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ENVIRONMENTAL PROTECTION

Geochemical Indicators of Technogenic Pollution of Bottom Sediments of Small Rivers in an Urbanized Environment

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Abstract—We have explored the possibility of using the geochemical characteristics of migration and accumulation of alkali metals (lithium, rubidium, and caesium) as the indicators of anthropogenic status of heavy metals in bottom sediments of small rivers of a technogenically disturbed (urbanized) environment. A study was made of the behavior of the above elements in bottom sediments of the rivers of Petrozavodsk (Republic of Karelia). The chemical composition of the sediments used in the study was determined with X-ray fluorescence spectrometer ARL ADVANT'X and mass spectrometer XSeries-2 ICP-MS; the content of organic matter in bottom sediments (from the LOI index) was estimated by the weighing method upon heating the samples under investigation to the temperature of 1100°C. As a result of the investigations, it was found that among the heavy metals, Co, Ni, Cu, Zn, Mo, Sb, W and Pb are of predominantly technogenic origin in river sediments, which Cr and Cs are of predominantly natural origin due to a high background of these elements in Quaternary formations of the study area. We determined a close association of the trace elements of technogenic status as well as of Li, Rb and Cs with content levels of iron-manganese formations and organic matter in river sediments, which does indicate a commonalty of the processes promoting their input into the urban water stream. It is established that the commonalty of accumulation of a number of heavy metals in bottom sediments with lithophylic elements (Li, Rb and Cs), due to the high chemical activity of these latter, makes it possible to use the geochemical characteristics of the alkali metals as the indicator of technogenic status of the main pollutants of the urban environment.

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FORMULATION OF THE PROBLEM

Study into the consequences of the transformation of the geological environment across technogenically disturbed territories is an important component of comprehensive ecological investigations and environmental measures. A disturbed state of the upper part of the Earth's crust brings about changes also in other environments thus influencing largely on communities of living organisms populating the aerial, solid and aquatic spaces of biosphere. In areas of large cities that are partial manifestations of technogenically disturbed territories, such research efforts are called for because of a continual interaction between humans and a modified environment [1].

Particularly dangerous among the pollutants of a technogenically disturbed environment are heavy metals (HM) and their compounds which do not decay, have toxic effects and are able to be involved in trophic chains of living organisms, and to accumulate in many of them. Water bodies in zones of enhanced anthropogenic pressures are vulnerable to various pollutants, including HM [2]. Material composition of bottom sediments (BS) in water bodies and streams that serves as a peculiar kind of repository for various mineral and organic compounds reflects the picture of a long-lasting (historical) anthropogenic impact on hydroecosystems as well s on their drainage areas [3].

Formation of chemical composition of BS is influenced considerably by the geochemical features of rocks in the study territory; therefore, determination of the technogenic status of HM is a most important problem in ecological geochemistry and toxicology. Abnormal concentrations of metal pollutants can be associated not only with anthropogenic pressures on the hydroecosystems under study but also with the natural background of the relevant trace elements forming part, as impurities, of various minerals. One possible way to resolve this problem would be by seeking indicator elements accompanying pollutants in the process of migration and/or accumulation in BS of anthropogenically disturbed territories.

The objective of this paper is to explore the possibility of using the geochemical characteristics of migration and accumulation of alkali metals (lithium, rubidium, and cesium) as indicators for the technogenic status of heavy metals in BS of the Lososinka and Neglinka rivers of Petrozavodsk.

OBJECTS AND METHODS

The stated objective and the accompanying goals were attacked in the study of BS from two small rivers, Lososinka and Neglinka, flowing in their low course across the central portion of Petrozavodsk, the main city of the Republic of Karelia (Fig. 1). Petrozavodsk is a large industrial and transport hub in the north-west of the Russian Federation. The main contribution to pollution of the urban environment comes from emissions from industrial enterprises (both in operation and currently closed), automobile and railroad transport (the railroad station, and the locomotive shed of Petrozavodsk are historically located in the central part of the city).

The accumulation level and the spatial distribution of HM in soil cover and BS of the Lososinka and Neglinka correspond to the geography of the main sources of pollution on the territory of Petrozavodsk [4–8]. The rivers receive HM largely with the water flows at the time of spring floods and heavy rains in summer and autumn [9]. A peculiar kind of funnels "capturing" pollutants and redirecting them to the river waters are provided by the controlled and uncontrolled systems of storm sewage situated along the entire length of the urban stretches of the Lososinka and Neglinka [10].

For the purposes of our investigation we analyzed BS samples from the Lososinka and Neglinka as collected as part of our summer fieldwork in 2011. Sampling was carried out by using the Ekman-Birge grab sampler according the generally accepted procedural recommendations [11, 12]. We investigated the upper (within 10cm) layer of fluvial sediments comprising a mixture of Quaternary bedrock formations and modern sediment loads. Mineral composition of BS is dominated by quartz, albite, microcline, actinolite, muscovite, diopside, augite, chlorite, tremolite, and richterite. Results of granulometric analysis indicated a predominance of sand fractions of fluvial sediments [13].

Samples were dried to reach an air-dry state. A maximum preservation of the clay fraction was achieved by a separate drying of the liquid portion of the sample, in glass Petri dishes flushed with distilled water. Samples were screened by using a standard screen with 0.1 mm meshes. Chemical composition of the clay-aleurite fraction of fluvial sediments is a reliable indicator of pollution of hydroecosystems, because it is governed by processes of anthropogenic nature on technogenically disturbed territories [5, 7, 14].

The contents of SiO_2, Al_2O_3, Fe_{tot} and TiO_2 in samples of aleurite-clay fraction of BS were determined with X-ray fluorescence spectrometer

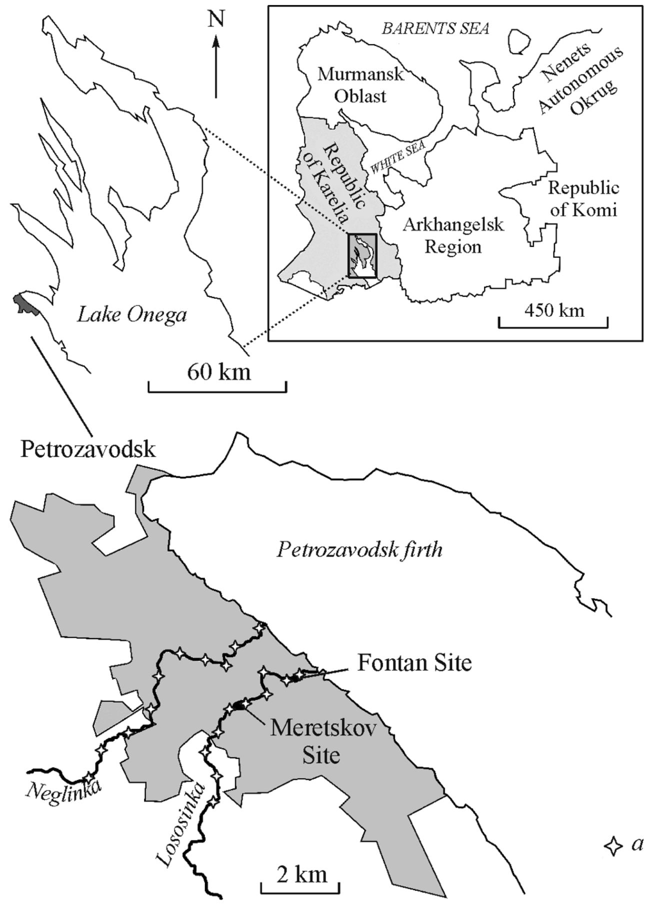

Fig. 1. Schematic location map of the study objects.
a – bottom sediment sampling locations.

ARL ADVANT'X (Thermo Fisher Scientific). The preparation of a sample for analysis included melting the sample and flux in gold-platinum crucibles in the electrical fusion machine for sample preparation, Katanax K1 (SPEX SamplePrep), cooling-down of vitreous (amorphous) melt, and the manufacture of a glass disk for measurements. Losses on ignition (LOI) were determined by a weighing method following the heating of the samples under investigation to 1100°C. In investigating BS, this indicator can serve as a good qualitative characteristic of organic content in water-borne sediments [15]. Data validation after calcinations was carried out with Thermal Analyzer NETZSCH STA 449F1 (temperature maximum 1200°C). The sample loses its main weight in the range from 200 to 450°C, which is the result of the burn-out of organic compounds in fluvial sediments.

The contents of Pb, Sb, Cu, Zn, Co, Mo, Ni, Cr, Cd, Li, Rb and Cs in BS samples (<0.1 mm fraction) from the Lososinka and Neglinka were determined by a mass spectrometric method with XSeries-2 ICP-MS (ThermoScientific). Samples were decomposed by oxygen flashing in an open system. Sample weights of

0.1 g were used in the analysis. In addition to samples analyzed, control idle samples, and one standard (control) sample (GSO 7126–94) – chemical composition of bottom silt from Lake Baikal BIL-1, were decomposed.

According to the recommendations commonly used in geochemical investigations, interpretation of results from determining the petrochemical composition of BS used a calculation of the hydrolisate (Al_2O_3 + TiO_2 + Fe_{tot}/SiO_2) and organo-silicious (LOI/SiO_2) moduli [16]. A Pearson correlation analysis and a factor analysis by the principal component method were made by using the Microsoft Excel 2007 and PSPP 0.8.1 software programs, respectively. The EasyCapture 1.2.0 and Inkscape 0.48.4 software programs were used for graphical displays of results.

RESULTS AND DISCUSSION

Heavy metals that are supplied to BS from technogenically disturbed territories are closely associated with the main carrier phases, i.e. finely dispersed mineral particles, organic matter, and iron-manganese formations. Furthermore, all of the aforementioned HM carriers in polluted fluvial water-borne sediments form an integral system [17], which leads to a unity of the tightness of correlations of total metal pollutants concentrations both with organic matter of BS and with iron and/or manganese content in them [13]. This is the first statement which, when validated, may well indicate the technogenic origin of HM in sediments of rivers, lakes and other water bodies on urbanized territories. Another confirmation of the technogenic status of HM in different environments (including in water bodies) can be provided by bioindication and its numerous methods which use quantitative (population and biomass) as well as qualitative (determination of species composition) characteristics of living organisms [18].

Accumulation of HM in BS of the rivers of Petrozavodsk is mainly due to the overland runoff from the polluted urbanized territory, which is responsible for the exceedances of median concentrations of pollutants in sediments of urban streams over the mean content of metals in soil cover [6] (Table 1). Background concentrations of HM in BS of the Lososinka and Neglinka, on the contrary, are lower for all of the chemical elements (except for cadmium) used in this study. In fluvial sediments of the urban stretches of the Petrozavodsk streams, the concentrations of HM, such as Cu (median 57 mg/kg), Zn (133.8), Cd (4.6), Sb (1.0), W (2.3), and Pb (25.8), exceed also the Clarke contents of these metals in the solid part of the Earth's crust (according to Vinogradov, 1962) [19]. The concentrations of the other elements (Li, Cr, Co, Ni, Mo, Rb, and Cs), because of their occurrence in rocks, were found to be below the Clarke numbers.

The largest accumulation (with the concentration coefficients > 1.5) in BS on the urbanized territory relative to the background corresponds to W, Sb, Cu, Zn, Co, Mo, Pb, Ni, and Li (Fig. 2). Results from a comparison of samples (city/background) made by using the Mann–Whitney U test also indicate a higher (than in arbitrarily clean sediments in the suburban stretches of the Lososinka and Neglinka) content level of the aforementioned elements in BS within the precincts of Petrozavodsk. It is for cesium and cadmium only

Table 1. Content of heavy metals, Li, Rb and Cs in bottom sediments of the rivers of Petrozavodsk, in the upper part of the Earth's crust, and in the urban soil cover, mg/kg

Elements	Me	S_{Me}	x_{Max}	x_{Min}	Me_{back}	Clarke of lithosphere [19]	Urban soils [6]
Li	15.1	5.3	29.5	6.7	9.3	32.0	–
Cr	53.9	11.0	90.8	33.7	38.4	83.0	–
Co	16.1	5.3	28.1	7.5	7.5	18.0	7.9
Ni	27.9	7.4	43.2	13.3	16.4	58.0	–
Cu	57.0	34.2	178.2	12.4	12.5	47.0	36.5
Zn	133.8	69.5	354.4	43.2	37.3	83.0	84.8
Mo	0.9	0.4	5.3	0.0	0.4	1.1	–
Cd	4.6	2.1	28.7	1.7	4.2	0.1	1.4
Sb	1.0	0.8	3.8	0.1	0.1	0.5	0.8
W	2.3	2.1	20.7	0.1	0.3	1.3	1.0
Pb	25.8	12.2	101.4	10.6	13.1	16.0	25.4
Rb	52.5	8.5	75.3	30.1	47.1	150.0	–
Cs	1.2	0.4	1.9	0.5	1,0	3.7	–

Note. Me – median, S_{Me} – standard deviation of the median, x_{Max} and x_{Min} – maximum and minimum values in a sample, Me_{back} – median value for suburban stretches of rivers. Dash – no data.

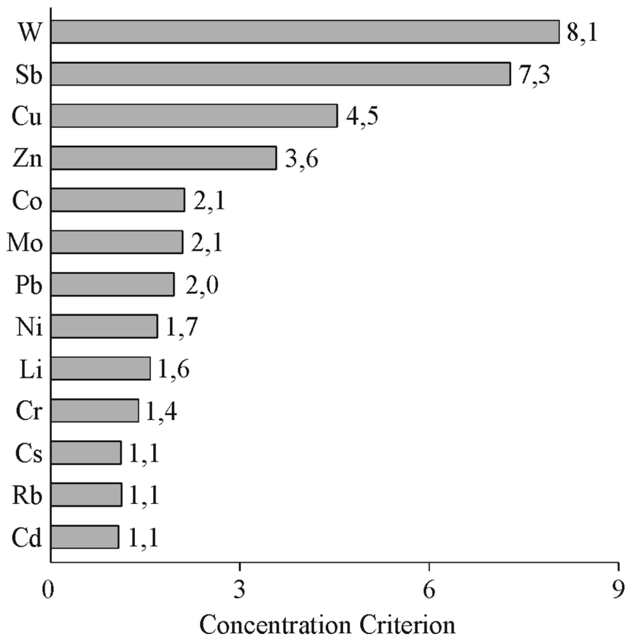

Fig. 2. Values of concentration coefficients of heavy metals, Li, Rb and Cs in bottom sediments of the urban stretches of the Lososinka and Neglinka rivers relative to the background.

that an insignificant difference was determined in the samples of concentrations of chemical elements (Uemp > Ucr at p – 0.01). The other elements are arranged in the following increasing order of the calculated values of U test and, accordingly, a decreasing order in the degree of difference of the samples: Zn (Uemp = 7) < Cu (37) < W (56) < Co (62) < Ni (71) < Pb (86.5) < Sb (103) < Li (117) < Cr (127) < Mo (134.5) < Rb (310) (Ucr = 343 at p – 0.01).

Correlation analysis revealed a statistically significant positive level of tightness of the correlations between all of the trace elements studied in BS of the rivers of Petrozavodsk at 99% confidence level (Table 2). The only exception is provided by cadmium which is a distinctive antagonist to all metals, except for chromium. It is found that cadmium, having an utmost importance in ecological investigations, but because of its toxic properties for living organisms, has mostly a natural origin in BS of the Petrozavodsk rivers [20]. As one of the accompanying elements, cadmium forms part of the structure of hydrothermal zircons of ore amphibolites in the north of Karelia (from 74 to 600 mg/kg of the weight of a mineral grain) [21]. Fluvial sediments of Petrozavodsk exhibit a high positive correlation of cadmium with zirconium as well as with hafnium and rare earth elements, the usual accompanying elements of zircons [20], which serves as a confirmation of the aforementioned statement about the geogenic origin of cadmium.

Interpretation of results of factor analysis implies identifying strong technogenic influence (factors 1 and 2) on the formation of trace element composition of BS in the rivers of Petrozavodsk (Table 3). Furthermore, there is a clear differentiation of the elements: Co, Cu, Zn, Mo, Sb, W and Pb tend to factor 1 associated with the input of the aforementioned metals into aquatic ecosystems as pollutants, whereas Li, Rb and Cs tend to factor 2 combining these lithophylic elements because of their high potential of migration activity in geological environments [15]. Nickel has about the same correlation coefficients with the two factors, which appears to suggest its dual origin in fluvial sediments of Petrozavodsk. Factor 3 that combines Cr

Table 2. Correlations between concentrations of heavy metals, Li, Rb and Cs in bottom sediments of urban stretches of the rivers of Petrozavodsk (N = 95)

Element	Li	Cr	Co	Ni	Cu	Zn	Mo	Cd	Sb	W	Pb	Rb
Cr	0.426	1.000										
Co	**0.717**	0.440	1.00									
Ni	**0.802**	0.600	0.904	1.00								
Cu	0.511	0.461	**0.817**	**0.857**	1.00							
Zn	**0.720**	0.408	**0.832**	**0.872**	**0.853**	1.000						
Mo	0.400	0.354	0.538	0.593	0.654	0.565	1.000					
Cd	–0.449	0.381	–0.270	–0.299	–0.182	–0.310	–0.173	1.000				
Sb	0.473	0.333	0.611	0.689	**0.784**	**0.769**	0.518	–0.211	1.000			
W	0.348	0.419	0.577	0.645	**0.789**	0.637	0.481	–0.137	**0.708**	1.000		
Pb	0.464	0.432	0.472	0.620	0.625	0.684	0.397	–0.152	**0.708**	0.411	1.000	
Rb	**0.846**	0.521	0.614	**0.753**	0.444	0.534	0.414	–0.411	0.350	0.371	0.309	1.000
Cs	**0.756**	0.565	**0.705**	**0.863**	**0.724**	**0.761**	0.571	–0.339	0.609	0.571	0.605	**0.731**

Note. The highest correlation coefficients (R > 0.700), R_{crit} = 0.267 (when p < 0.01) are highlighted in bold type.

Table 3. Factor model for content levels (mg/kg) of heavy metals, Li, Rb and Cs in bottom sediments of the urban stretches of the rivers of Petrozavodsk (N = 95)

Element	Factor		
	1	2	3
Li	0.31	0.88	–0.13
Cr	0.32	0.50	**0.77**
Co	**0.65**	0.59	–0.02
Ni	**0.68**	**0.70**	0.06
Cu	**0.90**	0.30	0.05
Zn	**0.80**	0.47	–0.08
Mo	**0.62**	0.29	0.03
Cd	–0.13	–0.38	**0.88**
Sb	**0.89**	0.16	–0.05
W	**0.80**	0.16	0.08
Pb	**0.71**	0.23	0.07
Rb	0.18	**0.93**	–0.02
Cs	0.58	**0.70**	0.03
Weight of factor, %	40.4	29.8	10.8

Note. The highest correlation coefficients for each element in the series of the factors under investigation are highlighted in bold type.

and Cd is associated with a significant influence of the natural geochemical background on the formation of trace element composition of BS in the Lososinka and Neglinka within the precincts of the city. The behavior of chromium may be attributed to the widespread occurrence of chromium-containing ultrabasic rocks on the territory of Karelia [22, 23].

Thus most of the HM, together with alkali metals, are supplied to the aquatic ecosystems from outside, as a result of the processes of mechanical and chemical weathering and migration from the technogenically disturbed territory. These facts are confirmed by the tightness of correlation between the concentrations of the elements under investigation, and by the hydrolisate modulus of BS in the rivers of Petrozavodsk (Fig. 3). Also, chromium shoed a statistically significant positive correlation for the 95% confidence level only, whereas cadmium exhibited a statistically insignificant negative correlation. Similar regularities are observed in the analysis of the correlation between trace element contents and the organo-silicious modulus (Fig. 4) thereby illustrating the unity of accumulation of the metals studies in fluvial sediments of the city. An analogous decreasing order of the correlation coefficients is observed in investigating the degree of correlation tightness of HM and iron-manganese formations in polluted BS of the Lososinka and Neglinka [17]. Consequently, the top pollutants of the hydroecosystems of the urban rivers include Zn, Mo, Co, Cu, Ni, Pb, Sb and W, while the alkali metals Li, Rb and Cs are their immediate accompanying elements, which is due to the chemical properties of the elements, specifically to the high chemical activity (in the series of electrochemical potentials of the elements, Li, Rb and Cs occupy three extreme left positions relative to hydrogen, with the values of –0, B: –3.04, –2.98 and –3.03, respectively [24]). On the whole, the series of the tightness of correlation between Li, Rb and Cs and HM in BS have a similar form (the elements are arranged in decreasing order of absolute values of correlation coefficients):

lithium:
Ni > Zn > Co > Cu > Sb > Pb > Cr > Mo > W > Cd;
rubidium:
Ni > Co > Zn > Cr > Cu > Mo > W > Sb > Pb > Cd;
cesium:
Ni > Zn > Cu > Co > Sb > Pb > W = Mo > Cr > Cd.

Lithium, rubidium and cesium occur widely in a geological environment. In mineral formations, these chemical elements occur largely as accompanying elements. Only Li and Cs forms their own minerals (very

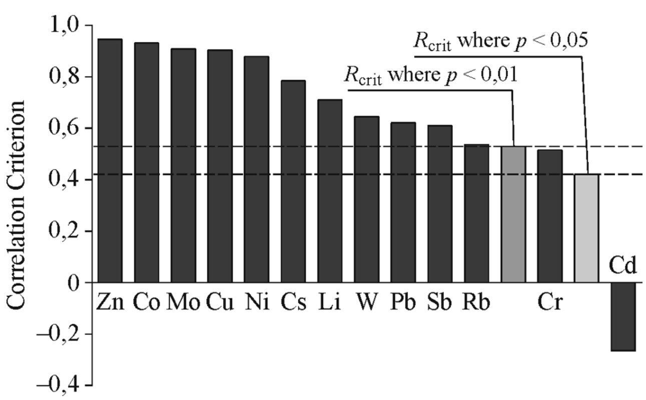

Fig. 3. Correlations between concentrations of heavy metals, Li, Rb and Cs and values of hydralisate modulus of bottom sediments in the rivers of Petrozavodsk (N = 23).

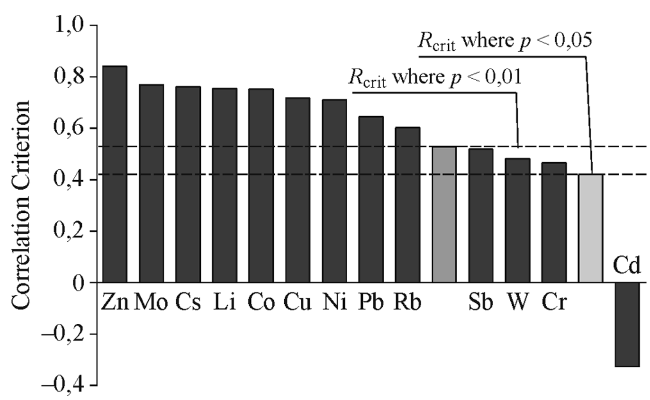

Fig. 4. Correlation between concentrations of heavy metals, Li, Rb and Cs and values of the organo-silicious modulus of bottom sediments in the rivers of Petrozavodsk (N = 23).

rare), and these metals occur less widely in the Earth's crust than Rb which is regarded solely as a dispersed element. In soil formations and BS, the content levels of Li, Rb and Cs correlates with their concentrations in parent rocks (geochemical background) [25]. It was found that an increase in content of clay fractions in loose sedimentary formations leads to an increase in Li, Rb and Cs concentrations. Investigations of Canadian scientists (a case study of Baffin Bay) showed the validity of using an indicator such as Li content in marine BS as the granulometric indicator as well as for standardization of HM concentrations [26, 27]. Also, emphasis is placed on the natural origin of Li (together with Rb in oceanic sediments due to terrigenous detritus-clay erosion material [28]. According to a tight correlation between Li, Rb and Cs in BS of the Sea of Okhotsk (R_{Li-Cs} = 0.76, R_{Rb-Cs} = 0.72 and R_{Li-Rb} = 0.53 (at $p < 0.05$)), evidence is given to the commonalty of migration and accumulation of alkali metals in aquatic ecosystems and their drainage areas [29]. Fluvial sediments of the Lososinka and Neglinka also showed a high level of correlation between the concentrations of the alkali metals under investigation (see Table 2).

The regularities of Li, Rb and Cs accumulation outlined above are characteristic for freshwater hydroecosystems (as exemplified by the RF and USA rivers) [30–32]. Russian researchers established a statistically significant correlation between the contents of Li and some HM in BS of the Volga reservoirs [31, 33]. However, the authors of the just cited references contend that the regularities which they have identified suggest a technogenic origin of Li as well as of metal pollutants in polluted fluvial sediments, because no significant correlation was determined between HM and the basic carrier phases of pollutants in the BS of the Volga used in the study. Since HM as accompanying elements form part of different mineral formations, such ecologo-geochemical investigations are to take into consideration the entire spectrum of methods for determining the technogenic status of pollutants supplied to aquatic ecosystems.

CONCLUSIONS

The Lososinka and Neglinka rivers flowing in the central part of Petrozavodsk are characterized by a high level of anthropogenic pressure along the entire length of their urban stretches. The content levels of HM in BS exceed markedly the arbitrary background concentrations, and the concentrations of metals in soil cover of the drainage area of the Petrozavodsk rivers. The most polluted urban stretches of the Lososinka and Neglinka are the areas with increased contents (in BS) of organic matter, iron-manganese formations and finely dispersed mineral particles, the main carrier phases of HM.

It is found that cobalt, nickel, copper, zinc, molybdenum, antimony, tungsten and lead have largely a technogenic origin in fluvial sediments, whereas chromium and cadmium have predominantly a natural origin due to a high background of these elements in the Quaternary mantle of the urban territory. We point out a unity of accumulation (in BS) of the top pollutions comprising HM and alkali metals (lithium, rubidium, and cesium) liberated from primary mineral formations due to weathering processes. Thus the widespread occurrence of lithium, rubidium and cesium in nature, their high chemical activity and sorption capacity permit the concentration indicators of these elements to be used as the benchmarks of the technogenic status of HM in BS of the Petrozavodsk rivers, a technogenically disturbed (urbanized) territory.

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