to take measures to improve the quality of human life and the environment. Environmental pollution by HM is a global problem. This phenomenon is very important for river ecosystems which accumulate HM that get from natural and anthropogenic sources (Peng *et al.* 2008). It has long been accepted that HM are very important at ecotoxicological point of view as these elements are very persistent and can be toxic. In order to assess the ecological status of surface sediment s and their effects on the biota and human health it is important to measure the concentrations of HM (Davutluoglu *et al.* 2011).

Mainly due to the lack of methodology and equipment analyses of heavy metals basically have not been carried out until 1990 in Lithuania. First environmental monitoring system was developed in 1991–1992 in Lithuania. Later in 1997 first Lithuanian Environmental Monitoring Program which provided research of HM (Zn, Cu, Cr, Pb, Ni, Hg, Cd) in surface water and sediments has been prepared and approved. Despite the investigations carried out there is still not enough information about the distribution of HM in Lithuanian river sediments (Lithuanian Environmental Protection Agency 2003).

Fig. 1. Soil contamination with heavy metals in Alytus town according to the index Zd (Gregorauskienė 2006)

The objective of this work was to investigate and assess the contamination by heavy metals of Nemunas river bottom sediments at Alytus city territory using X-ray fluorescence spectrometry. This territory was chosen due to the lack of this type of research in this area. Also industries are well-developed in this city thus it can lead to an increased concentrations of heavy metals. Such detailed research which is important in determining the pollution sources shows the real distribution of heavy metals concentrations.

Results of researches that were carried out in Alytus city territory clearly shows industry and arbitrary household waste areas that are contaminated with zinc, copper, silver and other elements. Anomalies of heavy metals concentrations were determined near "Snaige" refrigerator factory, textile factory, railway, agricultural machinery factory, garages, lake "Dailide" and other territories (Fig. 1). All industrial and other polluting objects are located in the western part (left bank of the river Nemunas) of the city (Gregorauskienė 2006).

1. Methodology

1.1. General description of the study area

Sampling locations are set out on the left and right banks of the Nemunas River. 4.5 km sampling length was selected and it is actually bounded by two bridges in the area. Sampling started above Alytus city from the ruins of the railway bridge (destroyed during the World War I). The end of sampling was below Alytus city near the new bridge of the city ("Lithuania Millennium Bridge").

4.5 km sampling length was divided into equal parts at 300 m (Fig. 2, grey round points). Coordinates of sampling points were set with Garmin GPS Navigator "Nuvi 40". Dischargers and tributaries formed additional samples which were taken and included to the list of ordinary samples (Fig. 2, white square points).

1.2. Sampling and preparation

Samples were taken from the surface (0–0.1 m) sediment layer 1 m away from the shore. Samples were taken using stainless steel unpainted 7 liter bucket. Sample collection was held in summer which is low runoff period (summer period: 15–18% of total runoff). Samples were taken 1 m away from the shore thus granulometric composition of samples were sand and sandy loam (85–95% of physical sand). Samples were placed in separate polyethylene containers to avoid the cross-contamination. Containers were registered indicating the river side and the sample sequence number. Sediment samples were taken to the laboratory and placed in petri dishes. Samples were dried to constant weight in drying oven at (105±10) °C temperature. Dried samples were grinded in porcelain mortar until homogenous mass. The obtained mass of the sample was filtered through 2.00 mm, 250 μm and 125 μm mesh filter. Filtrated mass were placed in special caps which were inserted into spectrometer (Shu-hai *et al.* 2006; Suthar *et al.* 2009). Samples were tested in Thermo Scientific Niton® XL2 series X-ray fluorescence spectrometer.

Measurement time varies from 30 to 600 seconds depending on the intended quality of the results. The optimal 240-second analysis time was chosen to analyze bottom sediments of the Nemunas River (US EPA 2006).

28 elements were analyzed in bottom sediments (As, Hg, Cd, Ba, Sb, Sn, Ag, Pd, Zr, Sr, Rb, Pb, Se, Au, Zn, W, Cu, Ni, Co, Fe, Mn, Cr, V, Ti, Sc, Ca, K, S). Further analysis involved those metals that are anthropogenic and potentially dangerous.

1.3. Research device

Research was carried out using X-ray fluorescence spectrometer – Niton XL2. Spectroscopy is an analytical technique in which dry river bottom sediment sample is exposed to X-rays. The X-rays from the source have the appropriate excitation energy that causes elements in the sample to emit characteristic X-rays. A qualitative elemental analysis is possible from the characteristic energy, or wavelength, of the fluorescent X-rays emitted (Fig. 3).

A quantitative elemental analysis is possible by counting the number (intensity) of X-rays at a given wavelength (Wrobel, Czyzycki 2013). The energy of this X-ray radiation is unique for each element (US EPA 2006).

Each sample is analysed with 3 spectra using 4 different energy lines. Device automatically counts each spectra and each energy line values giving counted errors, element concentration values and detection limit. Detection limit with 240 seconds scanning time gives 2–4 ppm detection limit depending on each element.

1.4. The reliability of the X-ray fluorescence (XRF) spectrometry results

The reliability of XRF analysis method depends on sample preparation (drying, grinding, sieving). Unreliable results are recorded when there are no compliance with recommendations for sampling and preparation (Guoren *et al.* 2013). The most important requirements for the preparation of samples are grinding and sieving. The particle size of 250 μm (preferably 125 μm) must be achieved. Continuous dry matter should be 3 to 5 grams in the sample cup.

An experiment was carried out analyzing lead levels in Massachusetts (USA). Fully prepared XRF samples showed excellent correlation with laboratory Atomic Absorption Spectrometry (AAS) for material split after the final grinding, sieving, and homogenization (Fig. 4). A set of 20 fully prepared XRF samples (oven dried, ground to 0.125 mm), including 11 bridge site samples, 6 residential lead samples, and 3 NIST SRM soils, gave a linear regression slope of 1.004 and an R^2 of 0.995. For the 17 samples with lead concentrations above 100 ppm, the mean recovery of the XRF relative to AAS was 0.952 and the standard deviation of the recovery was 0.068, for relative standard deviation (RSD) of 7.1 percent.

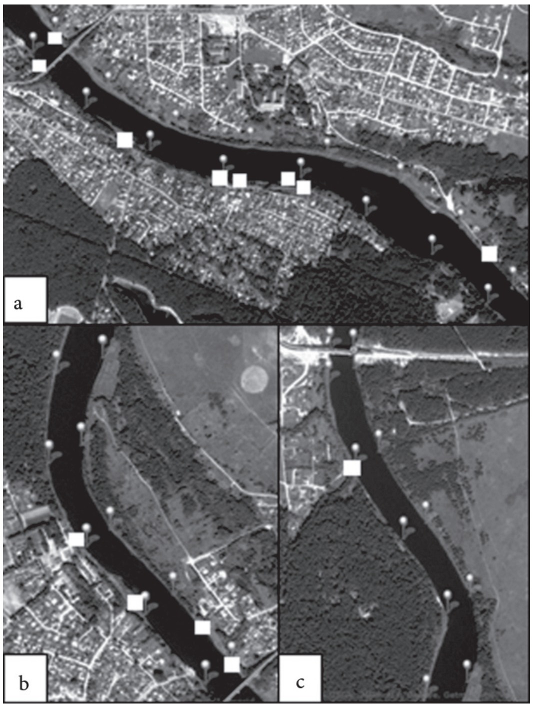

Fig. 2. Sampling locations downstream – Nemunas river above (a), middle (b) and below (c) Alytus city

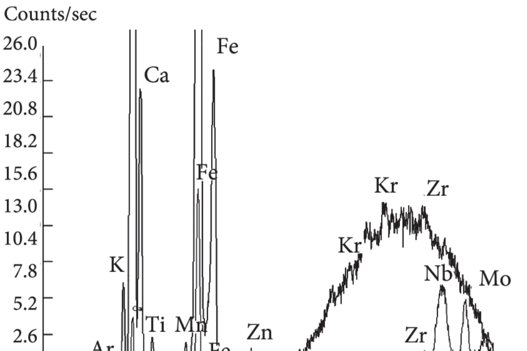

Fig. 3. Niton XL2 window showing wavelength and intensity measurement

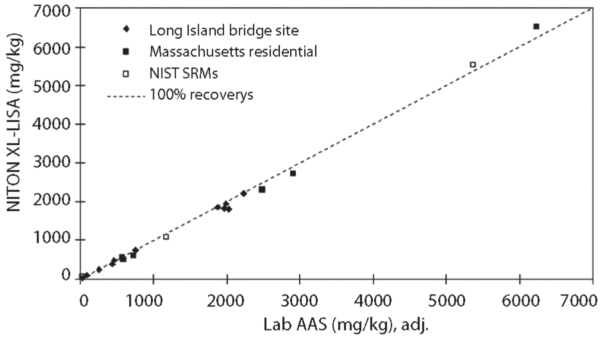

Fig. 4. Comparison of fully prepared XRF and laboratory AAS results

The subset of 11 bridge site samples gave a linear regression slope of 0.958 and correlation coefficient (R^2) of 0.994. The subset of 6 Massachusetts residential samples gave a linear regression slope of 1.010 and an R^2 of 0.994 (Shefsky 1997).

The XRF is much cheaper than the AAS analysis method thus it provides an opportunity to analyze a greater number of samples and obtain a more detailed distribution of HM concentrations in the territory.

1.5. Total contamination index (Zd)

Zd of HM (Cd, Ni, Cr, Cu, Zn, As, Mn) in bottom sediments was calculated for the left and right bank of the Nemunas River. These HM were chosen due to their potential anthropogenic origin.

Geochemical background values for these elements were taken from Lithuanian Hygiene Standard 60: 2004. There are values given for loam and clay that were chosen as a background values. This decision was made after the comparison of these concentrations with concentrations given in comparative characteristics of change of trace elements background values in Lithuanian surface sediments (Kadūnas, Radzevičius 2003). These values were closest.

Total contamination index is calculated by the formula:

$$Z_d = \sum K_K - (n-1). \tag{1}$$

Values of coefficient of element concentration (K_K) are summed up only if K_K values are greater than 1.

n – number of chemical elements (pollutants);

K_K – coefficient of each element concentrations which is calculated for each element separately by the formula:

$$K_K = C / C_f, \tag{2}$$

C – determined concentration of chemical element in the sample (mg/kg);

C_f – background concentration of chemical element in the sample (mg/kg).

Z_d index is used in cases when the subject that is analysed is contaminated by several substances or chemical elements (Lithuanian Hygiene Standard 60: 2004).

2. Results

42 samples were analyzed using XRF method and concentrations of heavy metals were determined on the right and left banks of Nemunas River at Alytus city territory.

Nemunas river sediments are not as polluted as some western European countries rivers (Table 1) such as Ireland (Glendasan). This contamination is caused by extraction of metals (mines), heavy industry companies and other polluting objects. There are no such polluting objects in Lithuania thus it leads to the less contaminated soils and river sediments. Heavy metal concentrations in Nemunas river sediments are similar to neighboring countries (Latvia, Poland, etc.).

Table 1. Comparison of HM concentrations in Nemunas river and some rivers in other Europe countries

River	Heavy metals (mg/kg)					
	Cu	Pb	Cr	Ni	Cd	Zn
Glendasan (Ireland)	291	10.22	–	–	56	17.5
Mala Welna (Poland)	13.8	13.4	3.9	8.3	0.85	43.4
Bogdanas (Greece)	44.35	10.98	–	5.23	0.12	130.2
Nemunas (left bank)	26.88	–	60.06	61.1	14.67	27.19
Nemunas (right bank)	21.07	–	39.42	–	13.23	21.6

Although concentrations are much lower in Nemunas river than in rivers that are in western Europe countries as industry develops there is a need to investigate the geochemical changes in Lithuania.

Concentrations of 21 metals were determined which were higher than the detection limit. Lithuanian environmental normative document 20-2005 (LAND 20-2005) requirements for sludge categorization by heavy metals were used to compare and evaluate the quality of the sediments.

There is no normative document that regulates concentrations of heavy metals in river bottom sediments in Lithuania. According to the measured concentrations of heavy metals mean values are calculated separately for the left and right banks of the Nemunas River. Those mean values are used to evaluate the general condition of the sludge and compare with the requirements of LAND 20-2005. Most of HM (Cr, Cu, Zn) concentrations in the surface (0–0.1 m deep) layer sediments meets I sludge category.

Higher pollution by heavy metals (Cd and Ni) is observed on the left bank of the Nemunas River. Those metals concentrations correspond to the II sludge category. Only Cd concentration on the right bank of the Nemunas River exceeds the LAND 20-2005 requirements (Table 2).

Table 2. Sludge categorization by HM concentrations (LAND 20-2005)

Sludge category	Concentrations of heavy metals, mg/kg (*LOD-Level of Detection)						
	Pb	Cd	Cr	Cu	Ni	Zn	Hg
I	<140	<1.5	<140	<75	<50		<1.0
II	140–750	1.5–20	140–400	75–1000	50–300	300–2500	1.0–8.0
III	>750	>20	>400	>1000	>300	>2500	>8.0
Right bank of the Nemunas River	<LOD*	13.23	39.42	21.07	<LOD*	21.6	<LOD*
Left bank of the Nemunas River	<LOD*	14.67	60.06	26.88	61.1	27.19	<LOD*

Such distribution of HM concentrations shows higher contamination load on the left river bank which is possibly caused by industries located in the urban area on the left bank of the river. There are no industrial facilities or other concentrated contamination hotspots on the right river bank.

Cadmium concentrations 8.8 and 9.8 times are greater than required for the I sludge category while nickel concentration exceeds 1.2 times only on the left bank of the Nemunas River. In terms of the worse Indicators River sediments on both banks are assigned to the II sludge category (LAND 20-2005).

Most of the heavy metals concentrations in bottom sediments of the Nemunas River have steadily increased with the exception of Pb and Zn. Cr and Ni concentrations are highest in River sediments (Fig. 5). New industrial companies in 2009–2010 were established in the western part of Alytus city instead of old auto repair, motor transport companies. These industrial companies are producing plastic, rubber products and electronics, thus it could have caused the change of heavy metals concentrations. The increase of Cr, Ni and Cd could probably be caused by the change of industrial activity.

Sediments of the Nemunas River were quite polluted in compare with clean (Skroblus) river: it dozens of times exceeded background values with the exception of Pb which was below the detection limit.

Cu concentration was similar in Kulpė and Nemunas Rivers bottom sediments (Fig. 6) while Cd concentration was 16 times higher in Kulpė river sediments (Lithuanian Environmental Protection Agency 2003).

2.1. The trend of HM concentrations in the sediments of the Nemunas River going downstream

Sample numbers are given on the x axis in Figures 7–12 (dis. – discharges and trib. – tributaries). The change of heavy metals (Cr, Cu, Cd) concentrations on the right and left banks of the Nemunas River was calculated using the trend feature. It is noticeable that Cd and Cu concentrations on the left and right banks increase downstream (Figs 7–12). Concentrations of Cr on the right bank of

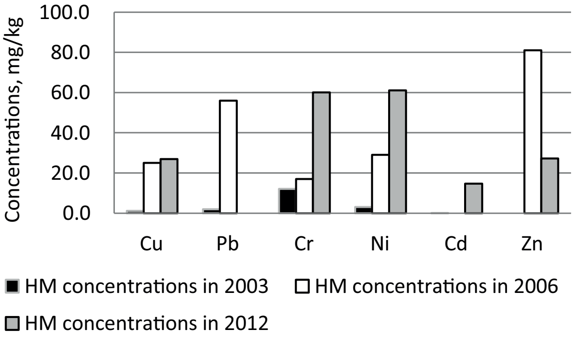

Fig. 5. HM concentrations in Nemunas River bottom sediments

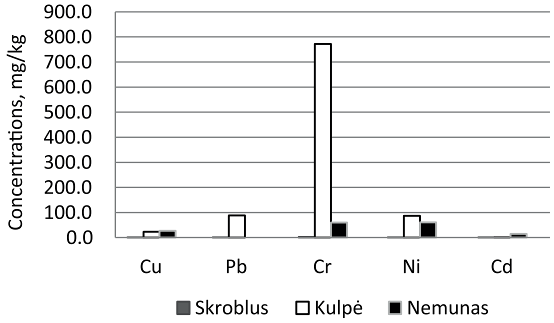

Fig. 6. HM concentrations in bottom sediments of the most contaminated and the cleanest Lithuanian rivers

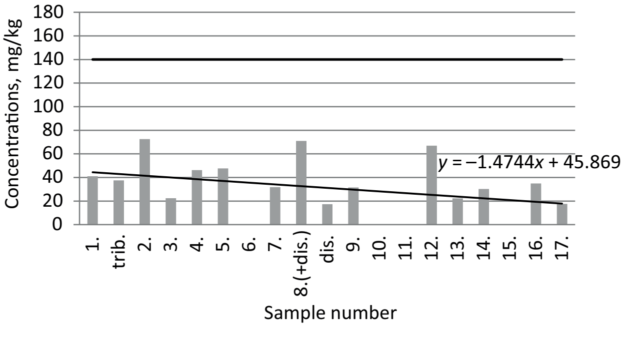

Fig. 7. Cr concentrations on the right bank, downstream trend (*y*), the concentration of the I sludge category (bold line)

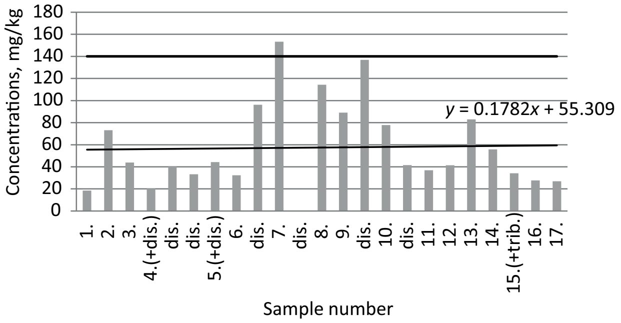

Fig. 8. Cr concentrations on the left bank, downstream trend (*y*), the concentration of the I sludge category (bold line)

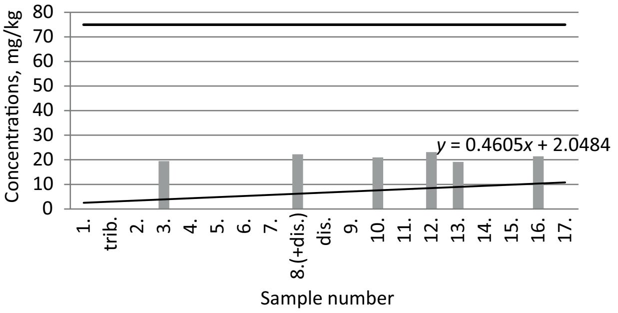

Fig. 9. Cu concentrations on the right bank, downstream trend (*y*), the concentration of the I sludge category (bold line)

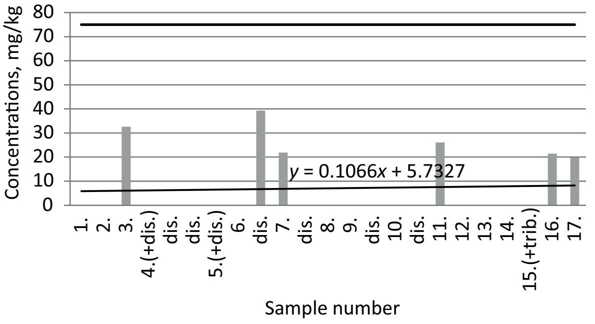

Fig. 10. Cu concentrations on the left bank, downstream trend (*y*), the concentration of the I sludge category (bold line)

the Nemunas River bottom sediments decreases downstream while on the left bank increases (Figs 7 and 8). Also concentrations of heavy metals on the left bank are much higher than on the right bank. This obviously shows higher pollution by heavy metals on the left bank of Nemunas river where are much more dischargers. Those dischargers bring more pollutants from Alytus city and its contamination sources.

All concentrations of heavy metals (Cr, Cu, Cd) are noticeably higher on the left bank of the Nemunas River (Figs 7–12). This is due to the fact that industry is concentrated in this part of Alytus city. Bottom sediments near some of discharges were highly contaminated by heavy metals, especially on the left river bank. Most of the concentrations of HM increased downstream.

The Total pollution index Zd showed that the left bank of the Nemunas River bottom sediments pollution by HM is greater than the right bank. Zd of the right bank is 68.3 while Zd of the left bank is 78.6. These Zd values are attributed to hazardous contamination category ($32 < Z_d < 128$) according to Lithuanian Hygiene Standard 60: 2004. It also shows the higher contamination by HM on the left bank of Nemunas River.

Conclusions

1. The analysis of heavy metals of the Nemunas River bottom sediments showed increased concentrations of 5 elements (Cd, Ni, Cr, Cu, Zn) in 2012. These heavy metals are considered to be the most dangerous.

2. The highest concentrations that exceed the permissible environmental standards were set to Cd and Ni. The major exceedances are specific to Cd from 8.8 to 9.8 times above the permissible limits respectively on the right and left banks of the Nemunas River.

3. Sediments were assigned to the II sludge category according to LAND 20-2005 in terms of the highest concentrations of Cd and Ni in bottom sediments on both banks of the Nemunas River.

4. Heavy metals concentrations in bottom sediments dozens of times exceed the cleanest river (Skroblus) values

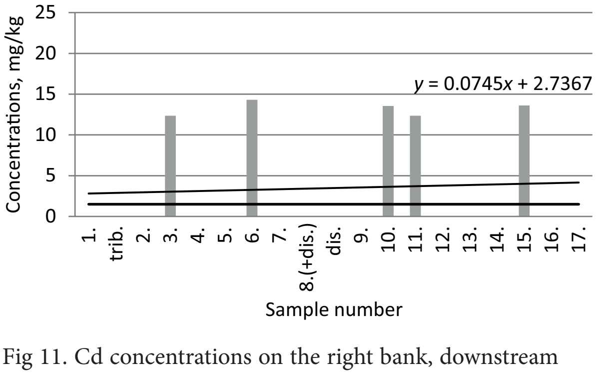

Fig 11. Cd concentrations on the right bank, downstream trend (*y*), the concentration of the I sludge category (bold line)

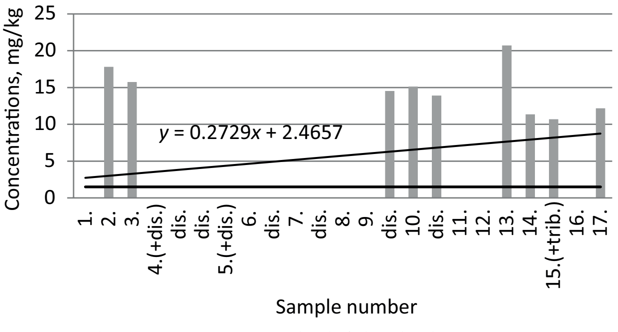

Fig 12. Cd concentrations on the left bank, downstream trend (*y*), the concentration of the I sludge category (bold line)

with the exception of Pb which was below the detection limit. Most of the heavy metals concentrations do not exceed the most contaminated river (Kulpė) values.

5. Zd of the right bank is 68.3 while Zd of the left bank is 78.6. According to Zd index – 13.1% higher contamination by heavy metals is observed on the left bank of the Nemunas River. Such distribution of contamination is due to the anthropogenic load and industrial enterprises located on the left part of the Nemunas River bank.

6. Trend equations showed the rising concentrations of heavy metals (Cr (except right bank), Cu, Cd) going downstream the urban area. This shows the anthropogenic impact of the Alytus city on the Nemunas River ecosystem.

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