

Assessment of Lake Bottom Sediment Pollution by Lead and Cadmium

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Abstract

The aim of the current study is to investigate the relationship between physico-chemical parameters, and to determine levels of certain heavy metals (Pb and Cd) in bottom sediments of Lake Sudotėlis in Lithuania. Organic matter (OM) content and pH were measured. Both positive and negative correlations were detected between the lake bottom sediment content and physical-chemical parameters. The pH of the sediment in all sites was slightly alkaline, e.g. from 7.76 ± 0.02 to 8.21 ± 0.02 . Quantity of organic matter in bottom sediments was 10.37 ± 0.30 to $76.50\pm 1.6\%$. The metals concentrations (in mg/kg of dry weight) ranged 0.42 ± 0.02 to 12.73 ± 0.60 for Pb, and 0.02 ± 0.004 to 6.89 ± 0.33 for Cd.

Keywords: lake bottom sediments, lead, cadmium, pH, organic matter

Introduction

Heavy metal (HM) contamination of the environment is a worldwide problem that has attracted a great deal of attention [1, 2]. Heavy metals are potentially toxic to crops, animals, and humans [3]. Toxic effects of metals depend on their properties [4]. Soil is a major reservoir for contaminants as it possesses an ability to bind various chemicals [5]. In the contaminated soil metals can be dissolved in solution, absorbed by organic and inorganic soil particles, and form a complex with the soil water [6].

Thus, evaluation of toxin concentrations in the environment allows taking measures to improve the quality of life and environmental conditions [7]. One of the most important methods of environmental pollution with heavy metals is industrial wastewater contamination [8]. Surface water bodies are polluted with heavy metals. Heavy metals accumulate in the bottom sediments – indicators of environmental pollution.

In Lithuania, there are 2,850 lakes larger than 0.5 ha and about 3,358 that are smaller (0.05-0.5 ha) [8, 9]. Total

national lake percentage is about 1.5% [10]. Intense economic activity in lake basins during the last decades has had a decisive influence on the majority of Lithuanian anthropogenic landscape, lake ecosystem, hydrochemical, and other feature changes, including changes in lake vegetation. Most affected were small (0.5-20 ha) and shallow (2-7 m average depth) lakes, where silting is noticeable in the majority. Over the past decade the quality of waste water has changed due to stricter environmental requirements, construction of wastewater treatment facilities in many settlements, and changes in the structure and intensity of agriculture, thus significantly reducing surface water pollution. In general, Lithuanian lakes are not contaminated, just several lakes located in towns, which still suffer from wastewater discharged into them a couple of decades ago. They are polluted with technogenic pollutants from factories, workshops, etc. The lakes are: Mastis (Telšiai), Talkša (Šiauliai), and Didžiulis (Trakai distr.), lakes smaller than 50 ha are Dailidės (Alytus), Sudotėlis (Švenčionys distr.), Babrukas (Trakai), and a few smaller lakes. Sedimentary deposits of eutrophic lakes generally have a high organic matter content (above 15%) [11], and can be used as equivalents to peat in different fields – balneology, agriculture,

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livestock farming, and construction. So far there is no specific methodology in Lithuania for sapropel contamination assessment, so it is measured on the basis of “Requirements for sewage sludge use for recultivation and as a fertilizer” LAND 20:2005 [12].

Probably because the majority of lakes in Western Europe in the 19th century were contaminated with heavy metals, lake silt (sapropel) is not included in EU directives (e.g. No. 889/2008) as valuable fertilizer and is often compared to sewage sludge containing heavy metals of technogenic origin.

In most cases, heavy metals in sapropel are incorporated and/or immobilized in organogenic origin compounds, in sewage sludge – heavy metals of technogenic origin are usually found [10]. Sapropel accumulation in lakes is a slow process. If there is more water vegetation, accumulation of sapropel increases tenfold [13]. There are opportunities in Lithuania to clean up lakes and use sapropel as fertilizer and for improvement of soils [14, 15].

The aim of this work is to investigate the relationships between physical-chemical parameters, and to determine levels of some heavy metals (Pb and Cd) in bottom sediments of lake Sudotėlis in Lithuania.

Methodology

Sudotėlis has been polluted with municipal and industrial wastewaters for a long time, because there were no sufficiently efficient wastewater treatment plants. An iron foundry started operating in the region in 1945. A water bowl was installed in 1974. Municipal and industrial wastewater was cleaned mechanically, polluted water went into the lake, and from the streamlet flowed into the Žeimena River. The iron foundry was closed in 1990. A wastewater treatment plant was built in 2008.

Heavy metals (lead and cadmium) were selected for study due to their big toxicity and potential pollution of wastewater according to LAND 20:2005 requirements. The quantity of organic matter and pH of lake bottom sediments were determined for evaluation of accumulation and migration of tested heavy metal regularities.

Sudotėlis is located near Švenčionėliai (Fig. 1). The research area lies between latitude 55°7'17 and 55°7'39 N, and longitude 25°58'26 and 25°58'49' E in Eastern Lithuania. Sudotėlis (10.09 ha area) is a lake of exogenous glacial origin in Northeastern Aukštaičių plateau, belonging to the Žeimena River basin. The lake is located 89 km from Vilnius. The average depth of the lake is 2.57 m. In the bottom of the lake, large amounts of sediment were accumulated; the average of formed silt layer is 6-7 m [16]. The estimated volume of the lake is about 260,000 m³.

At the northwestern coast of the lake, forest area is equipped with a modern city wastewater treatment plant, operating since October 6 2008. Wastewater treatment equipment, previously located near Sudotėlis lake, was not working efficiently and removed only about half of the pollutants. These wastewater treatment plants were built in 1974, wastewater was cleaned mechanically, polluted water went into the lake, and from the streamlet flowed into the Žeimena.

Lake Bottom Sediment Sampling and Chemical Analysis

Density of the boreholes in the lake depends on the size of the lake and the bottom undulation. Small lakes (up to 3.0 hectares) are drilled every 50-100 m, 3.0 to 10.0 hectares every 100-200 m, and 10-50 hectares every 200-300 m. The area of Sudotėlis is 10.09 ha, therefore 10 places for sampling located approximately 100 m from each other, according to the requirements (Fig. 2), were



Fig. 1. Sudotėlis Lake location.

Table 1. Depth of sampling boreholes.

Borehole	Depth, m	Coordinates
A	1, 2, 3	55°7' 37.00 N, 25°58' 29.79 E
B	1, 2.5, 4	55°7' 34.41 N, 25°58' 27.70 E
C	1, 3, 5	55°7' 35.62 N, 25°58' 32.09 E
D	1, 3, 6	55°7' 32.61 N, 25°58' 30.59 E
E	1, 4, 8	55°7' 29.46 N, 25°58' 31.86 E
F	1, 5, 9	55°7' 26.09 N, 25°58' 34.08 E
G	1, 8, 12.5	55°7' 23.34 N, 25°58' 34.47 E
H	1, 5, 10	55°7' 20.41 N, 25°58' 33.58 E
I	1, 5, 9	55°7' 19.47 N, 25°58' 41.15 E
J	1, 2.5, 4.5	55°7' 21.81 N, 25°58' 47.24 E

selected. Lake bottom sediments were sampled according to standard [17]. Samples were taken from 10 different locations closest to the source, the outflow from the lake site, and the bay from different depths: surface sediment layer (A - 1, B - 1, C - 1, etc.), subsurface sediment layer (A - 2, B - 2, C - 2, and so on), and the bottom of the lake with the mineral part of the bottom layer (A - 3, B - 3, C - 3, and so on). Sampling was performed in the following order: A to J. Water layer thickness ranges from 1 to 3.5 m and lake bottom sediment depths ranged from 3 to 12.5 m, so the sediment samples were taken in the following order (Table 1). The co-ordinates of the sampling locations were recorded with a hand-held GPS (Garmin 72H) according to the method (Table 1).

Thirty representative samples of bottom sediments were collected in total, with each sample representing a composite sample of at least 3 sub-samples. Lake bottom sediment humidity is determined by dewatering the sample at a temperature of $105 \pm 3^\circ\text{C}$ [18] up to the constant mass (analytical balance KERN 770, accuracy ± 0.00001 g) and is calculated considering its mass before dewatering. After drying, samples were passed through a 2 mm sieve and homogenized. Then these samples are burnt at $650 \pm 5^\circ\text{C}$ for 4 hr in a muffle furnace. The organic matter content was determined by means of the loss-on-ignition (LOI) method. The LOI value is expressed as a percentage of weight lost. Each sample (1 ± 0.01 g) was digested according to conventional Nitric-perchloric acid digestion [19, 20]. The digested solutions were diluted in a measuring flask to 50 ± 0.08 ml with deionized water. The solution was filtered through a $\varnothing 0.45$ μm glass filter and analyzed. Concentrations of elements in sediments can be analyzed using ICP-MS [21]. Total concentration of heavy metals, such as Pb and Cd, was determined using graphite atomic absorption spectroscopy (GFAAS, Buck Scientific 210 VGP, USA) at wavelengths (λ) for Pb = 283.3 nm, for Cd = 288.8 nm. A blank sample was prepared in the same way and was used as the standard sample [22]. Quality assurance and control were assessed using the duplicates and blanks method. 10% of samples

were measured repeatedly for quality control. Analytical precision, which was measured by the relative standard deviation of parallel samples, was less than 10 percent. Quantification of trace metal concentrations was based on calibration curves involving standard solutions prepared from a commercial stock solution (trace metals Pb and Cd in 2% HNO_3 , Buck Scientific). These calibration curves were determined several times during the period of analysis. The analysis limits were 0.01 mg/l for Pb and 0.001 mg/l for Cd. Electrical conductivity of deionized water was smaller than 10 $\mu\text{S/m}$, according to the standard [23]. It was measured with Inolab 740. Lake bottom sediment pH values were determined according to the standard [24]. The pH of lake bottom sediments is measured with a MultiCal 538 WTW pH meter with a glass electrode.

Statistical Procedures

All metal concentrations were determined as milligrams per litre for digestion solution and, on a dry weight basis, as milligrams per kilogram for sediment. Statistical analysis of data was carried out using Excel 2007 software.

Pearson rank correlation coefficient was used to test for significant associations between heavy metal levels in lake bottom sediments and physical-chemical parameters. Linear regression analysis was applied to the data to compare the relationships between heavy metals (Pb and Cd) concentrations in lake bottom sediments and pH and organic matter content. Also, linear regression analysis was applied to the data to compare the relationships between Pb

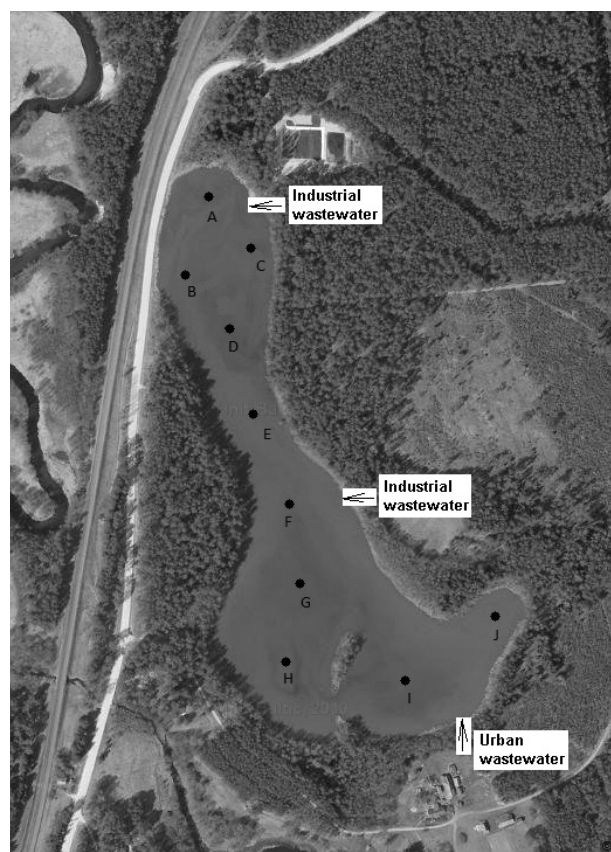


Fig. 2. Sampling points grid (www.maps.lt).

and Cd concentrations in lake bottom sediments. Arithmetic average, median, standard deviation, confidence intervals, and Pearson coefficients were estimated at $P < 0.05$.

Results and Discussion

The most important legal acts regulating waste water sludge and sapropel issues in Lithuania include the Hygiene Standards HN 60:2004 – Maximum Concentrations of Hazardous Chemicals in Soil of 08/03/2004, approved by the Minister of Health [25] and the Environmental Protection Normative Document LAND 20-2005 – Requirements on the use of Sewage Sludge for Fertilization and Reclamation of 29/06/2001, approved by order of the Minister of Environment No. 349 [12]. Order No. 349 aims at regulating sewage sludge in agriculture, energy crops (fast-growing plantations for direct use as biofuels) and the cultivation of damaged areas (quarries, closed landfills, road-beds, etc.) and dumpsites so that no negative effects are caused on soil, vegetation, animals, and humans. Its requirements apply to household and municipal or similar industrial (e.g. food) wastewater sludge.

The order provides limited values for heavy metal concentrations in sludge, soil, and average annual loads. Two kinds of limit values are provided for the concentrations of heavy metals in soil – background limits (which influence the frequency of performing soil analysis), and maximum permitted concentration values – for sand/sandy loam and loam/clay soils.

Furthermore, the sludge is differentiated to categories (I, II, III – depending on heavy metal concentrations) in LAND 20:2005. Sludge of category III and untreated sludge cannot be used. Sludge classification according to heavy metals concentration, presented in MPC, corresponding to II category sludge for Pb is 140-750, and for Cd is 1.5-20 (mg/kg), MPC values corresponding to category I sludge are Pb <140 and Cd <1.5 mg/kg.

If content of heavy metals exceeds the allowed values set in LAND 20:2005, such sapropel must be diluted with clean sapropel or peat before using it as a fertilizer.

Lithuanian Hygiene Standard HN 60:2004 provides maximum permitted concentrations of hazardous substances in the soil. MAC (the maximum allowed/permissible concentration, mg/kg) for Cd is 3.0 and for Pb it is 100.

HM migration depends on a variety of soil properties: texture, clay fraction, rock origin, humus reaction, potassium, soak, and carbonate content, as well as on finely divided fractions and rock background, less from the reaction of humus, potassium, and soak [26, 27].

Heavy metals in lake bottom sediments have been analyzed by many foreign scientists [28-33]. However, sometimes it is difficult to compare measurement results due to using different digestion methodolog. For example, Junhong Bai used a mixture of $\text{HClO}_4\text{-HNO}_3\text{-HF}$ for determination of As, Cd, Cr, Pb, Ni, Cu, and Zn total quantities [28].

Selection of heavy metals for research depends on the aim of the work. J. Jernström et al. selected heavy metals (Co, Cu, Fe, Mn, Ni, Pb, U, and Zn) for bottom sediments of Lake Umbozero in the Murmansk Region (Russia), stemming from contamination by airborne emissions and river transportation from the nearby metallurgical and mining industries [29].

W. Tylmann et al. selected Cd, Cu, Ni, Pb, and Zn for research of lake sediments in northeastern Poland [30]. According to literature data, Pb and Cd are between the most analyzed heavy metals in lake bottom sediments. Quantities of Pb in lake bottom layers are presented in Table 2.

The highest value of Pb concentration (12.73 ± 0.60 mg/kg) was statistically higher in the surface layer than in other layers ($P < 0.05$) (Table 2).

Pb is hardly mobile in oxidious neutral-alkaline conditions because at $\text{pH} > 6.0$ it falls in the form of lead hydroxide. Pb is inert in reduction conditions. Pb forms stable complexes with humus. Mobile forms of Pb make up only about 1.5%, and potentially mobile – up to 10-18% [34]. The highest (6.89 ± 0.33 mg/kg) concentrations of Cd were significantly higher in the sediments of the surface layer ($P < 0.05$).

A very weak negative correlation ($r = -0.200$) between lead and cadmium in all sampling locations of all lake sediment layers was determined. It can be assumed that accumulation of Cd and Pb in lake bottom sediments did not take place at the same time. According to Suresh et al. [33], if the correlation coefficient between the metals is higher, metals have common sources, mutual dependence, and identical behavior during transport. The absence of correlation among the metals suggests that the contents of these metals are not controlled by a single factor. However, it is controlled by a combination of geochemical support phases and their mixed associations.

These heavy metals have partly different sources – the main sources of Pb – industrial wastewater and transport, the main source of Cd – industrial wastewater. There is the road (300 m) near Sudotelis Lake. The level of the natural concentration of metals in lake sediment depends mainly on the geological structure of the catchment and intensity of material transport from the catchment into the lake, as well as on the properties of the sediment itself, e.g., granulometric composition, and organic matter content [31]. Estimating natural fluctuations of metal content in lake sediments of a given region is very important because they constitute a reference value when determining the degree of sediment pollution. If data concerning local or regional natural values are unavailable, usually data concerning the mean content of elements in Earth's crust are used. These values can deviate from the actual natural content, which in turn can lead to an erroneous estimation of the pollution value. A comparison of the mean values does not offer complete information and can be misleading: the variability of natural metal content in sediments of different lakes of the same region can be high. Due to this reason it is better to compare median values of heavy metals concentrations.

Table 2. Total heavy metals contents (mg/kg) of the different lake bottom layers.

Borehole	Layer thickness, 0-1 m		Layer thickness, 1-2 m		Layer thickness, 2-3 m	
	Pb	Cd	Pb	Cd	Pb	Cd
A	2.97±0.20	6.89±0.33	0.42±0.020500	0.16±0.00600	1.03±0.05	0.13±0.006
B	2.20±0.16	6.53±0.30	0.95±0.04200	3.40±0.19	5.77±0.24	0.05±0.002500
C	3.71±0.26	0.09±0.006	1.82±0.12	0.02±0.004	1.56±0.08	0.02±0.004
D	2.44±0.17	0.10±0.005	3.37±0.23	0.05±0.00300	3.44±0.13	0.05±0.00200
E	12.73±0.605	0.33±0.0146	8.40±0.37	0.26±0.00900	11.54±0.52500	0.25±0.012
F	7.12±0.32	0.25±0.0126	3.45±0.19	0.16±0.007	4.91±0.24	0.22±0.011500
G	3.62±0.21	0.19±0.0116	5.23±0.24	0.29±0.011	3.68±0.20	0.15±0.00600
H	2.22±0.15	0.08±0.004	3.19±0.14	0.15±0.00600	4.20±0.21	0.14±0.0070500
I	6.20±0.29	0.23±0.0126	3.77±0.16	0.18±0.00700	5.64±0.28	0.11±0.00500
J	5.30±0.29	0.19±0.0136	3.22±0.18	0.10±0.005	3.94±0.20	0.08±0.00400
Min	2.20	0.08	0.42	0.02	1.03	0.02
Md	3.67	0.21	3.30	0.16	4.07	0.12
Max	12.73	6.89	8.40	3.40	11.54	0.25

Min – minimum concentration, Md – median concentration, Max – maximum concentration.

G. Suresh et. al. studied the contents and spatial distributions of heavy metals (Cd, Cr, Cu, Ni, Pb, and Zn) in surface sediments of Veeranam Lake. The total heavy metal contents in the sediments decrease in the order Zn>Cu>Cr>Ni>Pb>Cd [32]. We fixed a similar tendency for Pb>Cd in almost all boreholes of Sudotelis Lake except A and B points. The reason for increased Cd concentration in these points can be input of industrial wastewater near point A. Quantities of Cd in lake bottom sediment layers are presented in Table 2.

The pH value range of 7.76±0.02 to 8.21±0.02 for the studied bottom sediments suggests slight alkaline conditions of the soils after fertilization. The concentration of organic matter in the bottom sediments of Sudotelis Lake varied from 10.37±0.30 to 76.50±2.0% (dry matter). Accelerated soil acidification promotes increased element mobility, which raises the risk of their higher accumulations in crops, and their transfer to the food chain and/or their transport to groundwater [35]. Correlation levels between concentrations of Cd and pH are low ($r=-0.300$), but correlation levels between concentrations of Pb and pH are moderate ($r=0.422$).

Organic material forming in the water, migration activity, and trans-boundary transfer of elements have the greatest influence on background values of biogenic-anthropogenic association (Ag-Zn-Pb-Mo-Sn-P); therefore, river and lake sediments are characterized by their highest contents [36]. Analyzed concentrations of Pb in Sudotelis lake bottom sediments weakly depends on the organic matter quantity ($r=0.177$), but correlation level between concentrations of Cd and quantity of organic matter is moderate ($r=0.496$).

Moisture content of the Sudotelis Lake bottom sediments is presented in Table 3. During the production of bri-

quettes with sapropel binder, moisture content in sapropel is an important and integral factor [26]. Recommended moisture content in the briquette production samples is 78-82%, in pellet production 52-60%. Sapropel moisture content in Sudotelis is from 70.37±0.50 to 98.20±0.40% (Table 3). Samples taken from Sudotelis are only partially appropriate for production of briquettes with sapropel binder due to its high content of moisture and low content of organic matter in some samples. The average value of organic matter contents of the sediments was 49.33%, indicating sediments with average organic matter quantity (Table 3).

The European Water Framework Directive [37] that imposes the obligation to improve the ecological condition of the lakes by 2015 upon EU member countries introduces activities aimed at restoring the original, good state of water. The lakes are important for the preservation of freshwater and recreational purposes if they are located near cities, thus they ought to be cleaned. During the cleaning process of lakes, large amounts of sapropel are extracted. The implementation of lake restoration projects will have a direct economic benefit by creating new (or maintain existing) jobs; realization of sapropel would allow at least partial lake cleaning cost reimbursement.

Solving the problem of utilizing the silt removed from the lake as early as during the design stage of lake cleaning is recommended. Since only in a couple of Lithuanian lakes (e.g. Mastis, Talkša) was silt (sapropel) in some places contaminated with heavy metals and/or oil products, the absolute majority of lake silt (or sapropel) can be used without restrictions for fertilization of surrounding fields or settlement greenery. The HM concentrations in Didžiulis Lake silt also does not exceed maximum permitted concentrations (MPC) in the soil, whereas the concentrations of man-

Table 3. Moisture and organic matter content of the of Sudotėlis Lake bottom sediments (%).

Borehole	Moisture content (%)			Organic matter content (%)		
	Layer thickness, 0-1 m	Layer thickness, 1-2 m	Layer thickness, 2-3 m	Layer thickness, 0-1 m	Layer thickness, 1-2 m	Layer thickness, 2-3 m
A	98.20±0.40	93.94±0.33	93.83±0.40	74.81±1.8100	26.88±0.8100	17.73±0.5100
B	96.58±0.45	94.17±0.40	94.57±0.33	76.50±1.6000	74.70±1.6100	24.88±0.7100
C	94.40±0.40	89.25±0.45	89.08±0.31	31.43±1.12,100	30.09±0.5100	23.21±0.9100
D	91.40±0.49	90.65±0.40	81.10±0.28	54.48±1.500	36.63±0.6100	10.37±0.3100
E	96.18±0.35	96.39±0.51	70.37±0.50	55.98±1.400	60.80±1.3100	47.96±0.8100
F	95.96±0.42	95.80±0.28	79.22±0.41	62.52±1.600	58.68±1.200	49.90±0.7100
G	97.49±0.39	97.15±0.40	74.15±0.40	70.37±1.600	68.39±1.3100	55.01±0.7100
H	97.04±0.40	96.38±0.42	89.04±0.38	64.18±1.500	57.05±1.4100	45.35±0.7100
I	96.08±0.48	95.87±0.52	93.71±0.40	63.86±1.500	59.64±1.3100	39.85±0.8100
J	93.08±0.39	97.29±0.43	96.06±0.43	57.29±1.400	38.38±0.900	43.12±0.7100
Min	91.40	89.25	70.37	31.43	30.09	10.37
Md	96.13	95.84	89.06	63.19	57.87	41.49
Max	98.20	97.29	96.06	76.50	74.70	55.01

Min – the minimum value, Md – median value, Max – the maximum value.

ganese, copper, and lead in some samples are close to MAC [38]. For fertilization of about 100 m³/ha, and for soil improvement of about 500 m³/ha, dried-up silt or sapropel can be used.

Conclusions

1. Sudotėlis Lake bottom sediments, according to organic matter quantity, can be attributed to silica-organic type. Samples taken from Sudotėlis lakes are only partially appropriate for the production of briquettes with sapropel binder, due to its high content of moisture and low content of organic matter.
2. The pH of the sediments in all sites was slightly alkaline. Hence the migration of heavy metals into deeper lake bottom sediment layers and migration to deeper layers of soil is limited if sapropel is used as fertilizer.
3. A weak correlation ($r = -0.200$) between lead and cadmium in Sudotėlis lake sediments was determined at chosen significance level $\alpha = 0.05$.
4. Analyzed concentrations of Pb in Sudotėlis lake bottom sediments weakly depend on the organic matter quantity ($r=0.177$), but correlation level between concentrations of Cd and quantity of organic matter is moderate ($r=0.496$).
5. Concentrations of tested heavy metals (Cd and Pb) did not exceeded their MPC values for II category sludge presented in LAND 20:2005; therefore, Sudotėlis lake bottom sediments can be used as fertilizer without restrictions.

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