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СТРУКТУРНЫЕ ОСОБЕННОСТИ СМОЛ И АСФАЛЬТЕНОВ НЕФТЕЙ МЕСТОРОЖДЕНИЙ УДМУРТИИ

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Исследованы физико-химические свойства, групповой химический и структурно-групповой состав нефтей месторождений Карсовай и Гремиха, а также состав стабилизационного слоя промысловой водонефтяной эмульсии, образованной нефтью месторождения Гремиха. Выделены и изучены методами ИК Фурье спектроскопии, ^{13}C и ^{1}H ЯМР спектроскопии смолы и асфальтены из исследуемых нефтей и асфальтены из межфазного слоя ВНЭ, и выявлены особенности их элементного и структурно-группового состава. Установлено, что в составе нефти месторождения Гремиха в 3 раза выше содержание асфальтенов, а содержание смол и твердых парафинов, напротив, ниже. Данная нефть характеризуется высокой плотностью и высокой кинематической вязкостью, что позволяет отнести ее к битумозным нефтям. Структурно-групповой анализ асфальтенов и смол, выделенных стандартными методами из исследуемых нефтей, показал, что асфальтены нефти месторождения Гремиха близки по значению молекулярных масс к асфальтенам, выделенным из нефти месторождения Карсовай, но характеризуются более высокой долей конденсированных ароматических и нафтеновых структур. В составе смол исследуемых нефтей наблюдается обратный порядок. Высокая агрегативная устойчивость водонефтяной эмульсии, образованной нефтью месторождения Гремиха, объясняется количественным и качественным составом природных поверхностно-активных соединений, присутствующих в данной нефти. Анализ природных стабилизаторов, выделенных из межфазного слоя ВНЭ, показал значительное преобладание в его составе асфальтенов (92,6%), отличающихся более высокой молекулярной массой и более высокой долей конденсированных циклических структур, с преобладанием ароматических колец и со значительной долей, по сравнению с исходной нефтью, нафтеновых структур. Повышенное содержание гетероатомов (S, N и O) в составе молекул асфальтенов из стабилизационного слоя, также указывает на более высокие поверхностно-активные свойства этих соединений.

Ключевые слова: нефть, асфальтены, смолы, парафиновые углеводороды, структурно-групповой состав, водонефтяная эмульсия

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THE STRUCTURAL FEATURES OF RESINS AND ASPHALTENES OF CRUDE OILS FROM UDMURTIA OILFIELDS

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The physicochemical properties, group chemical and structural-group composition of oils from the Karsovay and Gremikha fields, as well as the composition of the stabilizing layer of an oil-in-water emulsion (W/O) formed by oil from the Gremikha field were investigated. Resins and asphaltenes were isolated from the studied oils and the interphase layer of water-oil emulsion and they were studied by FTIR spectroscopy, ^{1}H and ^{13}C NMR spectroscopy, and the features of their structure-group composition were revealed. It was determined, that content of asphaltenes is 3 times higher in oil from Gremikha field, while content of resins and wax is lower than thouse in oil from Karsovay field. Structural group analysis of asphaltenes and resins isolated from the studied oils by standard methods showed, that the asphaltenes of the Gremikha oil field are close in molecular weights to the asphaltenes extracted from the oil of the Karsovai oilfield, but are characterized by a higher contant of condensed aromatic and naphthenic structures. In the composition of the resins of the studied oils, the opposite pattern was observed. The quantitative and qualitative composition of natural surface-active compounds presented in the oil of the Gremikha field explains the high aggregate stability of the water-oil emulsion formed by this oil. Analysis of nature stabilizers of the W/O emulsion interphase layer showed a significant predominance in its composition of asphaltenes (92.6%), characterized by a higher molecular weight and a higher content of condensed cyclic structures, with a predominance of aromatic rings and with a significant content of naphthenic structures in comparison with the original oil. The increased content of heteroatoms (S, N and O) in the composition of asphaltene molecules from the stabilization layer also indicates the higher surface-active properties of these compounds.

Key words: oil, asphaltenes, resins, wax, structure-group composition, oil-water emulsion

INTRODUCTION

A general trend in the development of the oil industry both in Russia and in a number of other oil-producing countries is a gradual increase of heavy, high-viscosity oils with a high content of paraffinic hydrocarbons and resin-asphaltene substances (RAS). RAS show high surface-active properties, and have a significant effect on viscosity-temperature and structural-mechanical properties of oil and mostly determine its tendency to form stable oil-water emulsions (WOE) [1-5].

A notable feature of resin-asphaltene substances, are high molecular weights, presence of poly condensed aromatic structures [6-8] and high content of heteroatoms [9, 10]. These structural features of the SAR contribute to their tendency to association, aggregation and, as a consequence, the formation of deposits, which leads to complications in the processes of oil production, transportation and preparation [11-15]. At the same time, the RAS because of the presence of polar and non-polar fragments in one molecule have both hydrophilic and hydrophobic properties and exhibit high surface-active properties at the oil/water interface, which leads to the formation of stable oil-water emulsions. [4, 16, 17]. The stability of WOE is caused by the qualitative and quantitative composition of natural stabilizers concentrated in the interfacial layer [18-20].

The oilfields of the Republic of Udmurtia belong to one of the old oil-bearing regions and are characterized by a high content of high-viscosity heavy oils in the total balance of produced oil and the attendant complications in the processes of oil production, transportation and treatment of oil [21].

The purpose of this work was to study the structural-group composition of resins and asphaltenes of oils from two fields in Udmurtia, and asphaltenes involved in the formation of stabilization layers of the WOE.

EXPERIMENTAL PART

The objects of the study were pre-dehydrated oils from the Karsovay and Gremikha fields and a stable field water-oil emulsion from the Gremikha field, with a water phase content of 35% vol. Physicochemical properties and group composition of oils (Table 1) were determined by standard methods of analysis. The separation of asphaltenes from oil and natural emulsion stabilizers was carried out by the "cold" Golde method, resins and oils were separated by a chromatographic (column-adsorption) method, followed by the separation of wax from the oils with a mixture of acetone and toluene at -20 °C. Natural stabilizers of the field emulsion were isolated using washing solutions (tridecane, petroleum ether) and centrifugation (3000 rpm for 10 min) to destroy the adsorption layers and then concentrate them at the oil/water interface. The resulting jelly-like emulsion was boiled with distilled water until the stabilizer was isolated in the form of a suspension, followed by filtration [22].

The IR spectra were obtained on an instrument FTIR-spectrometer FSM 1201 in the range of wave numbers 4000-500 cm^{-1}. Using the optical densities of the characteristic bands, the spectral coefficients were calculated: aromatic Car = D_{1600}/D_{720}, aliphatic Cal = $(D_{720}+D_{1380})/D_{1600}$, branching Cb = D_{1380}/D_{720}. The molecular weight distribution of n-alkanes in oil was determined by gas chromatography on a Kristalluks-4000 chromatograph from a quartz capillary column (25 m; 0.24 mm) with a stationary phase SE-30. The analysis was performed under linear temperature programming (from 100 to 310 °C, heating rate 6 °C/min). The elemental composition of resins and asphaltenes was determined using an Elementar Vario micro cube CHNS analyzer. To determine the structural group composition of resins and asphaltenes, we used the ^1H and ^{13}C NMR spectra obtained on the Jeol "JNM-ECA 600" high-resolution spectrometer with an operating frequency of 600 MHz based on a permanent magnet. The calculation was carried out according to the method [23].

RESULTS AND DISCOUSSION

Oil from the Gremikha field has a higher density (923.0 kg/m^3) and kinematic viscosity (103.5 mm^2/s) and a large share of high-boiling fractions (76%). In the group composition of oils, we note, that oils are similar in resin content: 21.5% and 24.2%, noticeably different in the content of paraffins and asphaltenes.

Figure shows the molecular weight distribution (MWD) of n-alkanes in the composition of wax isolated from these oils. A similar monomodal character is observed in the MMD of n-alkanes with maximum points in the region of high-molecular hydrocarbons (C_{24}-C_{26}) and a slight predominance of the highest-molecular-weight C_{30}-C_{36} n-alkanes in the oil of the Gremikha field is noted.

Table 1

Physicochemical properties and group composition of the studied oils

***Таблица 1.* Физико-химические свойства и групповой состав исследуемых нефтей**

Property	Oilfield	
	Gremikha	Karsovay
Density at 20 °C kg/m^3	923.0	880.6
Kinematic viscosity at 20 °C, mm^2/s	103.5	30.6
Fractional composition, % vol. IBP до 100 °C до 200 °C до 300 °C	70 °C 3 11 24	61 °C 3 15 32
Sulfur content, %	2.99	1.66
Group composition, %		
Wax, %	2.2	3.7
Resins, %	21.5	24.2
Asphaltenes, %	11.7	4.3
R+A	33.2	28.5
R/C	0.54	0.18
(A+R)/W	15.1	7.7

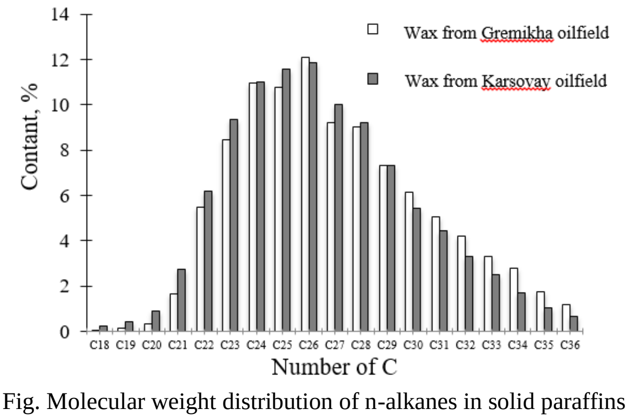

Fig. Molecular weight distribution of n-alkanes in solid paraffins isolated from the studied oils

Рис. Молекулярно-массовое распределение н-алканов в твердых парафинах, выделенных из исследуемых нефтей

More significant is the noticeable difference in the content of asphaltenes and in the ratio of high molecular weight components: (C+A)/P. In the oil of the Gremikha field, RAS content is more than 33.2% (wt.), which determines the high values of density and viscosity. The higher A/C parameter (0.54) observed for the Gremikha oil field, which explains the high tendency of the asphaltenes of this oil to associate, caused by the lower stabilizing effect of the resins. Table 3 shows the spectral coefficients characterizing the structural features of the structure of resins and asphaltenes in these oils.

It should be noted, that the studied oils differ in the content of asphaltenes and in the ratio of high molecular weight components: the total content of RAS significantly exceeds the content of wax, which is evident from the value of the coefficients (R+A)/P: 15.1 for oil from the Gremikha field and 7.7 for oil from the Karsovay field. The content of RAS in the oil of the Gremikha field is more than 33.2%, which leads to the high values of density and viscosity. Such a group composition of oil eliminates the influence of wax in the processes of structuring and emulsification of oil, the decisive factor is the interactions between RAS molecules. Probably, the higher value of the A/R parameter (0.54) observed for the Gremikha oil field explains the higher tendency of the asphaltenes of this oil to associate, caused by the lesser stabilizing effect of the resins, which leads to a higher viscosity of this oil. Table 2 shows the spectral coefficients characterizing the structural features of resins and asphaltenes structure in the studied oils.

Table 2

Spectral coefficients of the studied oils and their high molecular weight components

Таблица 2. **Спектральные коэффициенты исследуемых нефтей и их высокомолекулярных компонентов**

oilfield	Samples	Spectral coefficients		
		Car	Cal	Cb
Karsovay	oil	1.01	2.2	0.67
	resin	1.7	3.4	0.11
	asphaltenes	3.2	1.9	0.42
Gramihka	oil	1.5	3.7	0.16
	resin	1.9	1.6	0.31
	asphaltenes	7.1	1.2	0.22

The objects from Gremikha field are characterized by a higher degree of aromatic and a lower degree of aliphatic structures (resins and asphaltenes). Such a distribution of structural fragments, especially in the RAS, reduces their solubility in a hydrocarbons and, accordingly, leads to a decrease in their aggregate stability and the formation of deposits in oilfield equipment.

On the other hand, condensed aromatic structures exhibit high surface-active properties, which causes their high concentration at the oil/water interface and stabilization of oil-water emulsions. These results are confirmed by field data: the WOE formed by oil from the Gremikha field is a very stable and cannot be broken under the influence of temperature and sedimentation. From this point of view, it is of interest to study the composition of the interphase layer of the field WOE and the structure of its stabilizing components. According to the results of the analysis, it was found that the interphase layer of the field oil-water emulsion of the Gremikha field contains 0.34% of stabilizing components, represented mainly by asphaltenes (92.6%) – the main compounds that causes the formation of highly stable oil-water emulsions [17].

Table 3

Structural-group parameters of asphaltenes and resins isolated from the studied oils and the interphase layer of the field WOE from the Gremikha field

Таблица 3. **Структурно-групповые параметры асфальтенов и смол, выделенных из исследуемых нефтей и межфазного слоя промысловой ВНЭ с месторождения Гремиха**

Параметры	Karsovay oifield		Gremihka oifield		
	Resins	Asphaltenes	Resins	Asphaltenes	Asphaltenes from interphase layer
Molecular weigh. g/mol					
Mr	860	1971	905	2040	3364
Number of atoms in average molecule:					
C	61.6	137.6	61.9	140.9	230.7
H	80.6	159.5	88.6	154.8	262.7
H/C	1.3	1.2	1.4	1.1	1.1
N	0.61	2.5	0.67	3.3	5.6
S	1.0	2.7	1.4	3.3	5.6
O	1.1	2.4	1.2	2.7	5.2
Rings composition:					
K_r	9.9	21.1	7.1	24.2	41.4
K_a	5.9	20.1	4.4	22.6	29.7
K_n	4.0	1.02	2.8	1.6	11.7
The amount of carbon and hydrogen atoms of different types in the "average molecule"					
C_{ar}	24.83	75.57	22.59	83.75	117.9
C_{sat}	36.81	62.08	38.54	57.13	112.8
C_i	9.80	38.19	6.72	46.39	57.5
H_{ar}	7.33	20.94	7.06	21.45	32.2
H_{sat}	73.31	138.54	80.34	133.30	230.5
Characteristics of the aromatic substances:					
% Ca	40.3	54.9	36.9	59.5	51.1
h_a	0.09	0.13	0.08	0.14	0.12
N	7.7	16.4	8.8	15.9	28.3

Table 3 shows comparative data on the structure of resins and asphaltenes isolated from the studied oils and the structure of asphaltenes from the stabilization layer of the WOE. Based on the data of C, H, S, N, O-analysis and ^{1}H NMR-spectroscopy, the following quantitative structural parameters were calculated:

C, H, N, S, O – the number of atoms of the corresponding element in the molecule; K_r – the total number of rings, K_a, K_n – the number of aromatic and naphthenic rings in the "middle molecule"; C_{ar}, C_{sat}, C_i – the number of carbon atoms in aromatic and saturated structures, the number of nodal carbon atoms in the "average molecule"; H_{ar}, H_{sat} – the number of hydrogen atoms in aromatic and saturated structures in the "average molecule"; % C_a is the content of carbon atoms in aromatic structures; h_a is the part of aromatic protons; N is the number of alkyl substituents of the aromatic substanses in the "average molecule".

For the oils the general tendencies are: when passing from resins to asphaltenes the molecular weight, the number of heteroatoms in the average molecule, amount of cyclic and aromatic structures increase. The resins extracted from the oil of the Karsovay field have a higher content of aromatic rings in the average molecule, which indicates a more similarity with asphaltenes, and, as it is shown in table 1, the content of asphaltenes in this oil is 3 times less than those in oil from Gremikha field, it can be assumed that the oil of the Karsovai field contains asphaltenes in a more dispersed state, which, in particular, affects the viscosity of this oil. Asphaltenes from Gremikha oil have a high molecular weight (2040) and contain more aromatic structures in the "average" molecule, while the number of nodal carbon atoms Ci = 46.39 is also higher, which indicates a higher degree of condensation of aromatic fragments.

Comparison of the asphaltenes contained in the stabilization layer of the WOE and extracted directly from the oil shows that the most high-molecular compounds participate in the formation of the stabilization layer of the WOE: the molecular mass of the asphaltenes in the interphase layer is 1.5 times greater than their thouse of the average asphaltene molecule in oil. The average molecule of asphaltenes in the stabilization layer contains more amount of heteroatoms, which increases their polar properties, and at the same time, a significantly higher amount of saturated structures, in particular naphthenic rings.

CONCLUSION

The abnormal behavior of high-viscosity heavy oils in the processes of their production and preparation is associated with the presence of natural surfactants: resins and asphaltenes, and, as the study showed, their quantity in the composition of oil are of significant importance. The study of the composition of the WOEs interphase layer from the Gremikha deposit revealed that the main natural stabilizers of the emulsion are the highest molecular weight asphaltenes, which are characterized by a higher content of ring structures in the composition of the average molecule, both aromatic and naphthenic, and a higher content of heteroatoms.

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