Chemistry for Sustainable Development **18** (2010) 623–630

UDC 665.7: 543.31 (282.256.341)

Modern Level of Petroleum Products in Water of Lake Baikal and Its Tributaries

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(Received January 15, 2010; revised January 21, 2010)

Abstract

Data on the determination of petroleum products in surface and near-bottom water of Lake Baikal and its tributaries by means of express method (fluorometry, 168 samples) on board the research vessel are presented, as well as the data on *n*-alkanes and polycyclic aromatic hydrocarbons (PAH) obtained under laboratory conditions by means of gas chromatography/mass spectrometry. It was established that the oil product content of surface waters at the deep-sea stations of reference section does not exceed 10 $\mu g/dm^3$, in water depth and in the near-bottom layer 5–7 $\mu g/dm^3$, including *n*-alkanes – 0.15 $\mu g/dm^3$, PAH (priority pollutants) – 0.012 $\mu g/dm^3$; the concentration of benzo[a]pyrene is up to 10 times below MPC for drinking water (0.005 $\mu g/dm^3$), total content of six PAH compounds under control in drinking water of EEC is up to 50 times lower than MPC_{EEC} (0.2 $\mu g/dm^3$). Increased background of oil products reaching 50 $\mu g/dm^3$ (MPC for fishery) was detected at limited regions of intense navigable waterways. Extremal concentrations of oil products (up to $1.3 \cdot 10^4$ $\mu g/dm^3$), *n*-alkanes (up to 500 $\mu g/dm^3$), PAH (up to 20 $\mu g/dm^3$) were discovered in the samples taken from the water surface of the lake in the regions of natural oil shows. With an increase in the distance from oil show boundaries (oil spots on water surface) the concentrations of oil products decrease sharply but exceed the average value for the water area of the lake almost by a factor of two (11 $\mu g/dm^3$).

Key words: petroleum products, *n*-alkanes, polycyclic aromatic hydrocarbons, pollution level, Lake Baikal

INTRODUCTION

Natural oil shows at Lake Baikal relate to one of the phenomena of its ecosystem. The flux of oil hydrocarbons from the bottom of the lake varies from 0.1 to 2 t/y in oil seepage sites near the Eastern lakeside among which the best known regions are those near Tankhoy station and in the mouth of the Bolshaya Zelenovskaya River. The incoming oil in the form of oil patches on water surface and oil clods in ice cracks relates to biodegraded oil [1]. A new oil show near cape Gorevoy Utes that was discovered in 2005 is characterized by the emission of crude oil on water surface in the amount of up to 4 t/y [1–4]. At the moment of its discovery, the contamination of water surface appeared as oil spots up to 1 m^2 in size over the area approximately equal to 1 km^2.

Emissions from the motors of Baikal boats should be related to the major technogenic sources of petroleum products (PP) in the water of Lake Baikal. According to the records of the Eastern Siberian Ship Inspection, more than 400 boats are officially registered at the water area of Lake Baikal and the Irkutsk water reservoir; the number of boats increases every year [5]. A definite contribution into the pollution of water with oil products is made by residential wastes waters from cities and settlements situated and the lakeside, as well as by the Eastern Siberian and the Baikal-Amur railways passing along the lakeside line [6].

The presence of natural oil shows in the ecosystem of Lake Baikal determines the necessity to take into account the geochemical background when evaluating the anthropogenic contribution into the pollution of water in the lake with PP. However, monitoring of PP in the water of Lake Baikal is performed by controlling organizations using the fluorometric method [7]. With this technique, oil hydrocarbons of technogenic and natural origin, in particular n-alkanes, remain outside the range of determination, while the result of the measurement of the concentrations of aromatic derivatives can be overestimated due to the fluorescence of biogenic products. In this connection, PP determination using various analytical methods is very urgent and has substantial practical importance for the evaluation of the modern level of these pollutants in the water of Lake Baikal and its tributaries.

OBJECTS AND METHODS OF INVESTIGATION

The concentrations of PP in the surface and near-bottom water of Lake Baikal were determined during the joint expedition of the Limnological Institute, SB RAS (Irkutsk), and Irkutsk Centre for Hydrometeorology and Environmental Monitoring in June 2006. To measure PP concentrations, 168 water samples were collected at 84 stations in three hollows of the lake, including the regions of discharge of purified waste water from the Baikalsk Pulp and Paper Mill (BPPM) and at intense navigable paths, in the mouths of the tributaries of the northern Baikal (the Verkhnyaya Angara, the Kichera, the Tyya, the Tompuda and the Rel) and in the source of the Angara River. Petroleum products in the samples were determined directly on board the research ship Vereshchagin using the procedure described in [7]. The resulting extracts (n-hexane, 200 cm^3 of water) were repeatedly analyzed under laboratory conditions by means of gas chromatography/mass spectrometry adding the standard solutions before analysis: 50 mm^3 of squalane (220 $\mu g/mm^3$) and 50 mm^3 of the standard solution of PAH (naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, perylene-d_{12} in acetonitrile, 5 ng/mm^3 of each compound).

Water samples from the tributaries of the Southern Baikal (the Utulik, the Solzan, the Khara-Murin, the Snezhnaya, the Pereyemnaya) and in the delta of the Selenga River were taken in May 2008 and 2009. In the region of oil shows near cape Gorevoy Utes and the mouth of the B. Zelenovskaya water samples were taken from water surface and from different depths of the water body of the lake in July–August since 2005 till 2009. In the points of the reference section of the Baikal, passing along the central part of the lake, surface and near-bottom water was sampled in 2006 and 2009. Water was sampled into glass bottles 1.0 dm^3 in volume, 30 cm^3 of methylene chloride was added, the mixture was shaken for 2–3 min and stored before analysis at 5 °C. Prior to analysis, we added 50 mm^3 of squalane solution (220 $\mu g/mm^3$) and 50 mm^3 of the standard solution of PAH to the samples; then extraction was carried out twice, with 50 cm^3 of methylene chloride. The extracts were united and concentrated to the volume of about 500 mm^3.

Concentrated extracts were twice analyzed by means of gas chromatography/mass spectrometry (Agilent 6890/5973 GC/ MSD system) introducing into the chromatographic column (DB 17ms, 30 m × 250 μm) 2 mm^3 of the concentrate without flow separation in the injector. To determine n-alkanes, the samples were chromatographed with the column temperature gradient from 50 to 300 °C at the heating rate of 10 °C/min and then in the isothermal mode for 5 min at 300 °C; PAHs were determined at the column temperature gradient from 95 to 310 °C with the heating rate of 10 °C/min and isothermal mode for 5 min at 310 °C. The peaks of n-alkanes were recorded in the mode of monitoring the ions with m/z 57 and 71, PAH peaks in the mode of monitoring molecular ions within the analyte retention time intervals under ionization with electron impact at 70 eV.

The quantitative determination was carried out using the internal standard. The calibration characteristics were determined using equation

$m_{an}/m_{st} = kS_{an}/S_{st}$

where m_{an} is the mass of the analyte) PAH or n-alkane), ng; m_{st} is the mass of the standard, ng; S_{an} is the analyte peak area (PAH or n-alkane), rel. units; S_{st} is the peak area of the standard, rel. units; k is the calibration coefficient. Correlation coefficients were not less than 0.985–0.998. Calibration solutions were prepared using individual PAH and a mixture of paraffins in octane (n-C_{18}, n-C_{20}, n-C_{22} and

n-C_{24}) (Supelco Inc., USA). The degree of extraction of n-alkanes and PAH was not less than 75–80 %, the intralaboratory precision of the determination of alkanes and PAHs was 10 %.

RESULTS AND DISCUSSION

Estimation of the concentrations of petroleum products, n-alkanes and PAHs in the water of Lake Baikal and its tributaries

The data obtained by means of fluorometry on the concentrations of PP in lake water (<5–50 μg/dm^3), the tributaries of the Southern (<5–18 μg/dm^3) and Northern Baikal (7–38 μg/dm^3) do not exceed the MPC for fishery water reservoirs (50 μg/dm^3) [8]. The concentration of PP in the surface water exceeds than in the near-bottom water almost by a factor of 2. On the basis of monitoring results, the PP distribution over the water area of Lake Baikal was mapped (Fig. 1).

In the samples of surface and near-bottom water taken in the Southern Baikal, on the side of which the major potential sources of pollutants are situated – residential waste waters from Slyudyanka and Baikalsk cities, the Baikal port, Listvyanka settlement – the concentrations of PP are within the range <5–15 μg/dm^3 (Kultuk–Slyudyanka, Tankhoy–Listvyanka sections). In lakeside water near Slyudyanka and Utulik, PP content is 10–16 μg/dm^3, in the water of the source of the Angara 6–9 μg/dm^3 (the average value of the lake water area is 11 μg/dm^3). It was noted that the concentration of PP in the samples of near-bottom water taken at a distance of 100 m from the deep-laid discharge of wastewater from the BPPM (7–40 μg/dm^3) is higher than that in the surface water (9–22 μg/dm^3). In the samples, PAH and n-alkane content determined by means of GC/MS was within the range ≤0.03–0.10 and ≤0.15 μg/dm^3, respectively (Table 1).

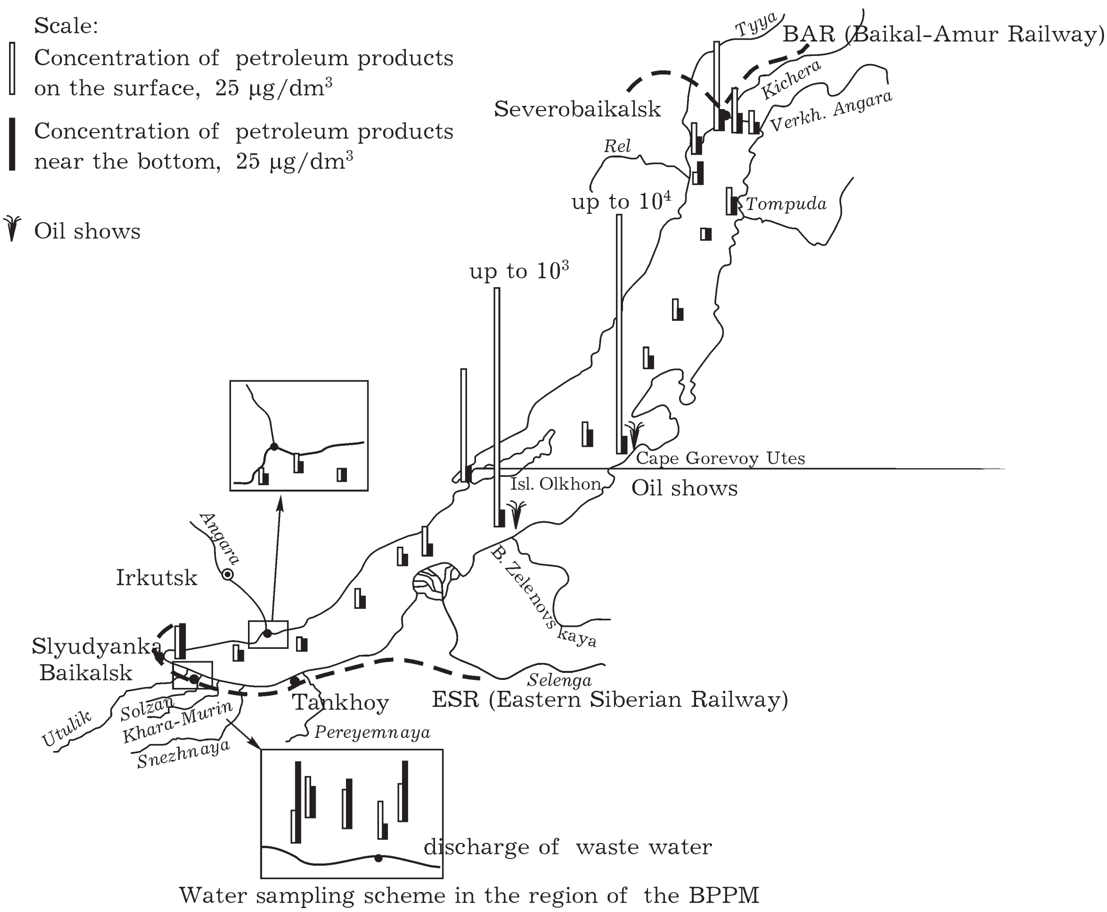

Fig. 1. Petroleum product content in surface and near-bottom water of Lake Baikal, June–July 2006.

The tributaries of the lake at the southern lakeside (see Table 1) are characterized by the high concentrations of PAH in spring [9]. This is likely to be connected with the accumulation of these organic pollutants in the snow cover of the catchment area and their income with melt water. This phenomenon has a clearly pronounced seasonal and year-to-year variability and is not reflected in water purity in the southern hollow of the lake: total concentration of the priority PAHs does not exceed 0.08 μg/L, benzo[a]pyrene 0.0004 μg/dm^3 (<0.1 MPC). The PP content in the water of the Solzan, the Khara-Murin, the Snezhnaya, the Pereyomnaya is characterized by the low level (≤5–10 μg/dm^3), except for the samples from the Utulik River (up to 18 μg/dm^3).

In limited regions of navigable routes – along the south-western side of Lake Baikal, in the Maloye Sea – an increased background of PP is detected. The PP content in water samples collected in this region reached 50 μg/dm^3 (MPC for fishery), while the concentration of oil hydrocarbons (PAH and n-alkanes) determined by means of GC/MS did not exceed 0.03–014 and 0.15 μg/dm^3, respectively.

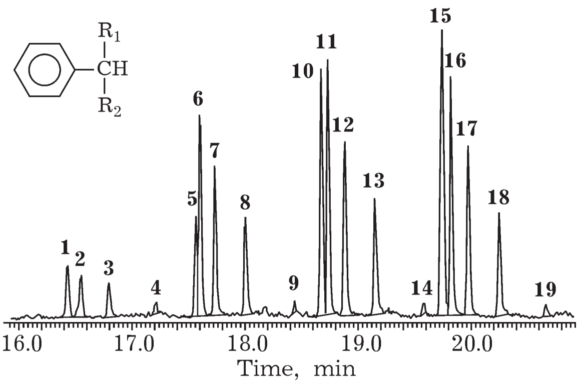

Fig. 2. Mass chromatogram over m/z 91 ion of the extract of near-bottom water from the coastal water near Severobaikalsk. Peaks of alkylbenzenes: **1** – (1-butyl)-hexylbenzene, **2** – (1-propyl)-heptylbenzene, **3** – (1-ethyl)-octylbenzene, **4** – (1-methyl)-nonylbenzene, **5** – (1-pentyl)-hexylbenzene, **6** – (1-butyl)-heptylbenzene, **7** – (1-propyl)-octylbenzene, **8** – (1-ethyl)-nonylbenzene, **9** – (1-methyl)-decylbenzene, **10** – (1-pentyl)-hectylbenzene, **11** – (1-butyl)-octylbenzene, **12** – (1-propyl)-nonylbenzene, **13** – (1-ethyl)-decylbenzene, **14** – (1-methyl)-undecylbenzene, **15** – (1-pentyl)-octylbenzene, **16** – (1-butyl)-nonylbenzene, **17** – (1-propyl)-decylbenzene; **18** – (1-ethyl)-undecylbenzene, **19** – (1-methyl)-dodecylbenzene.

According to the data of the survey of the Selenga River, the ranges of PAH content and the concentrations of aliphatic hydrocarbons in its surface water was 0.001–0.017 and <0.05–38 μg/dm^3, respectively [10]. In the main channels of the delta, PPP were determined at a level of 15–78 μg/dm^3, including PAH up to 0.34 μg/dm^3. With an increase in the distance from the delta, at the sections Krasny Yar–Kharaus, Buguldeika–cape Sredniy, PP concentration did not exceed 5–9 μg/dm^3, PAH (priority compounds) 0.03 μg/dm^3.

In the northern Baikal – along the lakeside line from cape Kotelnikovskiy in the west to the Tompuda River in the east – the concentration of PP in water samples is within the range 5–12 μg/dm^3. In water samples from the tributaries (the Rel, the Tompuda, the Verkhnyaya Angara, and the Tyya Rivers) PP content does not exceed 8–10 μg/dm^3, the concentrations of PAH and n-alkanes do not exceed the detection limit of the procedure used, that is ≤0.03 and ≤0.15 μg/dm^3, respectively.

In the samples of near-bottom water collected in a 0.5-km zone from the mouth of the Kichera River and Severobaikalsk, an increased background of PP was detected: up to 38 and up to 22 μg/dm^3, respectively. The analysis of the sample from the region near Severobaikalsk by means of GC/MS revealed n-alkanes in the concentrations from 0.4 to 1.1 μg/dm^3 and mono-alkyl benzenes with the overall formula $C_{16}H_{26}$–$C_{19}H_{32}$. The latter compounds have unique structure and are represented by five four groups of homologues; five isomers were identified in each group (Fig. 2). The source of the compounds of this class in the environment is thought to be connected with crude oil, in particular, with their presence in natural bitumen [11, 12].

At the deep-water stations of the reference section passing though the central part of the lake (n = 29) PP content in the surface water layer does not exceed 10 mg/dm^3, deeper in water and in the near-bottom water layer 5–7 μg/dm^3, the total concentration of n-alkanes 0.15 μg/dm^3, PAH (priority pollutants) 0.012 μg/dm^3. The concentration of benzo[a]-pyrene is not more than 0.0007 mg/dm^3, which is about 7 times lower than the MPC level for

TABLE 1

Concentrations of petroleum products (PP), n-alkanes, PAH compounds in the surface water of Lake Baikal and its tributaries, μg/dm^3

Sampling region (number of samples)	PP	Sum of n-alkanes	Sum of PAH
Coastal zone, Slyudyanka city (3)	10–16	≤0.15	0.03–0.10
The same, Baikalsk city (2)	9–22	≤0.15	0.03–0.07
Listvyanka–Tankhoy section (2)	<5–15	<0.15	≤0.03–0.08
The same, Krashyi Yar–Kharaus (2)	5–8	≤0.15	0.03–0.13
The same, Buguldeyka–cape Sredniy (2)	6–9	≤0.15	≤0.03
The Angara's source (3)	6–9	<0.15	≤0.03
Oil show, mouth of the B. Zelenovskaya River (10)	50–2000	0.9–7.5	0.43
The same, cape Gorevoy Utes (14)	60–13 000	0.5–13	0.40–20
Maloye sea (4)	<5–50	≤0.15	≤0.03–0.14
Lakeside line cape Kotelnikovskiy–Tompuda River (4)	5–12	≤0.15	≤0.03
Coastal zone Severobaikalsk– the mouth of the Kichera River (4)	22–38	0.4–1.1	≤0.03
Tributaries: the Utulik, the Solzan, the Khara-Murin, the Snezhnaya, the Pereyemnaya (4)	<5–18	–	0.04–0.30
Tributaries: the Rel, the Tompuda, the Verkhnaya Angara, the Tyya (2)	8–10	≤0.15	≤0.03
Delta of the Selenga River (3)	15–78	–	0.04–0.34
Stations of the reference section (29)	<5–10	≤0.15	≤0.03

drinking water, and the total concentration of six PAH compounds that are under control in EEC countries in drinking water is up to 50 times lower the MPC_{EEC} (0.2 μg/dm^3) [13]. Previously [4] the background concentration of n-alkanes in the water of Lake Baikal (homologous series from n-C_{18} to n-C_{33}) in the surface filtrated water and on suspended particles from the depth of 300 and 900 m was estimated at a level of 0.1 and 260 to 90 μg/dm^3, respectively. According to the data published in [15], in the samples of surface water taken along the south-western lakeside of the Baikal, the total concentration of PAH was 0.012 μg/dm^3, in particular for priority pollutants 0.008 μg/dm^3.

Concentrations of petroleum products, n-alkanes and PAH in the water of Lake Baikal in the regions of natural oil shows

The results of monitoring of PP, n-alkanes and PAH in the regions of oil shows are presented in Table 1. The concentration of PP in water samples taken from oil spots reached ex-

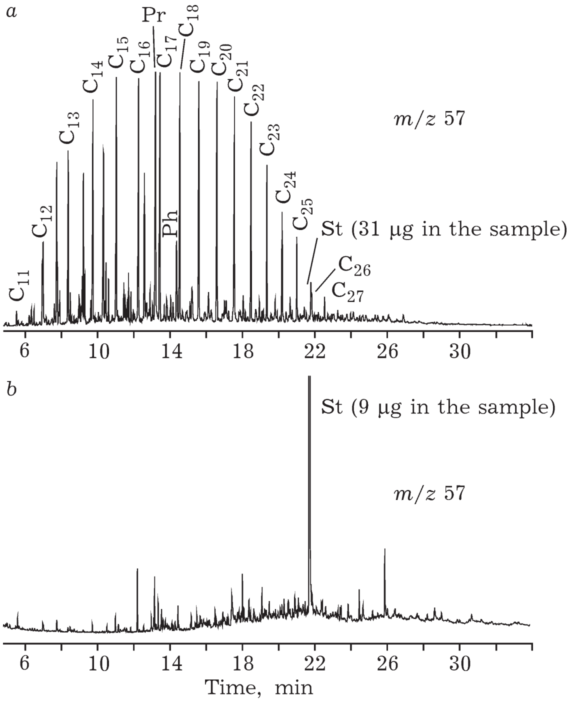

Fig. 3. Mass chromatograms over m/z 57 ion of the extracts of surface water samples collected in different regions: a – oil show in the region of cape Gorevoy Utes, total n-alkane content 13 μg/dm^3; b – the section Listvyanka–Tankhoy, total content of n-alkanes 0.15 μg/dm^3). St is internal standard (squalane).

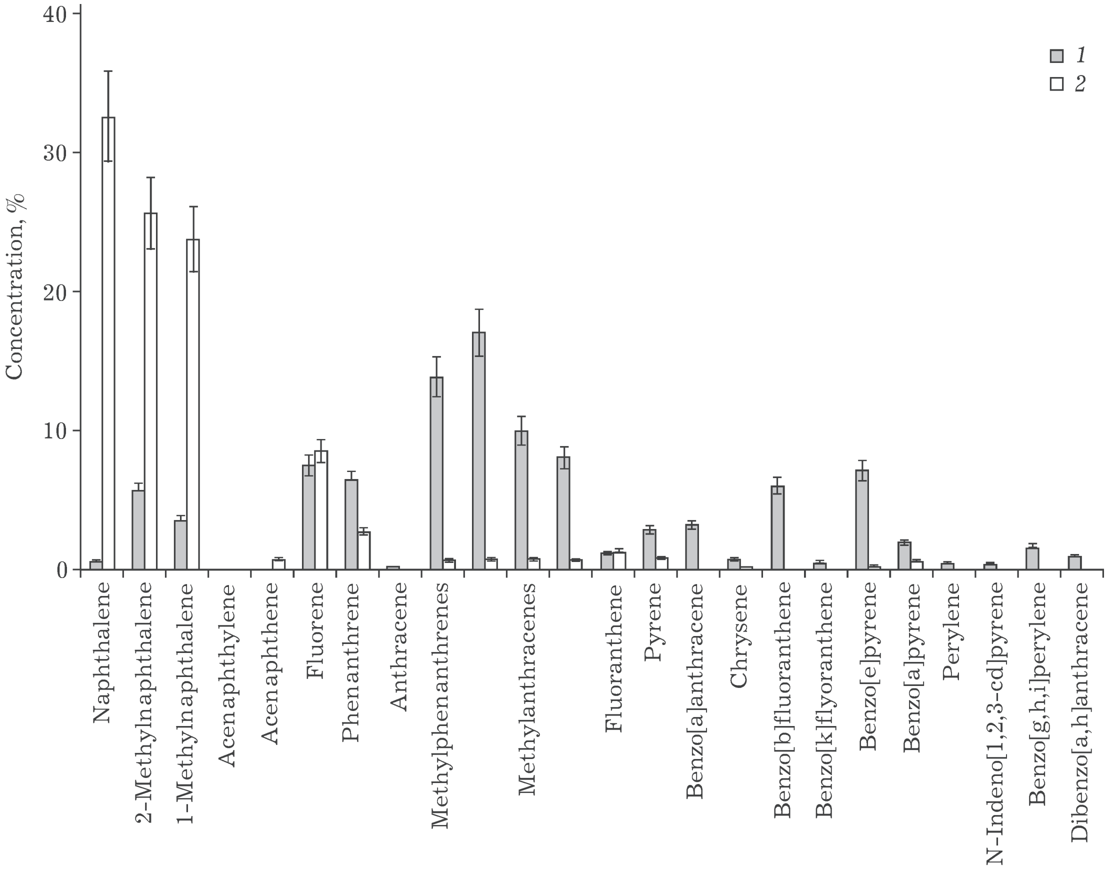

Fig. 4. Qualitative composition of PAH compounds in the samples of surface water in Lake Baikal: *1* – oil show near cape Gorevoy Utes, total concentration of detected PAH compounds 20 μg/dm^3, total concentration of detected PAH 6.8 μg/dm^3; *2* – Listvyanka–Tankhoy section, total concentration of detected PAH compounds 0.16 μg/dm^3, total concentration of priority PAH 0.08 μg/dm^3.

tremal values ($1.3 \cdot 10^4$ μg/dm^3), while it did not exceed 5–8 μg/dm^3 in the samples of near-bottom water. The concentrations of *n*-alkanes in the samples with oil film vary from 100 to 550 μg/dm^3. In water sampled from the lake surface without oil spots and from the deep horizons of the lake, the concentration of *n*-alkanes did not exceed 0.5–13 μg/dm^3. The qualitative composition of hydrocarbons in the samples from the region of oil show near cape Gorevoy Utes was characterized by the homologous series from n-C_{11} to n-C_{33} (Fig. 3), while the samples from the region of oil show in the mouth of the B. Zelenovskaya River, differing by the arrival of biodegraded oil into the lake, *n*-alkanes are represented by the highest homologues (n-C_{22}–n-C_{33}).

The maximal total concentration of PAH in water samples including oil film reached 20 mg/dm^3, for priority pollutants 6.8 mg/dm^3. Among PAH, 22 compounds were identified (14 of them are the priority compounds), at the ratios characteristic of the hydrocarbons of oil origin (phenanthrene/anthracene ≥ 30, fluoranthene / pyrene = 0.4) [16].

With an increase in the distance from the regions of water surface with oil spots, the concentration of PP in near-bottom and surface water decreased sharply and did not exceed 7–40 μg/dm^3 (0.1–0.8 MPC for fishery), while the concentration of *n*-alkanes in the surface water was not more than 12 μg/dm^3. In this situation, a number of discovered hydrocarbon homologues was much more narrow and included hydrocarbons from n-C_{16} to n-C_{24}. At the stations of the reference section the concentration of *n*-alkanes in the samples of surface water did not exceed 0.15 μg/dm^3 (see Fig. 3).

The total amount and the ratios between PAH compounds in water samples outside the

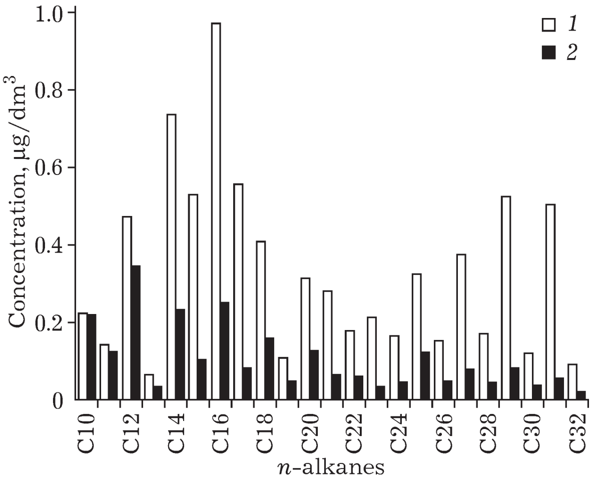

Fig. 5. Relations between n-alkanes in the extract of water sampled in the region of cape Gorevoy Utes: *1* – surface layer (0 m), for fraction n-C_{10}–n-C_{17} OI = 0.54, for fraction n-C_{18}–n-C_{31} OI = 1.55; *2* – water horizon (760 m), for fraction n-C_{10}–n-C_{17} OI = 0.33, for fraction n-C_{18}–n-C_{31} OI = 0.94.

oil show regions changed essentially, too. For example, in the samples from the section Tankhoy–Listvyanka (Fig. 4) among identified PAH the dominating compounds were naphthalenes – up to 80 % of the sum of detected arenes (0.16 μg/dm^3). Most likely, due to the high water solubility (up to 32 000 μg/dm^3 [17]), naphthalenes are conserved in water and transported over long distances, while high molecular mass arenes get sorbed on solid particles of suspension and pass to bottom sediments.

A sharp decrease in the concentrations of PP, n-alkanes and PAH compounds outside the oil show regions points to the limited contribution from this natural source into the pollution of water in Lake Baikal with oil hydrocarbons. It was marked that oil spots are formed on lake surface and disappear with a definite periodicity. The disappearance of spots is possible as a result of their rapid transformation participated by the microbial community of Baikal microorganisms. The contribution from the latter into the degradation process is confirmed by the number of hydrocarbon-oxidizing bacteria: it increased substantially in the region of oil show near cape Gorevoy Utes during the observation period since 2005 till 2007 (from 8 to 2000 times) in comparison with the number of these bacteria in the region of stations that are remote from oil show sites. In the zone of oil spots, the distribution of microorganisms is mosaic, the highest concentrations were detected in surface water with oil film and in the near-bottom region; the major part of cultivated microbial community is able to use hydrocarbons as the only source of carbon [18].

Oil biotransformation process is also confirmed by the appearance of chromatograms of the extracts of water samples; they have a bimodal kind of the distribution of n-alkane homologues with the opposite values of the oddness index (OI) for the low molecular and high molecular weight fractions (Fig. 5). As a result of bacterial action on low-molecular alkanes of oil, even homologues are formed [19], so the ratio of even to odd homologues <1 for n-alkanes with the chain length less than 20 carbon atoms points to biotransformation of oil hydrocarbons. In the case of Baikal oil, OI for the fraction n-C_{10}–n-C_{17} reaches 0.3–0.5 (see Fig. 5). Evidently, as a result of the activity of Baikal microorganisms, limitation of the region of water surface polluted with PP occurs, so that outside it PP content does not exceed MPC for fishery (0.1–0.8 MPC for fishery).

CONCLUSION

On the basis of PP monitoring results obtained by means of rapid fluorometric method on board the research ship for the water of Lake Baikal and its tributaries, and the investigation of the samples under laboratory conditions by means of GC/MS, it should be stated that at the background stations of the reference section the concentrations of PP in the surface water layer do not exceed the level of 10 μg/dm^3 (0.1 MPC for drinking water), deeper in water and in the near-bottom layer 5–7 μg/dm^3, in particular n-alkanes 0.15 μg/dm^3 and PAH (priority pollutants) 0.012 μg/dm^3, the concentration of benzo[a]pyrene does not exceed 0.0007 μg/dm^3 (10 times below MPC). The total content of six PAH compounds under control in drinking water in the countries of EEC is up to 50 times lower than the MPC_{EEC} (0.2 μg/dm^3). It should be noted that in the case of the absence of oil hydrocarbons in the samples of surface and near-bottom water the data of fluorometry are most probably an estimation of the hydrocarbon geochemical background but not pollution of the water of Lake Baikal with oil products. At the same time, identification of n-alkanes and alkyl benzenes in water samples from the region of Severobaikalsk provide evidence of possible anthropogenic character of pollution.

Water in the regions of natural oil shows is characterized by the extremal concentrations of PP at limited regions of water surface (up to $1.3 \cdot 10^4$ μg/dm^3) and includes up to 500 μg/dm^3 n-alkanes and 20 μg/dm^3 PAH compounds. With an increase in the distance from oil show sites, the concentration of PP in surface water decreases sharply; it does not exceed MPC for fishery (0.1–0.8 MCP), which points to the limited contribution from this natural source into the pollution of water in Lake Baikal with oil hydrocarbons. The low level of PP content in the tributaries of the lake, which does not exceed the average value for the whole water area of the lake (11 μg/dm^3), confirms the absence of the pollution of catchment areas with this class of ecotoxicants.

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