

Research Article**Transformation of Oil-Contaminated Soils of Cryolithozone****Yu. S. Glyaznetsova*, I. N. Zueva,
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1, Oktyabrskaya str., 677980, Yakutsk, Russia; gchlab@ipng.ysn.ru***ABSTRACT.**

The work shows the results of ten year's monitoring of oil contaminated soils in the area of the "Talakan-Vitim" oil pipeline (South-Western Yakutia) where the emergency oil spill occurred in 2006. The results demonstrate that despite an overall trend towards decrease from 91 g/kg in 2006 to 46 g/kg in 2015, after 10 years of the oil spill the contamination level was still very high. As appeared, that self-soils microorganisms had only increased after three years within the emergency spill area. Thus, may say about low rate of self-remediation soils in cryolithozone conditions. The obtained results of study of soils extracts showed, that oil contamination composition had modified and became asphaltene-resinous character as result of transformation oil with domination of oxygen break down processes.

If no new spills occur, an increase in the oil contamination level in individual observation years (2010, 2011, 2015) may consider as oil's ability to migrate in permafrost soils for a long time. The presence of a permafrost layer determines the lateral character of oil contamination spreading. At the same time, during season thawing of grounds oil components partly transported up by thawed and ground waters in overlying soil layers in process of migration along permafrost layer. The process of oil contamination soils becomes stable character as result of supposition of annual additions of new portions of oil components.

High values of residual oil content in soil indicate formation of abnormal hydrocarbons technogenic fields. It is necessary to prolong as a monitoring of the areas disturbed by oil spill as a remediation works.

Keywords: emergency oil spill, oil transformation, cryolithozone, permafrost layer, oil migration, hydrocarbons and asphaltene-resinous components.

INTRODUCTION

Operation of oil and gas complex facilities is often accompanied by various spills, leaks and sometimes even large-scale accidents. Spilled oil getting to a soil is absorbed by it. Oil components mix with soil's native organic matter that leads to increase hydrocarbons content in soils up to formation of abnormal superficial geochemical technogenic fields. The nature of distribution of oil components in soils depends on a number of factors the main of which are: morphological, structural, morphological, structural, material and genetic features of a concrete soil profile, its position in the system of geochemical interfaces of landscape facies, quantity and composition of

the spilled oil, time since contamination (Pikovskiy, 1993; Goldberg, 2001; Margesin, 2001). All this determines the picture of real oil components distribution in the soil profile. It is difficult to estimate consequences of oil spills as oil contamination breaks natural processes and interrelations, changing conditions of dwelling of all live organisms.

Abundance of the cryolithozone is the main feature of northern natural ecosystems, which defines conditions of distribution and destruction of oil in the soil. Permafrost soils in Yakutia are formed in severe climatic conditions and characterized by poor development of soil processes, backwardness of a soil profile, ability

to accumulate both the light and heavy oil products (OP) (Savvinov, 2007). Unlike regions with favourable climatic conditions cryolithozone soils are characterized by much slower bioremediation of spilled oil and OP which conditions lower self-remediation ability of permafrost soils (Glyazentsova et al., 2012; Yang Si-Zhong et al., 2009). While in regions with favorable climatic conditions self-remediation disturbed soils without additional remediation measures lasts 10-25 years in northern conditions destruction of oil and its derivatives may take up to 50 years and more (Pikovskiy, 1988; Oborin et al., 1988; Foght, Westlike, 1992; Bioremediation..., 2008; Lifshits, 2017).

When oil gets on the soil surface and migrates down, the profile its asphaltic-resinous components are mainly absorbed in the upper humic layer. Sometimes these components pack-harden the humic layer decreasing the soil pore space. Asphaltic-resinous components are hydrophobic. They envelope plant roots, dramatically impairing ingress of moisture and nutrients, which results in the death of plants (Oborin et al., 2008). To understand the mechanisms of soil self-cleaning and remediation enhance, the efficiency of cleanup operations and conduct monitoring of objects for their contamination with oil or OP it is necessary to study destruction processes of oil and OP that penetrated soil as a result of emergency spills or leaks at storage or transportation sites.

The main features of adverse oil and OP impact on soil are non-uniform nature of contamination of objects: local OP accumulations in various technogenic forms. Particular forms of local accumulations primarily relate to specific properties of oil and OP soil attributes and presence of permafrost rocks (Glyazentsova et al., 2016).

Talakanskoye oil-gas condensate field is one of the largest in Yakutia on oil production. Before the "Eastern Siberia – Pacific Ocean" pipeline system was commissioned oil, from the field had been delivered to consumers by "Talakan-Vitim" temporary oil pipeline. 24 May 2006 at this oil pipeline there was a technogenic

emergency, as a result of which spilled oil contaminated the surface water of Bezymyanny stream with a total area of 71,650 m², Taloye lake with a water area of 100,000 m², Peleduy river with contamination of its water body 103 km long with an oil film from the mouth of the stream that runs into Taloye lake to the confluence of Peleduy river into Lena river. Apart from water surfaces lands with a total area of some 2.31 ha were contaminated with oil. The oil spill width was 3.5-5 m. The oil contamination penetrated to the depth of 60 cm according to the report of the Ministry of Nature Protection of the Republic of Sakha (Yakutia) it reached from 8.6 to 15 g/kg and more. The total volume of the oil spilled from the oil pipeline as a result of the emergency amounted to 244.68 tons. Some 173.199 tons have got into Taloye lake, Bezymyanny stream and Peleduy river.

The total volume of the oil which has poured out from the oil pipeline as a result of emergency was 244.68 tons. From them about 173.199 tons have got to the lake Taloye, Bezymyanny a stream and the river of Peleduy.

The purpose of studies was to analyse and sum up the results of ten years of monitoring of the soils contaminated with oil as a result of emergency spill, to study peculiarities of oil migration in near-surface soils (0-20 cm) and its transformation in the conditions of natural self-remediation of soils.

MATERIALS AND METHODS

Field works to assess the ecological and geochemical condition of cryogenic soils were conducted each year in summer in the emergency oil spill area (59°57' 44.5"N 112° 10' 03.3"E). To that end monitoring platforms were arranged. At one of the platforms 0.5 ha in the area located in the influx of Bezymyanny stream, 12 test sites 1×1 m in the area were chosen. During all the following years, soil samples were collected from these sites.

The site under study is located within Lensky region of the Republic of Sakha (Yakutia). The emergency spill was recorded at a distance of 1.9 km to a mobile plant 5. Oil flowed

downwards along the slope to the stream contaminating Taloye lake and further on Bezymyanny stream, from the lake to Peleduy river with ingress to the Lena river valley. Geomorphologically, the site belongs to the slope terrain; it features smooth slopes with deeps and corresponding downward direction of runoff towards lower areas. The formation of the latter is due to peculiarities of landscape-forming rocks (geologic environment – parent rocks within the entire near-Lena plateau). A specific peculiarity of the plateau is the prevalence of karstification processes and existence of karst landforms. The landscape is matching – intrazonal watershed-swampy, middle-taiga, continuous permafrost with frequent underlake and underflow taliks. Permafrost thickness is 20-30 m, seasonal melting deepness is 0.3 to 2m.

Dense forests with prevailing pines, birch trees, pine, alder and fir undergrowth, bushes (blueberry, cowberry) are typical of the territory under study; mosses present the ground cover. The terrain is a smooth slope, the microrelief is pitted, pit-and-mount. Soils have a cold profile and below-zero temperatures for 7-8 months a year. A soil profile was arranged to characterize soils on the platform under study. The land cover at a depth of 0-10 (15) cm features peaty humidified dark-grey (closer to black) clay loam with plant roots. Light-brown waterlogged clay loam with inclusions of peaty dark-grey clay loam is found at a depth of 16-60 cm. Seasonally thawed layer water enters the soil profile bottom.

Integrated soil samples were collected from test sites using the “envelope” method as per GOST 17.4.4.02-84. Locations for soil cross sections were chosen so as to cover all soil types of various landscape types and background soil cover areas as much as possible. Samples were selected, labeled and transported according to state standards subject to recommendations. The content of oil and OP was determined by the soil profile: at a dip of 0-20 cm. The total weight of each integrated sample was 1 kg. Soil samples for the quantification of oil and OP were placed in polymer bags, numbered, logged specifying the serial number, sampling date, time and

place, terrain, climatic conditions, soil type and contamination year.

Soil samples in the sites outside of the area affected by the emergency that characterized the natural geochemical background were selected for comparison. Background soil samples were selected pursuant to clause 4 of GOST 17.4.3.01-83: “If comparative results need to be obtained, contaminated and uncontaminated soil samples are collected in identical natural conditions”. Background samples were collected with a clean tool at a distance of 100-150 m to the contaminated area that features the same soil type as monitoring platform soils. All the soil samples collected were dried and plant roots were removed.

The oil contamination level of soil was determined using the method of cold chloroform extraction of soils by extracts yield. To study oil transformation the obtained extracts were studied using IR spectroscopy, liquid adsorption chromatography, and combined gas chromatography mass-spectrometry methods. IR spectra were recorded using “Protege 460 IR Fourier spectrometer” by Nicolet. Liquid adsorption chromatography was used for fractional division of soil extracts into hydrocarbon, resinous and asphaltic components (Uspensky et al., 1975; Method..., 2014).

The hydrocarbon fraction was studied using chromatography-mass spectrometry (GC-MS) method. GC-MS studies were conducted using a system that comprised “Agilent 6890 gas chromatograph” with an interface and “Agilent 5973N high-performance mass-selective detector”. The chromatograph was fitted with a fused silica column 30 m long, 0.25 mm in diameter, impregnated with phase HP-5MS. Helium with a flow rate of 1 ml/min was used as a carrier gas. Evaporator temperature was 320°C. Temperature rise was programmed from 100 to 300°C at a rate of 6°C/min. Ionizing source voltage was 70 eV. A detailed description of study methods is given in (Kashirtsev, 2003; Method..., 2014; Jovančičević B., 2007).

Three replications were used. Data provided in figures and tables are arithmetical averages.

Compared to reference, results obtained are statistically valid.

RESULTS

The average background content of oil hydrocarbons in soils of the area under study was 0.697 g/kg.

Oil contamination level determined in the chosen sites revealed highly, varying pollution rates. Apparently, this is due to the pit-and-mount microrelief with deeps and corresponding downward direction of runoff towards lower areas. Consequently, an average contamination level at all selected sites was calculated as integrated assessment of the oil contamination level of the area under study. Fig. 1 shows the average residual soil content of OP by

observation years from 2006 to 2015. As seen, despite an overall trend towards decrease (from 91 g/kg in 2006 to 46 g/kg in 2015), after 10 years of observation, the contamination level varies strongly and remains very high. It should be noted, that no regulations of the soil residual content of oil and OP have been elaborated for Yakutia, which is why the contamination level was determined by the classification offered by Goldberg (Goldberg, 2001). This classification is developed taking into account the soil type and genesis, climatic zone, OP composition. It allows a differentiated approach to the assessment of the contamination level of area and identification of the local sites, which need top-priority remediation works.

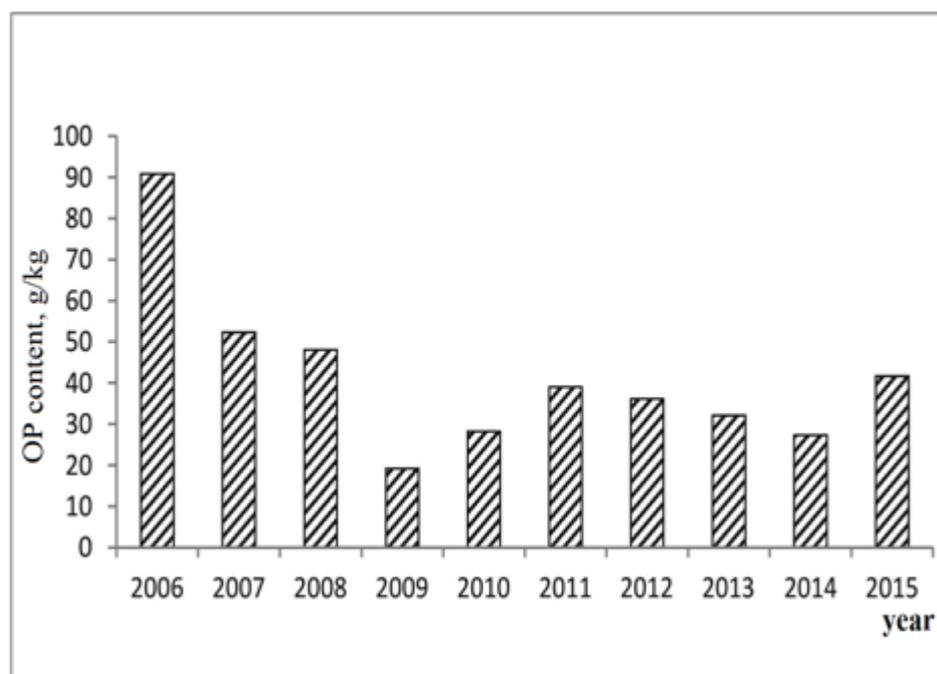


Figure 1. Changes of the average residual OP content in the soil of test sites of the monitoring sites in the area of the oil spill at the “Talakan-Vitim” oil pipeline.

The maximum decrease twice in OP content was determined in the first year of observations. At this stage, oil transformation in soil is mainly due by physical and chemical destruction, gas removal, dissolution, UV destruction (Oborin, 2008). Further, in case of self-remediation of soils the transformation processes connected with biodegradation of contamination under the influence of self-soil oil-destroyers microorganisms usually join (Oborin, 2008).

Consider the changes in oil composition over the period of observations. Fig. 2 shows IR spectra of soil extracts and fig. 4 – changes in relative coefficients of absorption of oxygen groups in reference to hydrocarbon groups by observation years.

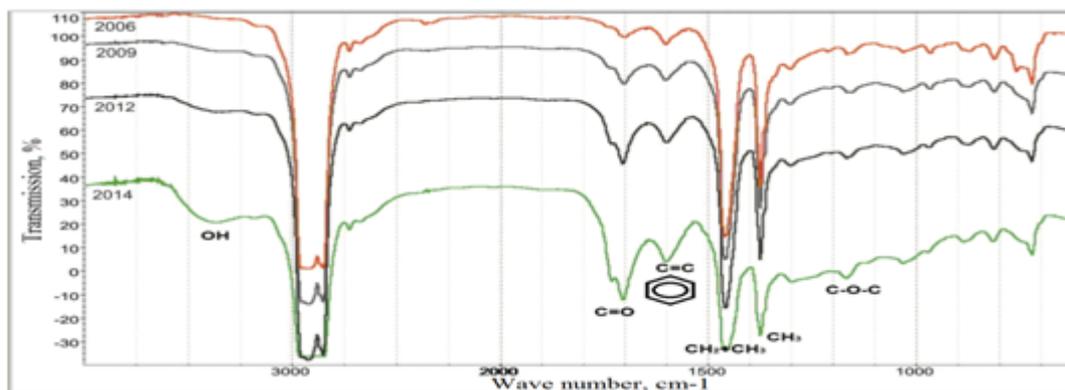


Figure 2. IR spectra of soil extracts collected over different years of observations.

As seen from fig. 2, the following hydrocarbon groups and bonds prevail in the chemical structure of the extract of oil contaminated sample collected in 2006 almost right after the spill: compounds with long methylene chains (absorption band (a.b.) 720cm^{-1}), aromatic hydrocarbons (a.b. 750 , 810 and 1600cm^{-1}). With time, absorption of oxygen groups and bonds increase in spectra compared to hydrocarbon structures. Intensity of a.b. 1700cm^{-1} typical for carbonyl groups and a.b. in the range of $3300\text{-}3400\text{cm}^{-1}$ for hydroxyl groups increases whereas intensity of bands 1460cm^{-1} (methyl and methylene groups) and 1600cm^{-1} (aromatic cycles) decreases. Increase in relative coefficients of absorption of oxygen-containing bonds – carbonyl groups (D'_{1700}) and ether bonds (D'_{1170}), is as a result by observation years (fig. 3 a, b).

Changes in value of relative absorption coefficients of structural groups were calculated by the formula:

$$D'_v = D_v / D_{1460}$$

where D_v is absorbance of a.b.v; D_{1460} is absorbance of a.b. 1460cm^{-1}

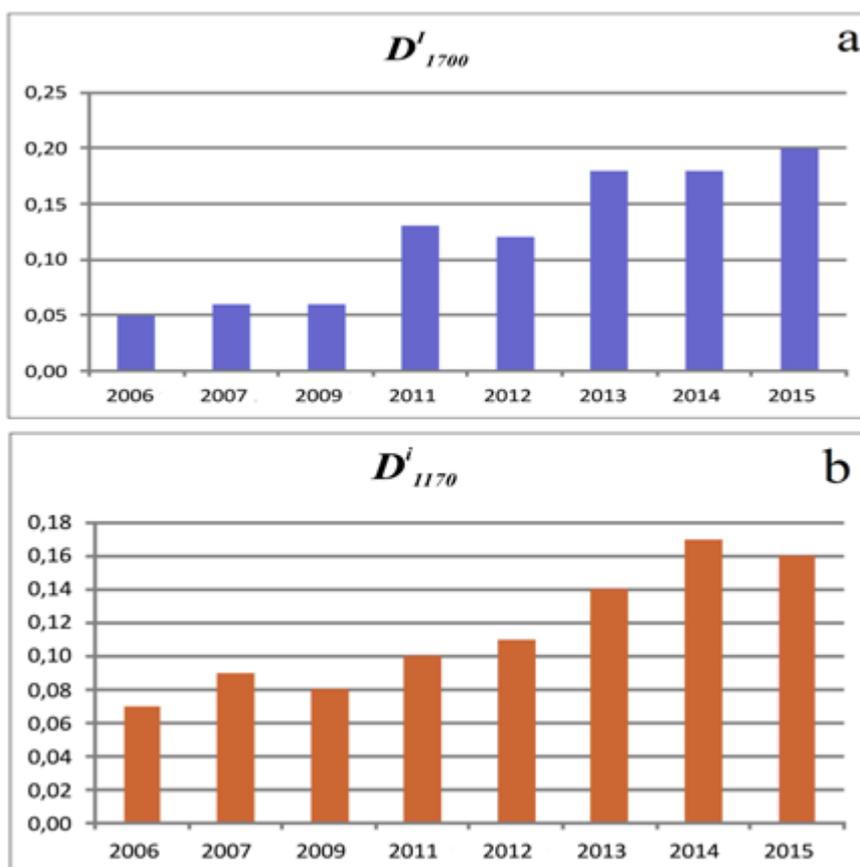


Figure 3. Change in the relative absorption coefficient of oxygen groups in IR spectra of extracts:

a – carbonyl groups; b – ether bonds.

All the above changes in the nature of IR spectra and relative absorption coefficients of oxygen groups compared to hydrocarbon ones is the evidence of oil oxidative breakdown processes in soils.

This is also confirmed by the data on the group fractional analysis of soil extracts (fig. 4). Over 10 years of observation changes in the composition of soil extracts included a decrease in the share of hydrocarbon components and increase resins and asphaltenes. By the end of observations, the contamination acquired asphaltic-resinous composition.

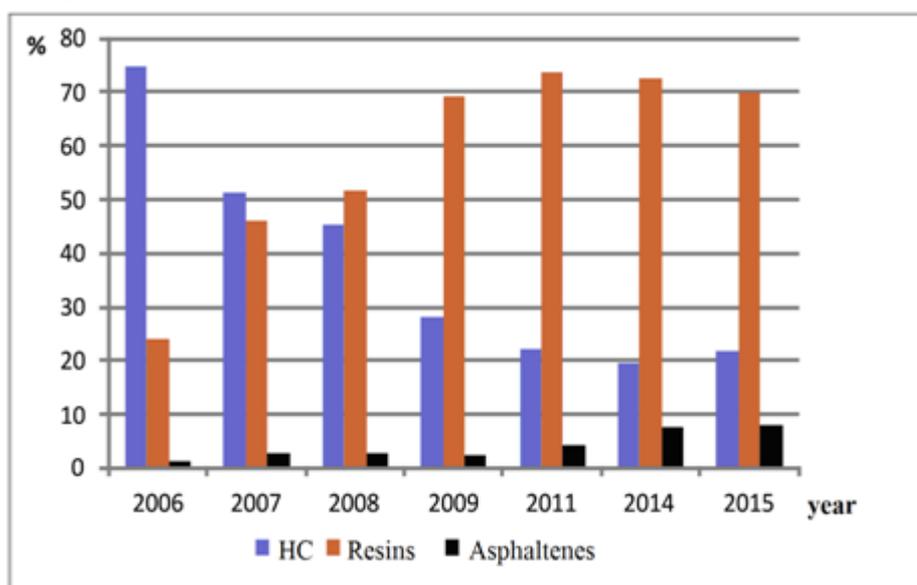


Figure 4. Changes in the content of hydrocarbon (HC) and asphaltic-resinous components in the composition of soil extracts by observation years.

According to chromatography-mass spectrometry data, oil transformation process was also characterized by changes in the hydrocarbon composition, namely, by redistribution of hydrocarbons both inside homologous rows and between different homologous rows (table 1, fig. 5).

Table 1. Composition of saturated hydrocarbons in extracts of soil samples

PARAMETERS	YEAR					
	2006	2007	2009	2011	2012	2015
Group composition of alkanes, % for \sum of identified alkanes:						
n-alkanes	52.39	50.85	30.03	30.51	26.81	none
isoprenoids	19.81	18.40	25.76	30.76	42.65	49.37
12- and 13-methylalkanes	12.03	14.52	16.37	12.92	19.28	10.61
$\sum n.c.-nC_{20}/\sum nC_{21-c.c.}$	1.23	1.33	1.17	1.26	0.69	none
max n-alkanes	$nC_{15,17}$	nC_{15}	nC_{17}	$nC_{17,18}$	nC_{25}	ND
isoprenoids/n-alkanes	0.38	0.36	0.86	1.01	1.59	ND
CPI	0.96	0.96	1.2	1.30	1.34	ND
iC_{19}/iC_{20}	0.80	0.76	0.71	0.69	0.61	0.41
iC_{19}/nC_{17}	0.97	0.96	1.87	2.10	4.50	ND
iC_{20}/nC_{18}	1.57	1.56	3.54	3.52	10.17	ND
$iC_{19}+iC_{20}/nC_{17}+nC_{18}$	1.23	1.23	2.58	2.76	6.88	ND

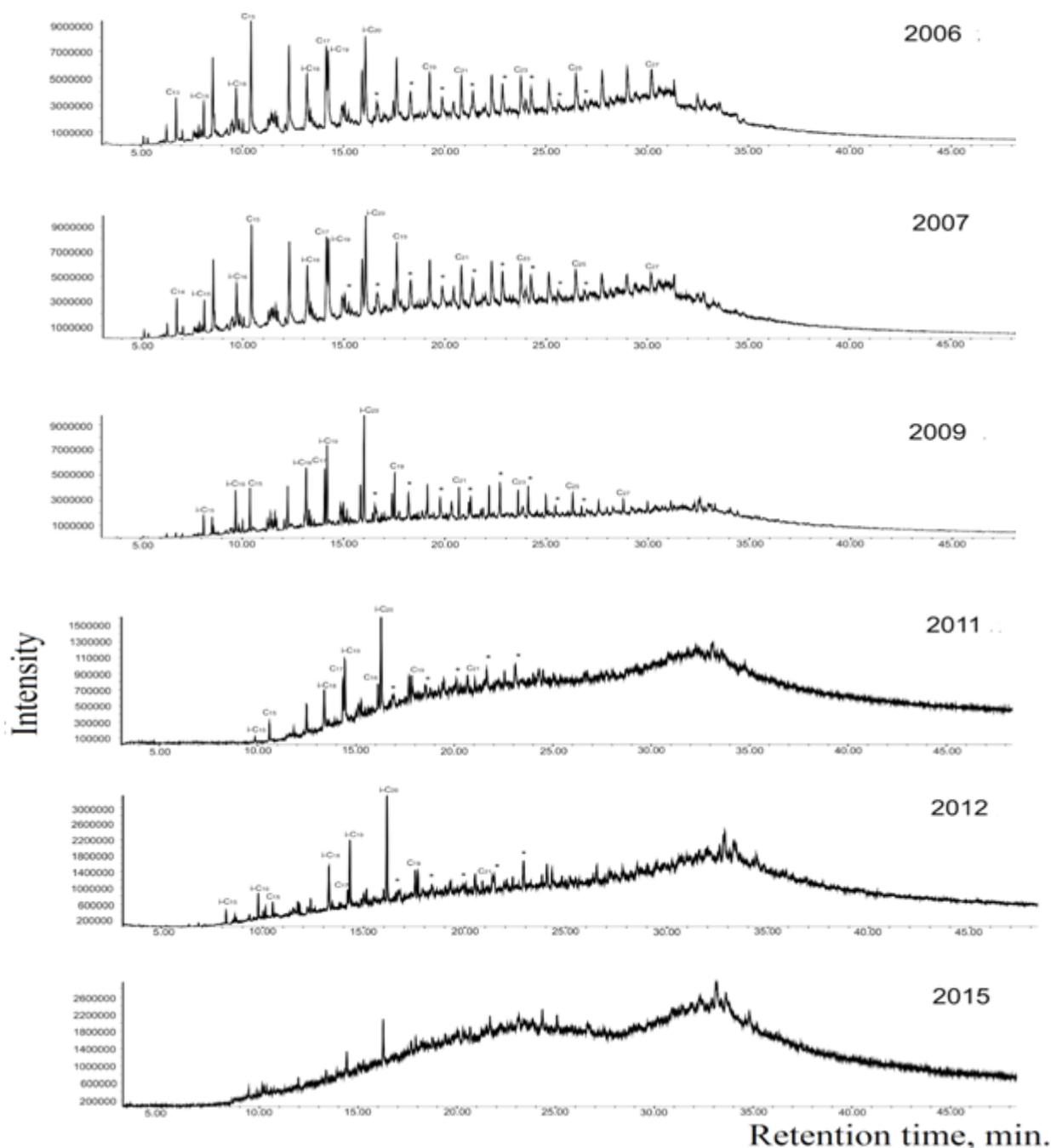


Figure 5. Chromatograms by total ion current (TIC) of saturated hydrocarbons in soil extracts.

$C_{13} \dots C_{27}$ – n-alkanes, $i-C_{15} \dots i-C_{20}$ – isoprenoids, * - 12- and 13-methylalkanes.

In the initial contaminated samples collected in 2006 and not yet exposed to transformation, prevailing saturated hydrocarbons were alkanes of regular structure among, which relatively low-molecular homologous compounds with distribution maximum of nC_{15} , nC_{17} prevailed (table 1, fig. 6). The ratio of n-alkanes with an odd number of carbons to n-alkanes with an even number (CPI) was close to one. Isoprenoids amounted to 18.40-19.81%. Phytane

($i-C_{20}$) dominated over pristane ($i-C_{19}$) and n-octadecane ($n-C_{18}$). The pristane/ nC_{17} ratio was close to one. This distribution of saturated hydrocarbons is typical for oils of the Vendian-Cambrian deposits of the Nepsko-Botuobinskaya oil and gas-bearing region, where the Talakanskoye oil-gas condensate field is located (Oil Geochemistry..., 2009). Oil from this field was the source of contamination.

In time, with oil transformation process in soil the amount of n-alkanes in saturated hydrocarbons decreased from 50.85-52.39% in 2006-2007 to 26.81-30.51% in 2009-2012 (table 1, fig. 5). The distribution maximum of n-alkanes shifted to a more high-molecular range: from nC_{15,17} in 2006-2009, to nC_{17,18} in 2011 and to nC₂₅ in 2012. CPI increased to 1.34. Isoprenoids content in the hydrocarbon composition increased from 20 to 49%. They became to dominate over n-alkanes (table 1, fig. 5). While the isoprenoids/n-alkanes ratio in the samples collected in 2006-2007 was 0.36-0.38 in 2011-2012, it was already 1.01-1.59. In the sample collected in 2015 n-alkanes were almost completely absent. Among isoprenoids, phytane dominates over pristane ($iC_{19}/iC_{20}=0.41-0.80$) that indicates the genetic nature of the oil contaminant and is typical for Talakan oils (Oil Geochemistry..., 2009).

All the samples were characterized by prevalence of the sum of pristane and phytane over the sum of n-heptadecane and n-octadecane ($iC_{19}+iC_{20}/nC_{17}+nC_{18}$). By most researchers, this ratio is considered as indicator of oil biodegradation degree (Oil Geochemistry..., 2009; Svarovskaya, 2004). The biodegradation processes are the more intense the higher values of this ratio.

A peculiar feature of all samples is the presence of hydrocarbons of row of 12- and 13-methylalkanes among isoalkanes. They are typical for oils of the Vendian-Cambrian deposits of Western Yakutia (Petrov, 1984; Oil Geochemistry..., 2009). Presence of these hydrocarbons is one of the indicators, which allows to identify oil contaminant of this type.

RESULTS AND DISCUSSION

According to some researchers, duration of oil degradation processes especially in cold regions can last decades (Glazovskaya, Pikovsky 1980; Oborin et al., 2008).

Many researchers differ three types of oil transformation in soils specifically physical and chemical destruction; microbiological transformation of oil hydrocarbons and the stage, when the most stable, high-molecular compounds remaining in soils and weakly

undergoing to microbial attack. As a result, in the soil there is a biocenosis different from background (Glazovskaya, Pikovsky, 1980; Kireeva, 1994; Oborin et al., 2008; Okolelova et al., 2015). The duration of each stage is a result of a group of factors, affecting the oil destruction rate: temperature, moisture content, oil composition and concentration, oxidation-reduction conditions, soil type, presence of native hydrocarbon oxidizing microorganisms, etc. Despite the common trend of oil transformation, these processes may vary to a certain degree, in different bioclimatic zones. For example, Pikovsky in his work (1993) ascertained that the first degradation stage lasted 1-1.5 years but this period may be significantly longer subject to the oil spill volume. In time, with oil transformation in the soil extracts content of hydrocarbon fractions decreases and the amount of asphaltic-resinous components increases. Resins and asphaltenes are the stable compounds almost inaccessible to microorganisms; their degradation is very slow; it may last a few decades.

Our data over ten years of monitoring of the area disturbed by the oil spill also allow to distinguish the main stages of oil transformation in soils. During the first year, a decrease in the oil content in soil was mainly due to physical and chemical destruction, gas removal, dissolution, UV destruction. Increase of activity of soil oil-degrading microorganisms normally shows itself 2-3 months after oil getting into soil (Solntseva, 1998). In the conditions of the cryolithozone, where the emergency oil spill occurred, microbiological activity of soil apparently remained low by that time (August-September) due to low above-zero day-time and below-zero night-time temperatures. During the second year observations, the oil contamination level remained almost the same. Only on the third year after the spill, significant decrease of oil content became an evidence, which was perhaps due to the activation of self-soil oil-degrading microorganisms.

In the following years, the oil contamination level varied both downwards and upwards. The decrease of the oil contamination level is due to transformation processes. An increase of

contamination level in the absence of new spills is apparently due to the ability of oil contamination to migrate. Made studies showed that oxidative breakdown of oil contamination was developing in subsurface soils (0-20 cm) over these years, due to which oil contamination acquired the asphaltic-resinous composition and thus slow-moving character. Most possibly, oil components migrated to the surface from underlying soil layers (where they had accumulated) up to the permafrost layer. Due to lower temperatures, lack of oxygen and UV, transformation processes at depth are very slow or are absent (Glazovskaya, Pikovsky 1980; Solntseva, 1998).

Thus, may assume that the main distinctive features self-remediation processes of soils in cryolithozone conditions are as follows:

- increase in activity of self-soil oil-degrading microorganisms not in 2-3 months, as is the case with moderate climate but only on the third year after the spill;
- preservation of oil's ability to migrate over a long period (in the case under study – 10 years) despite that contamination already acquired the asphaltic-resinous composition and slow-moving character in subsurface soils (0-20 cm). Apparently, this is due to annual getting new portions of components of oil with thawed and ground waters at seasonal thawing of soil. This may consider as additional source of oil contamination of soils.

CONCLUSIONS

The results of researches, of ten-year monitoring of the territory of the “Talakan-Vitim” oil pipeline contaminated at emergency oil spill in 2006, showed, that despite an overall tendency towards decrease contamination level from 91 g/kg to 45 g/kg in 2015, 10 years after the spill the contamination level was still very high. The activity of self-soil oil-degrading microorganisms increased only on the third year after spill, it indicated low rates of self-soil remediation in cryolithozone conditions. Study of the composition of soil extracts evidenced the occurrence of oil oxidative breakdown processes in subsurface soils (0-20 cm). Oil contamination acquired asphaltic-resinous composition and

steady character. If no new spills occur, an increase of the oil contamination level in individual observation years (2010, 2011, 2015) may indicate oil's ability to migrate in permafrost soils for a long time.

At seasonal thawing of soil oil components from a permafrost layer partially moved up to overlying soil layers, migrating with thawed and ground waters along a permafrost layer. Thus, oil contamination becomes chronic due to annual additions of oil components.

High level of residual oil contamination of soils indicates formation of abnormal hydrocarbon technogenic fields that testifies need for monitoring prolongation of the disturbed territories and also remediation works.

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