

## Effect of biodegradation processes on the composition and structure of asphaltenes in West Siberian oils

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**Abstract.** NMR spectroscopy in combination with elemental analysis was used to study asphaltenes in biodegraded oils from Cenomanian pools of West Siberia. The sampling depths vary from 680 to 1800 m, formation temperatures – from 40°C to 70°C. For comparison, we used the data on asphaltenes in non-biodegraded oils of different genotypes. Given that biodegraded oils are very heavy (density: 910-950 kg/m<sup>3</sup>), they are characterized by high boiling point temperatures (145-270°C). Due to the loss of hydrocarbon components, they have higher resin and asphaltene content (9-20%) compared to non-degraded samples. Elemental analysis of asphaltenes in biodegraded and unaltered oils of different genotypes revealed an increasing trend for oxygen content in the asphaltenes from biodegraded samples, which may result from the oxidation of structural blocks of asphaltenes during microbial oxidation. It was shown that the aromaticity of the moderately biodegraded terrestrial-aquatic Novoaganskaya samples tends to increase with a decrease in asphaltene saturation, suggesting that the redistribution of structural groups of asphaltenes may be caused by biodegradation processes. High saturation of asphaltenes in strongly biodegraded Gubkinskaya and Novoportovskaya oils, along with a high degree of substitution of aromatic compounds in asphaltenes in Gubkinskaya oils (terrestrial and aquatic-terrestrial genotype) can be attributed to the formation of asphaltenes during strong biodegradation of hydrocarbon components in these oils.

**Keywords:** asphaltenes, oil, biodegradation, West Siberia

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Establishing patterns of changes in the properties of oils in the hypergenesis zone since the middle of the last century is an important task of geochemical research, both for the development of the theory of naphthidogenesis, and for solving practical problems aimed at predicting the quality of hydrocarbon fluids. The active influence of hypergenesis is characteristic of shallow oil deposits with low reservoir temperatures, which are often located in the zone of infiltration of meteoric waters. Vassoevich and Amosov (1953) identified two zones of hypergenesis: the lower – cryptohypergenesis, which is characterized by anaerobic environments; upper idiohypergenesis associated with aerobic conditions. Even in the works (Uspensky, Radchenko, 1947; Vassoevich, Amosov, 1953) it was noted that with the active influence of hypergenesis from the bottom upwards along the section, the oil becomes heavier, more resinous, depleted of paraffins and enriched with naphthenic components. To date, it has been established that the most important

factor of hypergenesis, affecting the change in the group and component composition of oils, is their microbial oxidation (Roanova, Kuznetsov, 1974; Petrov, 1984; Kurbsky, 1987; Kashirtsev, 2003; Philippi, 1977; Peters et al, 2005; Mullins et al., 2007, etc.), which grows from cryptohypergenesis to the idiohypergenesis zone. During biodegradation, mainly hydrocarbons are utilized, with the rate of microbial oxidation decreasing in a certain sequence – from n-alkanes to acyclic isoprenanes and further to polycyclic naphthenes. On the basis of indicators on the hydrocarbon composition, it has been established (Goncharov, 1987; Kontorovich et al., 1991; Fursenko, Borisova, 2006; Peters et al, 1994, etc.) that in West Siberia biodegraded oils are characteristic of shallow Aptian-Albian-Cenomanian and, partly, Barremian pools, where thermo- (reservoir temperatures <70°C) and hydrodynamic conditions are favorable for microbial oxidation of hydrocarbon fluids. In recent years, studies have discussed the effect of biodegradation on the composition and structure of asphaltenes (Gordadze et al., 2015; Mullins et al., 2007; Silva et al., 2008; Meredith et al., 2008; Liao et al., 2009; Snowdon et al., 2016, etc.). The chemical and geochemical features of asphaltenes in West Siberian

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oils was the focus of many previous studies (Borisova, 2009; Golovko et al., 2010), however, variability of these parameters during biodegradation was not discussed.

The aim of the work is to study the effect of microbial oxidation on the composition and structure of asphaltenes in the oils of West Siberia. Asphaltenes isolated from biodegraded oils from Cenomanian pools (9 samples) were analyzed. For comparative analysis, we used the data on asphaltenes from non-degraded oils of different genotypes (96 samples).

In the designation of genetic types we used the following classification of West Siberian oils proposed by Kontorovich and Stasova (1964): aquatic ( $C_1$ ) – oils, that were generated by organic matter derived from marine deep-water sediments with hydrogen sulfide contamination, they have high to moderate density, high contents of sulfur, resins and asphaltenes, and are distributed, mainly, in Middle Ob' Region; terrestrial ( $A_1$ ) – oils that are genetically associated with organic matter derived from higher terrestrial plants, lakustrine plankton and benthos, they have light to medium density, and are low in sulfur, resins and asphaltenes, and have high concentrations of solid paraffins, these oils are localized in the northern and arctic regions of the West Siberia; mixed  $C_2$  and  $A_2$  oils replace successively aquatic crudes for terrestrial ones from the Middle Ob' Region to the north. Biodegraded oils, according to previous studies (Petrov, 1984; Goncharov, 1987; Kontorovich et al., 1991; Fursenko, Borisova, 2006; Peters et al., 1994, etc.), are usually very heavy, and have high contents of asphaltenes and resins; on chromatograms (gas/liquid chromatography) they are characterized by a distinct unresolved “naphthenic hump”. N-alkanes in these oils are either absent or present in very low concentrations.

Genetic typification of the studied oils, which was based on the data on physicochemical properties, isotopic composition and distribution of hydrocarbon biomarkers (pristane/phytane,  $C_{29}/C_{27}$  steranes,  $C_{35}/C_{34}$  homohopanes,  $C_{19}-C_{20}/C_{23}-C_{26}$  tricyclic terpanes, etc.) and their spatial localization was discussed in previous studies (Goncharov, 1987; Kontorovich et al., 1991; Peters et al., 1994). In the studied collection, 4 genetic groups of non-biodegraded oils (Fig. 1) are distinguished: predominantly aquatic (16 samples) and predominantly terrestrial (5 samples) genotype; and two groups of mixed genotypes – aquatic-terrestrial (31 samples) and terrestrial-aquatic (44 samples). Biodegraded oils have been identified by gas/liquid chromatography. These oils were collected from Cenomanian pools ( $K_{2c}$ ) from depths of 680-1800 m with reservoir temperatures  $< 70^\circ\text{C}$ . Such conditions are favorable for the microbial oxidation (Rozanov, Kuznetsov, 1974; Petrov, 1984; Goncharov, 1987; Kashirtsev, 2003, etc.). Severely biodegraded oils from the Gubkinskaya and Srednemosoyakhskaya prospects are characterized

by the absence of n-alkanes and acyclic isoprenanes, the presence of 25-norhopanes in the m/z 177 mass-fragmentograms. The remaining samples are only slightly biodegraded oils. Analysis of the distribution of polycyclic biomarkers (steranes and terpanes) shows that samples from the Novoportovskaya, Messoyakhskaya, Srednemosoyakhskaya, Gubkinskaya and Ereminskaya prospects can be attributed predominantly to the terrestrial genotype, whereas samples from the Novoaganskaya prospect are of terrestrial-aquatic genotype.

Biodegraded oils differ significantly from non-biodegraded oils by their physico-chemical properties and group composition (Table 1). Due to the loss of hydrocarbon components, they have higher contents of resins and asphaltenes (9-20%). These are very heavy oils (910-950 kg/m<sup>3</sup>). The biodegraded oils are characterized by a high bubble point (145-270°C). All these characteristics are consistent with our recent understanding of biodegraded oils (see above).

Unlike biodegraded oil samples of a mixed genotype (Novoaganskaya prospect), the biodegraded mostly terrestrial oils (Ereminskaya, Novoportovskaya, Messoyakhskaya and Srednemosoyakhskaya prospects) have lower sulfur, resin and asphaltene contents, while paraffins are either absent or present in trace amounts. The latter may be caused by a strong degree of biodegradation rather than by genetic reasons (Petrov, 1984; Kashirtsev, 2003; Peters et al., 2005, etc.).

### Asphaltene Analysis

The separation of asphaltenes was carried out with petroleum ether with a boiling point of 40-70°C in a ratio 1:40 (Kontorovich, 1973). The composition and structure of asphaltenes were studied by nuclear magnetic resonance spectroscopy (NMR) in combination with elemental analysis.

The high resolution NMR method of protons (PMR), together with data on the elemental composition (integral structural analysis) (Brown et al., 1960; Borisova, 2012), provides information on the distribution of hydrogen and carbon between various structural elements of complex organic compounds. The PMR spectra were studied on a Bruker spectrometer with an operating frequency of 200 MHz. According to the PMR and elemental analysis, the carbon content was calculated in various structural groups of asphaltenes: saturated ( $C_{\text{sat}}$ : in methylene, methine and methyl groups, distant from the aromatic ring and replacing hydrogen aromatic structures) and aromatic ( $C_{\text{ar}}$ : in condensed and peripheral positions). The carbon content in naphthenic structures ( $C_{\text{naph}}$ ) is not included in the 100% of the above groups, but can be quantified as percent of saturated structures. Determination of the mass proportion of carbon, hydrogen, sulfur and nitrogen was carried out on a Flash EA2000 CNHS analyzer. The physical and

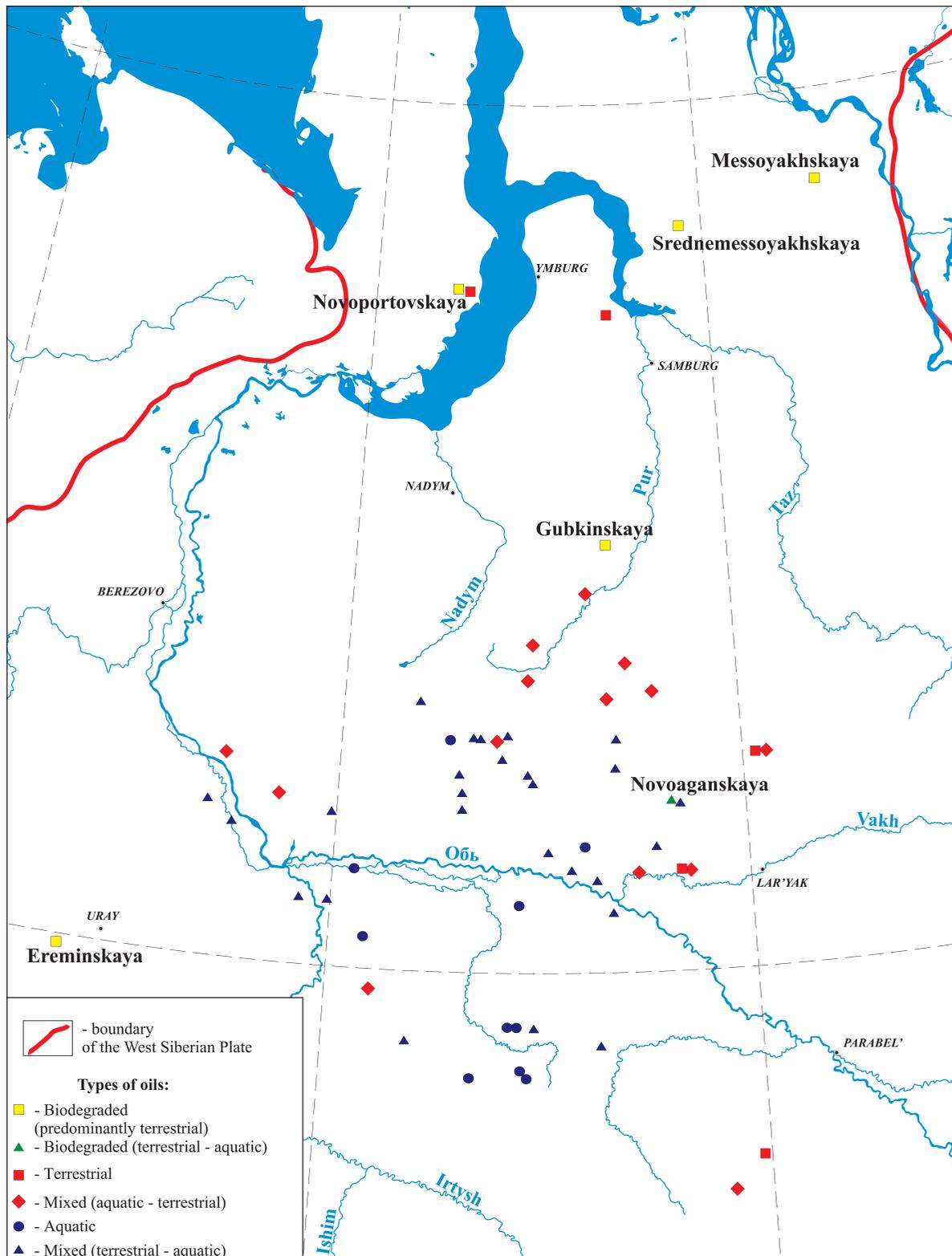


Fig. 1. Schematic map of oil sampling points

physicochemical methods of asphaltene analysis as well as the parameters of spectra acquisition and processing were discussed in detail in Borisova (2012).

**Discussion**

The studied samples are characterized by the elemental composition typical of asphaltenes (Table 2, Fig. 2a, b). The hydrogen content of asphaltenes in

biodegraded oils varies from 7.52 to 8.83%, and the carbon/hydrogen atomic ratio ((H/C)<sub>at</sub>) varies from 1.04 to 1.30. They also contain 0.71-1.87% sulfur and 0.30-7.05% oxygen.

The asphaltenes from predominantly terrestrial oils, both biodegraded and non-biodegraded, have lower hydrogen and sulfur contents, lower (H/C) at ratio compared to aquatic and terrestrial-aquatic

Prospect, well	Depth, m	Density, kg/ m <sup>3</sup>	S, %	Paraffins, %	Resins, %	Asphaltenes, %	Resins/Asphaltenes
<b>Biodegraded oils (terrestrial - aquatic genotype)</b>							
Novoaganskaya, 197	1734-1738	935.1	0.50	2.50	12.90	3.75	3.44
Novoaganskaya, 197	1574-1578	938.5	0.52	0.63	14.46	4.66	3.10
Novoaganskaya, 196	1302-1307	945.6	0.54	0.69	14.92	4.48	3.33
Novoaganskaya, 201	1204-1207	948.1	0.59	0.34	12.16	1.94	6.27
<b>Average values</b>		<b>941.8</b>	<b>0,54</b>	<b>1.04</b>	<b>13.61</b>	<b>3.71</b>	<b>4.04</b>
<b>Biodegraded oils (terrestrial and aquatic - terrestrial genotype)</b>							
Gubkinskaya, 642	1396-1398	914.3	0.37	1.78	7.63	0.43	17.74
Ereminskaya, 5	612-625	950.0	-	0.00	17.10	2.60	6.58
Messoyakhskaya, 31	897-899	953.1	0.23	0.01	11.40	1.49	7.65
Новопортовская, 46	888-897	910.0	-	0.00	8.70	0.40	21.75
Srednemesoyakhskaya, 25	887-894	953.9	0.24	0.05	9.10	1.97	4.62
<b>Average values</b>		<b>936,3</b>	<b>-</b>	<b>0.37</b>	<b>10.79</b>	<b>1.38</b>	<b>11.67</b>
<b>Genetic types of non-biodegraded oils in the studied collection (average values and scatter)</b>							
A <sub>1</sub> (terrestrial)		858/ (830-887)	0.27/ (0.09-0.56)	8.10/ (3.49-28.10)	5.44/ (2.53-9.83)	0.91/ (0.40-4.14)	4.24/ (1.17-8.47)
A <sub>2</sub> (mixed, aquatic - terrestrial)		851/ (755-918)	0.46/ (0.11-0.83)	3.76/ (2.60-5.68)	7.14/ (1.81-20.24)	1.72/ (0.12- 4.52)	5.24/ (1.51-15.08)
C <sub>1</sub> (aquatic)		887/ (829-925)	1.34/ (0.86-2.17)	3.64/ (2.39-4.59)	9.20/ (3.48-15.61)	4.69/ (0.28-11.16)	3.66/ (0.71-12.43)
C <sub>2</sub> (mixed, terrestrial - aquatic)		874/ (843-926)	0.88/ (0.26-1.77)	3.53/ (1.28-5.43)	12.33/ (4.34-27.58)	2.55/ (0.35-8.10)	7.00/ (1.22-27.66)

Table 1. Physical and chemical characteristics of oils

Prospect, well	Depth, m	Elemental composition, %					(H/C) <sub>at</sub>
		C	H	S	O	N+O+S	
<b>Biodegraded oils (terrestrial - aquatic genotype)</b>							
Novoaganskaya, 197	1734-1738	86.02	8.22	1.79	2.97	5.76	1.15
Novoaganskaya, 197	1574-1578	84.09	8.14	1.34	5.43	7.77	1.16
Novoaganskaya, 196	1302-1307	85.15	8.00	-	4.35	6.85	1.13
Novoaganskaya, 201	1204-1207	88.23	8.73	0.78	1.26	3.04	1.19
<b>Average values</b>		<b>85,87</b>	<b>8.27</b>	<b>1.30</b>	<b>3.50</b>	<b>5.86</b>	<b>1.16</b>
<b>Biodegraded oils (terrestrial and aquatic - terrestrial genotype)</b>							
Gubkinskaya, 642	1396-1398	82.35	8.89	0.71	7.05	8.76	1.30
Ereminskaya, 5	612-625	87.07	7.52	1.87	2.71	5.45	1.04
Messoyakhskaya, 31	897-899	86.15	8.06	1.02	4.77	5.79	1.12
Новопортовская, 46	888-897	85.38	8.23	0.91	4.46	6.39	1.16
Srednemesoyakhskaya, 25	887-894	83.55	7.54	1.26	6.65	8.91	1.08
<b>Average values</b>		<b>84,90</b>	<b>8.05</b>	<b>1.15</b>	<b>5.13</b>	<b>7.06</b>	<b>1.14</b>
<b>Genetic types of non-biodegraded oils in the studied collection (average values and scatter)</b>							
A <sub>1</sub> (terrestrial)		85.64/ (83.13-88.77)	7.57/ (7.11-7.90)	1.32/ (1.19-1.62)	4.74/ (1.88-6.67)	6.79/ (4.12-9.01)	1.06/ (0.96-1.14)
A <sub>2</sub> (mixed, aquatic - terrestrial)		86.57/ (82.76-88.64)	7.83/ (7.26-8.49)	1.64/ (0.48-2.69)	3.05/ (0.54-6.68)	5.60/ (3.19- 9.33)	1.09/ (1.01-1.18)
C <sub>1</sub> (aquatic)		85.80/ (80.03-87.97)	8.17/ (7.72-8.81)	2.80/ (2.49-3.41)	1.22/ (0.20-4.47)	6.82/ (3.69-13.86)	1.14/ (1.08-1.24)
C <sub>2</sub> (mixed, terrestrial - aquatic)		85.33/ (81.59-88.20)	8.08/ (7.51-8.77)	2.51/ (1.17-4.71)	3.39/ (0.47-6.95)	6.60/ (3.03-10.29)	1.13/ (1.05-1.21)

Table 2. Elemental composition of asphaltenes in oils

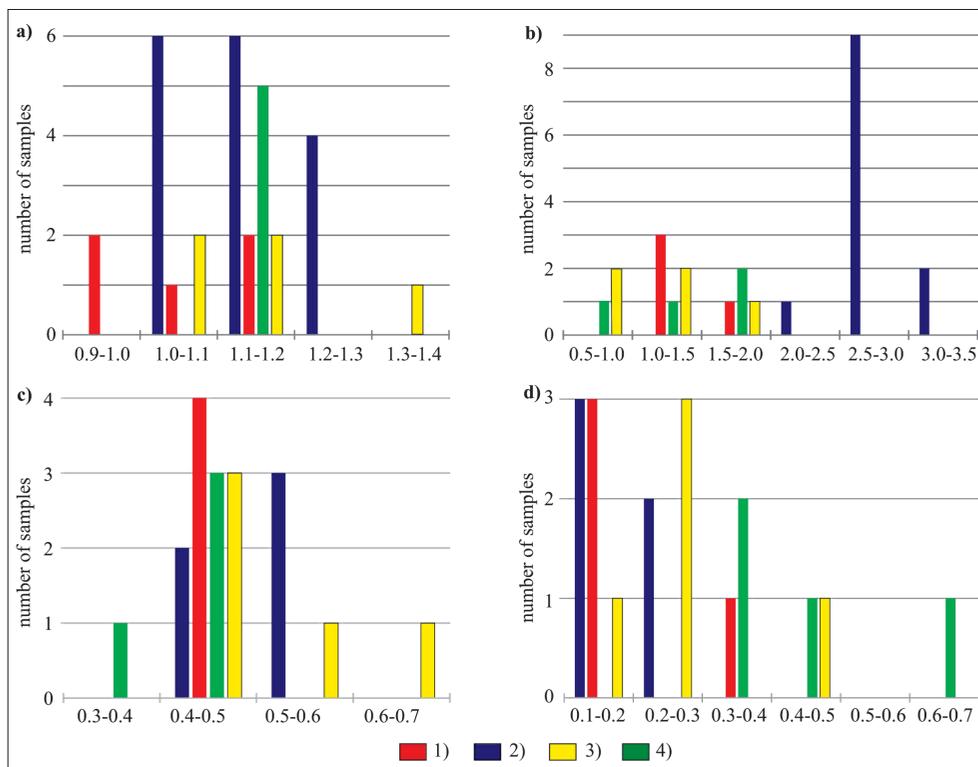


Fig. 2. Histograms of the distribution of elemental parameters: (a)  $(H/C)_{at}$ ; b) S, %) and group composition (c) saturation; d) naphthenic content) of the asphaltenes studied. 1) terrestrial genotype; 2) aquatic genotype; 3) biodegraded samples (predominantly terrestrial genotype); 4) biodegraded samples (terrestrial-aquatic genotype)

samples. However, a comparison of the  $(H/C)_{at}$  ratio by genetic groups shows that this indicator is higher for biodegraded samples. For example, the highest value of this ratio is observed in the asphaltene from severely biodegraded Gubkinskaya oil (1.30), which is classified to be of aquatic-terrestrial genotype, whereas the average value of this ratio in non-biodegraded oils of this group is 1.09 (Table 2). The  $(H/C)_{at}$  ratio varies from 1.16 for asphaltenes in moderately biodegraded Novoportovskaya oil to 1.13 for asphaltenes in non-biodegraded Novoportovskaya oil. It should be noted that asphaltenes in severely biodegraded Gubkinskaya (7.05%) and Srednemessoyakhskaya (6.65%) samples have high oxygen concentrations. Asphaltenes in the moderately biodegraded oils from the Novoaganskaya prospect have higher oxygen content (1.26-5.43%) than those in non-biodegraded oils (1.11%). Based on the results of the experiment on the microbial oxidation of oil from the Ashalchinskoe field, Filatov et al. (2017) showed that these parameters tend to increase in an experimentally biodegraded sample.

Let us consider the features of the structural-group composition of the studied asphaltenes obtained by the PMR method. The studied asphaltenes have almost similar saturation and aromaticity values (Table 3), and the average proportion of naphthenic structures in the composition of saturated ones does not exceed 0.3, except for biodegraded Novoaganskaya samples. The saturation of terrestrial non-degraded samples is lower compared

to aquatic ones, and their naphthenic content, on the contrary, is higher. Several asphaltenes in biodegraded oils of predominantly terrestrial genotype (Gubkinskaya and Novoportovskaya prospects) are characterized by the highest saturation (Table 3), which is significantly higher than that of asphaltenes in non-degraded terrestrial and aquatic samples. Thus, for example, the saturation of asphaltene in non-degraded Novoportovskaya oil is 0.49, and that of biodegraded Novoportovskaya oil is 0.63. At the same time, the naphthenic content of the biodegraded Gubkinskaya sample is comparable to that of the non-degraded terrestrial samples. This sample is also characterized by a high substitution value of aromatic structures and their low condensation. The naphthenic content of the asphaltenes in biodegraded Novoportovskaya oil is higher (0.47) than that of asphaltenes in the non-degraded sample (0.19), and the condensation of the aromatic structures of these asphaltenes is the lowest in the studied collection. The biodegraded terrestrial-aquatic Novoaganskaya samples are characterized by relatively low saturation and strong aromaticity compared to non-degraded aquatic samples. The condensation and substitution of the aromatic structures of the Novoaganskaya asphaltenes show the same variability in the corresponding genetic group of non-degraded samples. The studied asphaltenes of different oil types exhibit no distinct trend in the distribution of saturated structural blocks (methylene and methine groups located in chains and rings far

Prospect, well	Depth, m	Saturation	Aromaticity	Substitution	Condensation	Naphthenic content
<b>Biodegraded oils (terrestrial - aquatic genotype)</b>						
Novoaganskaya, 197	1734-1738	0.37	0.63	0.08	0.47	0.67
Novoaganskaya, 197	1574-1578	0.46	0.54	0.41	0.63	0.38
Novoaganskaya, 196	1302-1307	0.45	0.55	0.27	0.65	0.42
Novoaganskaya, 201	1204-1207	0.47	0.53	0.43	0.58	0.38
<b>Average values</b>		<b>0.44</b>	<b>0.56</b>	<b>0.30</b>	<b>0.58</b>	<b>0.46</b>
<b>Biodegraded oils (terrestrial and aquatic - terrestrial genotype)</b>						
Gubkinskaya, 642	1396-1398	0.60	0.40	0.79	0.51	0.20
Ereminskaya, 5	612-625	0.49	0.51	0.46	0.77	0.24
Messoyakhskaya, 31	897-899	0.45	0.55	0.41	0.64	0.21
Новопортовская, 46	888-897	0.63	0.37	0.50	0.11	0.47
Srednemessoyakhskaya, 25	887-894	0.48	0.52	0.56	0.59	0.22
<b>Average values</b>		<b>0.53</b>	<b>0.47</b>	<b>0.54</b>	<b>0.52</b>	<b>0.27</b>
<b>Genetic types of non-biodegraded oils in the studied collection (average values and scatter)</b>						
A <sub>1</sub> (terrestrial)		0.47/(0.45-0.49)	0.53/ (0.51-0.55)	0.52/ (0.47-0.61)	0.68/ (0.41-0.89)	0.23/ (0.18-0.35)
A <sub>2</sub> (mixed, aquatic - terrestrial)		0.45/(0.38-0.52)	0.55/ (0.48-0.62)	0.33/ (0.08-0.60)	0.66/ (0.41-0.94)	0.29/ (0.18-0.43)
C <sub>1</sub> (aquatic)		0.52/(0.46-0.58)	0.48/ (0.42-0.54)	0.58/ (0.49-0.65)	0.77/ (0.58-0.88)	0.20/ (0.17-0.23)
C <sub>2</sub> (mixed, terrestrial - aquatic)		0.51/(0.35-0.58)	0.49/ (0.42-0.60)	0.46/ (0.18-0.75)	0.54/ (0.21-0.95)	0.26/ (0.16-0.47)

Table 3. Parameters of the group composition of asphaltenes in oils (according to the PMR and elemental analysis). Saturation – the proportion of carbon in saturated structures; aromaticity – the proportion of aromatic carbon; substitution – the proportion of aromatic peripheral carbon in which hydrogen is replaced by saturated structures; condensation – the proportion of carbon in the condensation cores of aromatic structures; naphthenic content – the proportion of carbon in cycloaliphatic saturated structures

away from the aromatic cores ( $C\text{C}_3^{\beta\gamma}$  and  $C(\text{CH}_2+\text{CH})^{\beta\gamma}$ , respectively); substituents located in the  $\alpha$ -position to the aromatic rings ( $C^\alpha$ ).

## Conclusion

Analysis of the elemental composition of asphaltenes in biodegraded and non-biodegraded oils of different genotypes reveals an increase in the  $(\text{H}/\text{C})_{\text{at}}$  ratio and oxygen content of asphaltenes in biodegraded oils, which may be caused by the oxidation of various structural blocks of asphaltene complexes during microbial oxidation of crude oils.

A comparative analysis of the compositional and structural parameters of asphaltenes obtained by the PMR method reveals differences in biodegraded oils as a function of their genetic type. These data were used to identify at least two structural types of asphaltenes in the studied biodegraded oils from the shallow Cenomanian pools of West Siberia. Moderately biodegraded Novoaganskaya oils (terrestrial-aquatic genotype) differ from non-degraded ones of the same genotype by an increase in aromaticity and a decrease in the saturation of asphaltenes. Such changes in the group composition of asphaltenes may be indicative of the redistribution of structural groups of asphaltenes due to the microbial oxidation of oils. The high saturation

of asphaltenes in the severely biodegraded Gubkinskaya and Novoportovskaya oils and, at the same time, the high substitution of aromatic structures in the asphaltenes from Gubkinskaya oil may be due to the formation of new asphaltene complexes with the increasing degree of biodegradation of hydrocarbon components in these oils.

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