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Composition of Hydrocarbons in Soils and Bottom Sediments of The North Crimean Canal within the Settlements of Crimea

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ABSTRACT

The North Crimean Canal was built to irrigate the steppe part of the Crimean Peninsula with the waters of the Dnieper River. It was a source of water supply, so its water quality was controlled. Despite the fact that sediments are a reliable indicator of chronic processes in reservoirs, the state of the canal bottom sediments has not been studied previously. The aim of this work was to determine the hydrocarbon composition of the North Crimean Canal bottom sediments and soils from adjacent territories to assess the level of their pollution and identify probable sources of organic pollutants in the canal bottom sediments. The content of total petroleum hydrocarbons (TPHs) and n-alkanes was determined by the gas chromatography method. The TPHs concentration in the soil on the bank of the North Crimean Canal fluctuated in range of 18,1 to 163,0 mg/kg, in canal bottom sediments – in range of 8,5 to 74,3 mg/kg. The dominant sources of organic compounds entering the canal bottom sediments were the runoff and aeolian transfer from adjacent territories, which was expressed in a high proportion of allochthonous n-alkanes in the bottom sediments. The evidence of relatively recent oil pollution in the composition of the soil and bottom sediments have been identified. According to the biodegradation parameter (Ki) values, oil hydrocarbons in bottom sediments were predominantly more transformed than in soil. Consequently, it could be assumed that pollution of adjacent territories was the source of oil pollution entering the canal bed.

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INTRODUCTION

The most territories of Crimea belong to arid type. The problem of its water supply had been relevant throughout the entire period of the human activity. In order to irrigate the steppe part of the peninsula with the waters of the Dnieper River, the North Crimean Canal (NCC) in the XX century was built (Galkina & Koroleva, 2020). The canal started from the Kakhovsky reservoir and reached the city of Kerch. Its length was 402,6 km, and the length of the primary canal and all the branches exceeded 10000 km (Zotiev & Mokhnoshchekov, 1964). The result of the construction of the NCC, which was completed in 1964, was the water supply of the Crimean Peninsula in the volumes necessary to cover all the needs. The water from the canal was also used for agricultural needs. Due to the difficult geopolitical situation, there were several periods of the water supply of the canal interruption (2014–2022 and 2023–present time). Currently, the water from the Dnieper River does not enter the NCC. The lack of the Dnieper water is partially compensated by alternative sources, which provide a certain filling of the canal bed. Replenishment of the canal is observed during the periods of intense precipitation over the territory of Crimea. As a result of changes in the regime and sources of the water entering the

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NCC, the sources of various compounds, including pollutants, waters are very heterogeneous now.

The most common organic substances carried by river waters are, including those of petroleum origin. A great attention is paid to the problem of studying of hydrocarbon compounds in soils (Zhand *et al.*, 2012; Samia *et al.*, 2013; Gennadiev *et al.*, 2015; Sani *et al.*, 2022; Soloveva & Tikhonova, 2023). This is mainly due to the fact, that vast territories are contaminated with oil and petroleum products during the extraction and transportation of raw materials (Khusnutdinova & Dubinina, 2016). However, petroleum hydrocarbons can also enter the soils as a result of irrigation with polluted waters (Gennadiev *et al.*, 2015), which leads to the migration of hydrocarbons of various origin between soils and water bodies.

NCC is a source of water supply, so its water quality was controlled. Despite the fact that bottom sediments are a reliable indicator of chronic processes occurring in reservoirs, the state of the bottom sediments of the canal has not been studied previously. The assessment of the influence of adjacent land state (oil pollution of soils) on the status of the canal has not been carried out. To assess the influence the riverside soils on the ecological state of watercourses, in particular the NCC, the information on the hydrocarbon composition of soils is needed. The information on the degree of environmental change as a result of the input of oil and oil products, the characteristics of the sources, migration and accumulation of hydrocarbons is urgent. The total content of hydrocarbons in the environment, including oil, characterizes the total petroleum hydrocarbons (TPHs). Thus, the aim of this study was to determine the TPHs composition of the NCC bottom sediments and soils from adjacent territories in order to assess the level of their pollution and to identify probable sources of organic pollutants into the bottom sediments of the canal.

MATERIAL AND METHODS

Sampling. The samples of bottom sediments and soils taken during the spring-summer-autumn period of 2022–2023 according scheme presented (Fig. 1) were the material for the study. Planning of sampling stations position was conducted taking into account possible pass ways of TPHs entering the canal bed (with river water, washout from adjacent territories, atmospheric transfer and precipitation).

The bottom sediments were collected from the bottom of the canal at a water depth of about 20 cm. All the samples of bottom sediments were taken from the upper horizon (0–5 cm) using a manual sampler in three replications. Three samples collected from one sampling site were combined into a mixed sample, which was used for chemical analysis. 12 sediment samples were taken at 4 stations. The sediments in the canal at the station near Krasnoperekopsk city were absent. The pH values in the bottom sediments were measured in situ by a Neutron pH meter-thermometer. After that the samples were packed in special containers and delivered to the laboratory in the sampling day.

The soils were collected near the canal bank at a distance of about 10 m from the water's edge. The soil samples were collected with a metal spatula from the surface layer (up to 5 cm). The sampling was hold using so called the "envelope" method. Five samples collected from one sampling site were combined into a mixed sample, which was used for chemical analysis. 25 samples were collected from five sampling sites in total. The sampling sites were chosen mainly near large settlements (Armiansk city, Krasnoperekopsk city, Dzhankoj city).

Sample preparation. The bottom sediments and soil samples were dried under natural conditions in the laboratory. Large inclusions were removed. The samples were ground in a porcelain mortar and sifted through a 0,25 mm sieve. Further preparation of the extract was carried out according to FR.1.31.2010.08907 "Methodology for measuring the mass fraction of n-paraffin hydrocarbons in samples of soils and bottom sediments of fresh and marine water

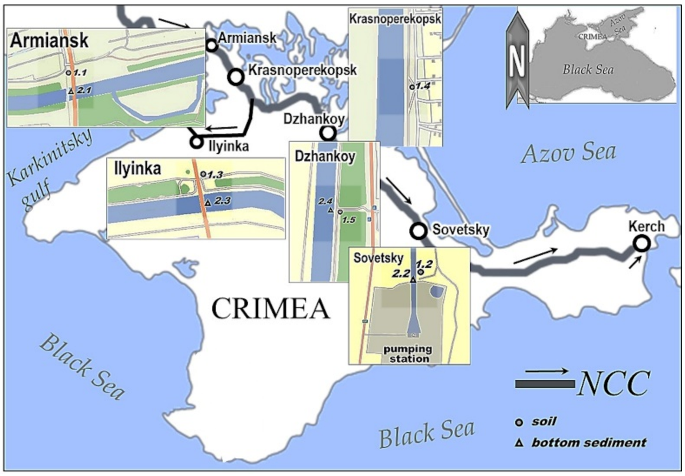

Fig. 1. Scheme of the sampling stations of the bottom sediments and soil from adjacent territories of the North Crimean Canal (NCC) location, 2022–2023

bodies using gas chromatography”. The prearranged sieved samples (1–2 g) ware extracted into 150 ml of n-hexane in a Soxhlet apparatus. The extraction time was one hour. The extract was purified on a glass column (the inner diameter 10 mm) with aluminum oxide (5 g) to remove polar compounds. Then it was concentrated to a volume of 1 ml.

Determination of TPHs and n-alkanes. An aliquot of the concentrated extract (1 μl) was injected with a microsyringe into the evaporator (heated to 250 °C) of Krystall 5000.2 gas chromatograph with a flame ionization detector (FID). TPHs were separated on a 25 m x 0,32 mm HT8 capillary column with a stationary phase thickness of 0,25 μm (SGE Analytical Science). The column temperature was programmed from 40°C to 330°C (temperature elevation rate: 10 °C/min). The carrier gas (helium) flow in the column was 2,5 ml/min without splitting. The detector temperature was 320 °C.

The n-alkanes were identified by matching the retention times of the samples with those of authentic standards of n-alkanes. Quantification of the total TPHs content was performed by calibrating the FID with an external standard TPHs mixture (C_{10}–C_{40}). The standard sample ASTMD2887 Reference Gas Oil (SUPELCO, USA) was used as a mixture of hydrocarbons. The software “Chromatec Analyst 3.0”, the method of absolute calibration and percentage normalization were used for the processing of the results.

Quality Assurance and Quality Control. Before analysis, standard solution of analytes was run to check column performance, peak height and resolution. To ensure quality analyses, standards were injected and analyzed under the same conditions as those used for the samples. Distinct calibration curves, each covering a different range of concentration, were used to quantify the analytes within the linearity range of calibration plot. In this work, the standard addition graph for all n-alkanes in sediments extracted was linear with coefficients of determination (R2) better than 0,97.

The limits of detection (LODs) were calculated as the quotient of three times the standard deviation of the linear coefficient of the calibration line and the slope of the curve. Also, analysis of method blanks (n=5) was performed, and the hydrocarbons concentrations were below their respective limits of quantification (LOQ). For the hydrocarbons, the LODs and LOQs were 0,09 to 0,27 μg/g and 0,27 to 0,81 μg/g, respectively.

The determination of TPHs was carried out on the basis of the Research and Educational Center for Collective Use “Spectrometry and Chromatography” of the Federal Research Center of A.O. Kovalevsky Institute of Biology of Southern Seas.

To determine the probable sources of TPHs input, a number of ratios were used: CPI, TAR,

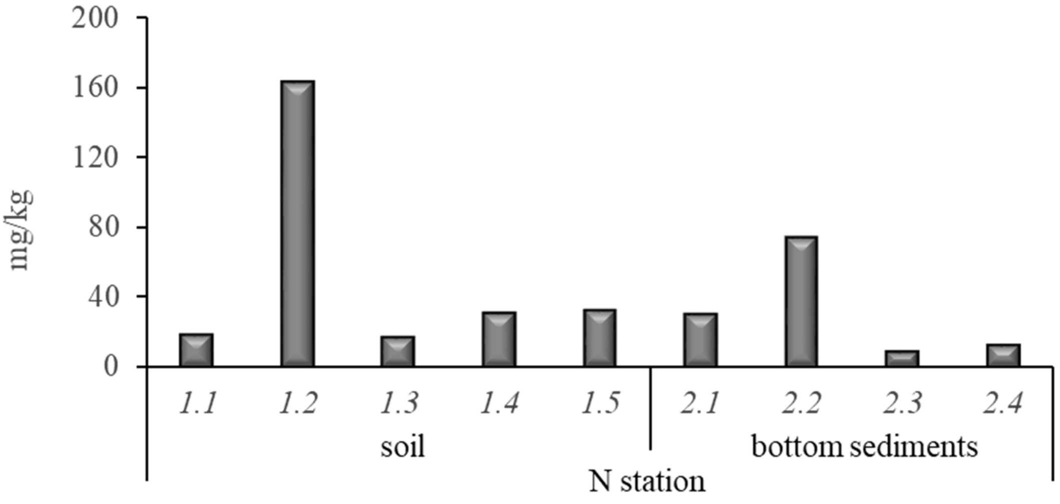

Fig. 2. The concentration of total petroleum hydrocarbons in the soil of adjacent territories and in bottom sediments of the North Crimean Canal (NCC), 2022–2023

Pr/Ph, Ki. The CPI index (ratio of odd and even n-alkanes), which can indicate the biogenic or petroleum origin of TPHs, the contribution of the bacterial community too. The ratio of individual compounds with odd and even numbers of atoms (TAR) allow to estimate the ratio of allochthonous and autochthonous n-alkanes. The ratio of pristane (Pr) to phytane (Ph) characterizes the organic matter diagenesis redox conditions and diagnoses oil pollution. The isoprenoid index (Ki) allows to assess the degree of oil biodegradation. The indices used were determined using the following formulas: $CPI=1/2*\{(C_{21}+C_{23}+\ldots+C_{31})/(C_{22}+C_{24}+\ldots+C_{32})+(C_{23}+C_{25}+\ldots+C_{33})/(C_{24}+C_{26}+\ldots+C_{34})\}$ (Liu et al., 2022);

$TAR=(C_{27}+C_{29}+C_{31})/(C_{15}+C_{17}+C_{19})$ (Bourbonnière & Meyers, 1996); Pr/Ph (Peters *et al.*, 2005); $Ki=(Pr+Ph)/(C_{17}+C_{18})$ (Peters *et al.*, 2005).

RESULTS AND DISCUSSION

It is known that in this region the soil reaction in the upper layer is neutral or slightly alkaline (Zelenskaya & Marinina, 2021). The TPHs content in the soil sampled directly on the bank of the canal ranged from 18,1–163,0 mg/kg (Fig. 2). The levels recorded correspond to slightly contaminated soils (GOST R 54039-2010) with permissible TPHs content (<1000 mg/kg) (Saifullin, 2017). Up to our previous data, the TPHs concentrations in Crimean soils did not exceed 20 mg/kg (Mirzoeva *et al.*, 2023). Similar, low values (55 mg/kg) were noted in soil samples remote from the sites of TPHs extraction, the Balakhani territory of the Absheron Peninsula (Azerbaijan) (Gadzhieva *et al.*, 2016). This territory is considered to be a close analogue of Crimea (Kayukova & Yurovsky, 2017).

In the soil samples, n-alkanes in the range C_{16}–C_{33} were detected (Fig. 3a). The composition of n-alkanes (Fig. 3a) was relatively homogenic and corresponded to the composition of n-alkanes in soils of some arid regions, as well as areas with active vegetation (Teunissen van Manen, 2020; Zhan *et al.*, 2022). A clear predominance of allochthonous compounds C_{29} and C_{31} was detected. An example of a typical chromatogram is shown in Fig. 4a.

In the sample with an increased level of TPHs (st. 1.2, C = 163,0 mg/kg), the appearance of the chromatogram was different from other samples: it had a bimodal character (Fig. 4b). The first maximum was in the low-temperature region, where the chromatogram demonstrated high content of even-carbon short-chain n-alkanes corresponding for the presence of fresh

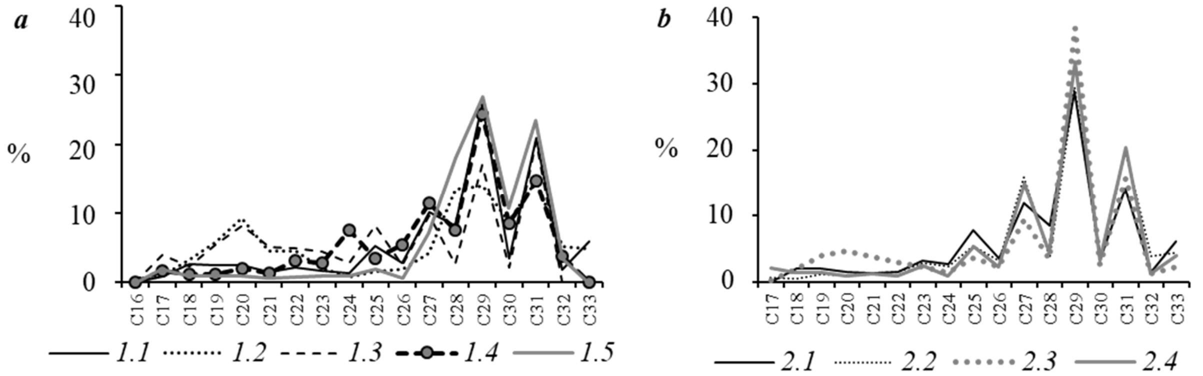

Fig. 3. Distribution of n-alkanes in soils of adjacent territories (a) and bottom sediments (b) of the North Crimean Canal (NCC), 2022–2023

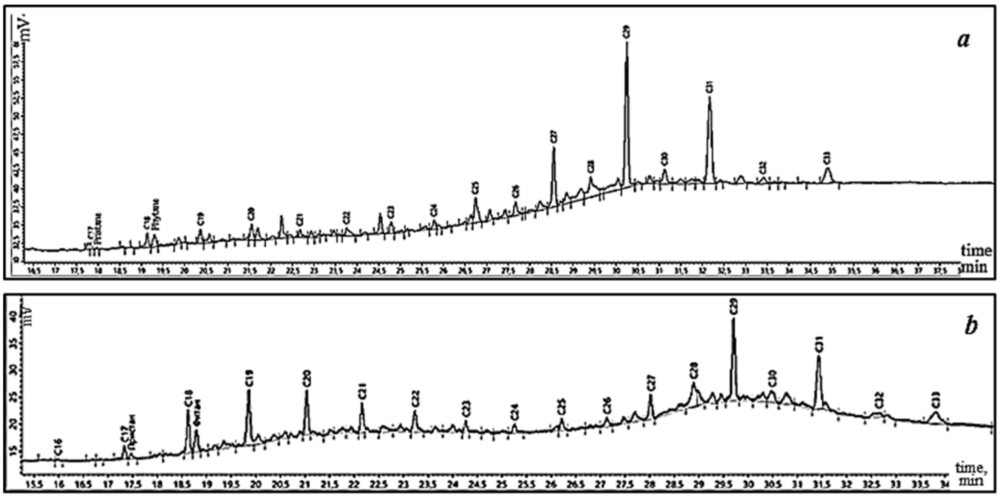

Fig. 4. Examples of hydrocarbon chromatograms in soils adjacent to the North Crimean Canal (NCC): a – typical; b – at station 1.2

oil contamination (Lichtfouse *et al.*, 1997; Jeng, 2006; Rosell-Mele *et al.*, 2018). The second maximum was associated with C_{29} and C_{31}, which was typical for all studied soils. The odd maxima C_{29} and C_{31} presence in surface soils of other regions was noted too (Stolpnikova *et al.*, 2020; Zhan *et al.*, 2022). Their presence is associated with terrestrial plants vegetation (Meyers, 2003; Peaple *et al.*, 2021). It is possible, that the increased content of TPHs was associated with the entry of petroleum products into the soil. The fact is that the bank of the canal, despite the proposed water protection zone, is actively used for recreation. The appearance of cars on the shore is common. Motor transport could be a source of petroleum products entering the surface layer of soil.

The bottom sediments were practically absent in upstream parts of the canal, and were formed mainly in its secondary sections. Thus, it can be assumed that the silt masses did not enter the canal from the Kakhovka Reservoir. They were formed as a result of processes occurring in the canal bed and in the adjacent territories. Sediment samples from the canal were represented by silted soils with pH values in the range of 7,65–7,8. The concentrations of TPHs in the canal bottom sediments fluctuated in the range of 8,5–74,3 mg/kg (Fig. 2). Due to the lack of the sanitary standards for this parameter in the Russian Federation, comparation was held according to the so-called "Dutch lists" (Dutch…, 2000), the standard value of which is 50 mg/kg. According to this document, in one of the samples (in the area of the pumping station in the Sovetskoye village) the elevation of threshold of limit value was noted. The concentrations of TPHs in bottom sediments of the canal corresponded to the concentrations previously noted in the coastal sediments of the river Chernaya (Crimea Peninsula, Sevastopol) – 15–299 mg/kg (Soloveva *et al.*, 2023). In the rivers of other regions, in particular, coastal sediments of the Neuquen River (Argentina), TPHs content varied from 0,4 and 125,0 mg/kg (Monza *et al.*, 2013). There are regions where, TPHs concentrations detected, are usually much higher. For example, sediments collected from the Sadong River (Sarawak, Malaysia) had concentrations of total aliphatic TPHs ranged from 310,4 to 1296,4 mg/kg (Omolayo *et al.*, 2020).

The TPHs content in the bottom sediments of the NCC (8,5–74,3 mg/kg) was somewhat lower than in the soils of adjacent territories (18,1–163,0 mg/kg). Concerning to the ratio of pollutants content in the bottom sediments of the of irrigation canals and the soils of adjacent territories, the literature contains data that the content of pollutants in the bottom sediments exceeds those in adjacent soils by several times. The authors attribute this to the fact that the canals flow near large urban areas (Savic *et al.*, 2013). The NCC flows mainly through agricultural territories, which are relatively favorable in terms of hydrocarbon pollution, so the levels of TPHs in its bottom sediments remain low.

The composition of n-alkanes in all the samples of bottom sediments was of the same type (Fig. 3b): n-alkanes were recorded in the range C_{17}–C_{33}; the distribution of homologs in all samples was unimodal, with the predominance of allochthonous peaks C_{27}, C_{29}, C_{31} (Meyers, 2003; Peaple *et al.*, 2021). These maximums are typical for bottom sediments of rivers and artificial watercourses, and reflect terrestrial plants derived hydrocarbons predominance (Garetova *et al.*, 2020; Commendatore *et al.*, 2004; Wang *et al.*, 2015). This distribution, highly likely, reflected the significant contribution of substances coming from the canal bank to the formation of bottom sediments. The example of a bottom sediment`s TPHs chromatogram is shown in Fig. 5.

The biogeochemical markers of the hydrocarbon`s origin analysis (Table 1) showed that biogenic components bring in the main contribution to the formation of hydrocarbons in the studied soils and bottom sediments (CPI>1). For all the bottom sediments samples CPI was much higher than 1, which indicated the predominance of biogenic components in the formation

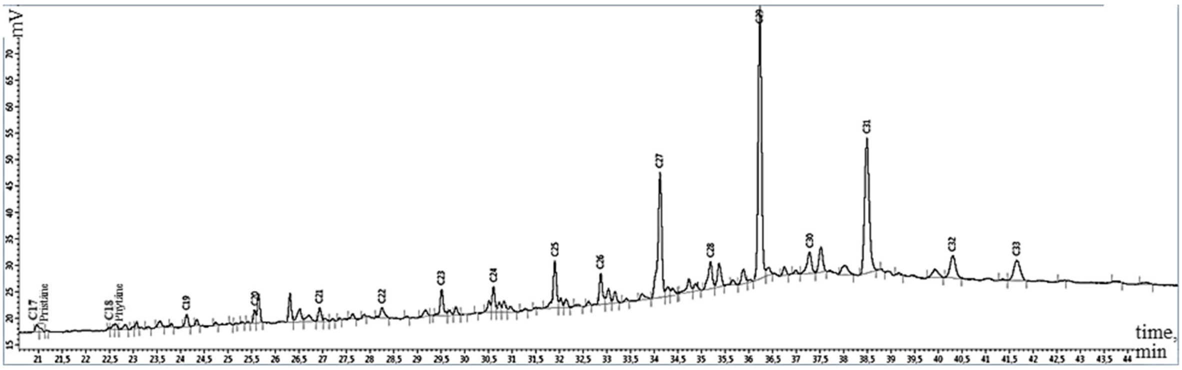

Fig. 5. Typical chromatogram of hydrocarbons from bottom sediments of the North Crimean Canal (NCC) (station 2.2)

Table 1. Biogeochemical markers of hydrocarbons origin

Marker	Soils adjacent territories					Bottom sediments			
	stations								
	1.1	1.2	1.3	1.4	1.5	2.1	2.2	2.3	2.4
CPI	3,39	1,19	4,10	1,59	1,77	3,30	4,30	5,30	6,20
Pr/Ph	0,14	0,18	0,00	0,00	0,20	0,18	0,60	0,00	0,10
Ki	0,72	0,53	0,45	0,28	0,34	1,23	0,68	0,09	0,49
TAR	16,92	4,92	5,04	18,56	20,16	28,19	35,55	15,62	19,96

of the hydrocarbon composition bottom sediments (Yan *et al.*, 2021). For the most soils samples CPI was higher 1. The exception was the station with an increased content of TPHs (station 1.2). For this station CPI = 1,19, which might correspond to the presence of fresh petroleum products. This was consistent with our assumption of oil contamination of the sample, based on the chromatogram configuration. It should be noted that in modern literature there is evidence that in some contaminated soil samples the CPI marker could fluctuate in the range of 1-3 (Liu *et al.*, 2022), so samples 1.2, 1.4 and 1.5 could contain oil pollution.

Phytane, which is associated with oil pollution (Powell & McKirdy, 1973), was found in all the samples, in contrast to the predominantly biogenic pristane (Powell & McKirdy, 1973), which was not recorded in all soil and bottom sediments samples. The ratio of these iso-alkanes (if both were present) had a value characteristic for the presence of petrogenic compounds. This ratio of pristane and phytane may also be a consequence of the reducing conditions of the transformation of organic matter (Peters *et al.*, 2005). However, the formation of reducing conditions in the surface soil horizon and bottom sediments of the flow canal seems less likely than the input of petroleum products.

Isoprenoid coefficient (Peters *et al.*, 2005) for bottom sediments was in the range from 0,09 to 1,23, which covers the range from the presence of fresh (Ki $\leq$0,3) to moderate degraded (0,8$\leq$Ki$\leq$1,5) oil. For soils, the Ki criterion varied within smaller limits (0,28–0,72). These indicators correspond to the presence of fresh (Ki$\leq$0,3) and slightly degraded oil (0,3$\leq$ Ki$\leq$0,80). Average Ki for soils and bottom sediments were 0,46 and 0,62, respectively. Thus, oil products in bottom sediments were deeper transformed than in soils generally.

High TAR marker values (15,6–35,6) in bottom sediments correspond to the great importance of allochthonous compounds in the formation of the hydrocarbon's mixture in the sediments at the bottom of the canal.

CONCLUSIONS

The content of TPHs in the soil on the bank of the NCC fluctuated in the range of 18,1 to 163,0 mg/kg, in canal bottom sediments – in range of 8,5 to 74,3 mg/kg. The elevation of threshold of limit value for soils (1000 mg/kg) and bottom sediments (50 mg/kg) were observed for bottom sediments once.

The dominant sources of organic compounds entering the canal bottom sediments were the runoff and aeolian transport from adjacent territories, which expressed in a high proportion of allochthonous n-alkanes in the bottom sediments.

In the composition of soils and bottom sediments the evidence of relatively recent oil pollution has been identified. Judging from the values of the biodegradation parameter (Ki), oil hydrocarbons in the bottom sediments were predominantly more transformed than in soil. As a consequence of this, it could be assumed that pollution of adjacent territories was the source of oil pollution entering the canal bed.

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CONFLICT OF INTEREST

The authors declare that there is not any conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/ or falsification, double publication and/or submission, and redundancy has been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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