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COMPARATIVE ASSESSMENT OF STRUCTURAL-MECHANICAL PROPERTIES OF HEAVY OILS OF TIMANO-PECHORSKAYA PROVINCE

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The physicochemical properties of heavy oils of Yaregskoe and Usinskoe deposits and the residues of atmospheric distillation of petroleum (fuel oil) recovered from them are presented. The group composition of oil and the residues of its atmospheric distillation (fuel oil) is determined. When using X-ray fluorescence energy dispersive spectrometer, the content of metals in the products is determined. A conclusion is drawn about the distribution of metals in the initial oil and fuel oil. On the basis of rheological characteristics, the type of liquids is determined in accordance with Newton's law, as well as the presence of an anomaly in the viscosity of the studied media at different temperatures. The energy values of the thixotropy of heavy oils of Usinskoe and Yaregskoe deposits, as well as the activation energy of the viscous flow of all media studied, are obtained. The phase transition of atmospheric residues at 60 °C is discovered. Dependences of the enthalpy and entropy of the viscous flow of the studied hydrocarbon media are obtained with an increase in temperature from 10 to 140 °C. The dependences of the oil molecules and atmospheric residues jumping frequency on viscosity are obtained for the first time.

***Key words*:** rheology, heavy oil, atmospheric residue, Usinsk deposit, Yaregskoe deposit, structural-mechanic properties, entropy, enthalpy of activation of viscous flow, frequency of molecule jump, hysteresis looping, thixotropy energy

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Introduction. A serious problem of the modern oil industry is the increase in the production of heavy oil, which contains a significant amount of impurities (asphaltenes, sulfur, metals), as well as the depth of its processing, since direct distillation of oil produces a heavier residue than light distillates, which reduces the value of such hydrocarbon feed [2, 4].

The main problems in application of heavy oil arise due to its low mobility both in formation conditions and on the surface. Its extraction and transportation to oil refineries are associated with great technological difficulties and material costs.

For the correct selection of technological regimes of oil production, transportation and processing, it is necessary to know not only the physicochemical properties of the liquid being studied, but also its rheological properties. In connection with this, the necessary stage in the implementation of a particular technological process is the carrying out of special rheological tests of these liquids, which will allow to predict their rheological properties, and also to choose effective methods for regulating these properties [6, 9].

Methodology and research methods. In order to compare the structural and mechanical properties of heavy oils, as well as identification of phase changes in oil and fuel oil residue, the object of the study was the heavy oils of the Timano-Pechorskaya province (Usinsk and Yaregsky deposits), as well as atmospheric residues obtained by direct distillation of these oils (Table 1).

The concentration of metals contained in the test samples was determined using an X-ray fluorescence energy dispersive spectrometer Elipson 3 (Table 1). It has been found out that the metals contained in the oil from Usinsk deposit almost completely turn into fuel oil residue, while for the oil from Yaregskoe deposit only vanadium, iron and nickel are concentrated in the atmospheric residue, and the aluminum and silicon content remains the same as in the original oil. On the basis of this, it can be assumed, that the distribution of the latter to the light fractions of oil during its atmospheric separation. The rather high content of the abovementioned metals in heavy oils allows us to consider these systems as an additional hydrocarbon source of raw materials for the recovery of valuable metals.

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Table 1

Physical-chemical properties of heavy oils from Yaregskoe and Usinskoe deposits and their atmospheric residues (fuel oils)

Indicator	Yaregskaya oil	Atmospheric residue of Yaregskaya oil	Usinskaya oil	Atmospheric residue of Usinskaya oil
Density at 20 °C, kg/m^3	939.8	964.0	900.0	954.0
°API	19	15	26	17
Kinematic viscosity, mm^2/s:				
40 °C	562.18	–	48.65	–
80 °C	–	150.53	–	548.94
Pour point, °C	–18	–10	–28	14
Sulfur content, % by weight	1.232	1.251	0.971	1.490
Aluminum content, % by weight	0.0070	0.0070	0.0056	0.0075
Silicon content, % by weight	0.0070	0.0070	0.0063	0.0071
Vanadium content, % by weight	0.0160	0.0210	0.0092	0.0175
Iron content, % by weight	0.0047	0.0060	–	0.0024
Nickel content, % by weight	0.0047	0.0063	0.0046	0.0085
Diesel fraction content 220-330 °C, % by weight	29	–	21	–
High boiling fraction (fuel oil) content, boiling away at temperature above 330 °C, % by weight	71	–	70	–
Saturated hydrocarbons content, % by weight	16	15	22	20
Linear paraffins content, % by weight	3.78	–	3.14	–
Aromatic hydrocarbons content, % by weight	40	38	21	24
Tar content, % by weight	27	27	39	30
Asphaltenes content, % by weight	17	20	18	26

To study the influence of fractional and hydrocarbon composition of heavy oil before and after rectification under atmospheric pressure on rheological properties, we have conducted the group analysis of test samples using the following procedure (Table 1). A sample of oil (fuel oil) was dissolved by a 40-fold volume of hexane, after that it was set aside to stand for 16 hours in a dark place in order to precipitate the asphaltenes. After filtration, the obtained deasphalted product was separated into saturated compounds, aromatic compounds and tarss using hexane, benzene and ethyl alcohol. It has been discovered that for Yaregskaya oil the tar and asphaltene content in fuel oil increases as a result of the separation of light fractions, while the content of saturated and aromatic compounds in it decreases, whereas in the fuel oil of Usinsk deposit the content of aromatic hydrocarbons and asphaltenes increases as a result of toppping of the light fractions.

Research results and their interpretation. As it is known [10], the formation of supramolecular structures in oil dispersed systems with a significant content of resinous-asphaltene substances (RAS) occurs on the basis of an asphaltene core, which is a layered pack-like associates of polyaromatic structures and a solvate shell consisting of molecules of resinous-oil components. With a gradual increase in the concentration of RAS in highly viscous oils, sharp changes in their rheological properties can be observed, which corresponds to the critical state of the system when the critical concentration is reached. In practical terms, oil in this state loses fluidity, as a result of which the transportation processes of such hydrocarbons become more complicated.

Creation of effective methods for regulating the rheological properties of heavy highly viscous oils is impossible without studying the structural transformations and quantitative evaluation of in-

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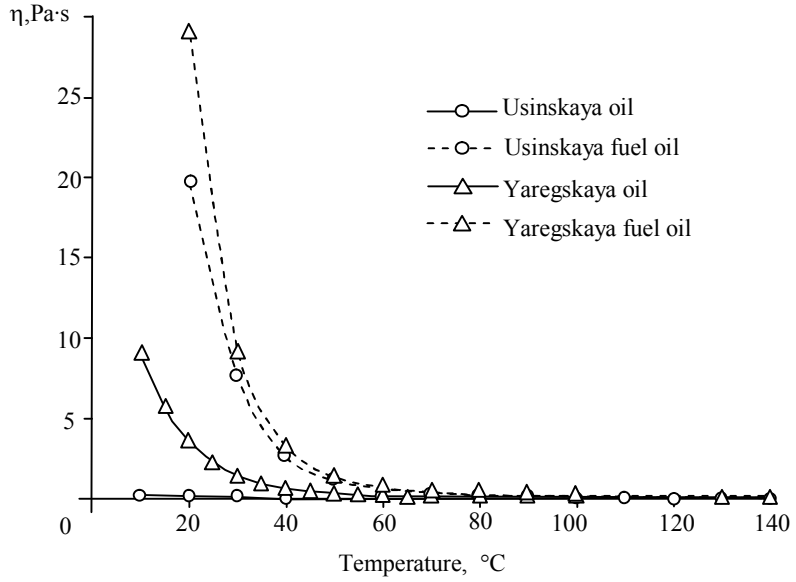


Fig.1. Dependency of dynamic vicsousity of oil from Yaregskoe and Usinskoe deposits and atmospheric residues from temperature

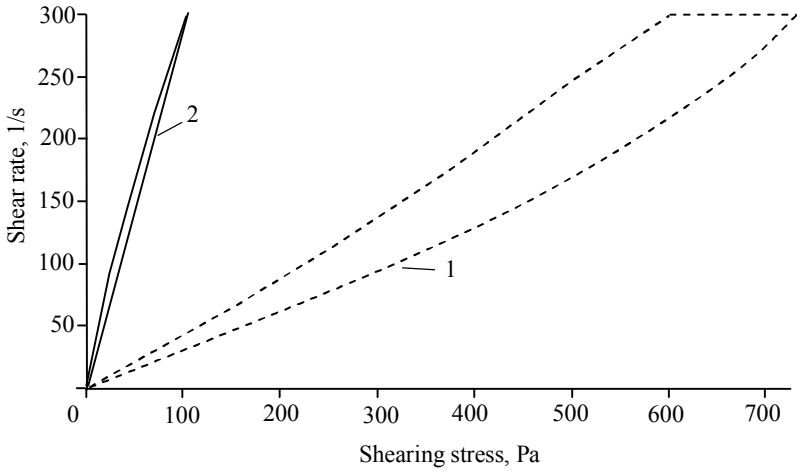


Fig.2. Hysteresis loops of oils from Yaregskoe and Usinskoe deposits
1 – Yaregskaya oil; 2 – Usinskaya oil

termolecular interactions in them [5, 13]. Such investigations can be carried out on the basis of the flow activation theory of Ya. I. Frenkel, H. Eyring [11, 15] and the results of experimental rheological studies of these oils.

A fluid, which properties obey Newton's law and can be described by an equation, are called Newtonian fluid:

$$\tau = \eta\gamma, \qquad (1)$$

where τ – shearing stress; γ – shear rate.

If shearing stress is not proportional to shear rate, then such fluid is called non-Newtonian fluid and a relation τ/γ is an effective (apparent) viscosity.

The experiments of studying structural and mechanic properties of oils and resulting from them atmospheric residues have been conducted according to the following procedure. The shear rate during dynamic tests gradually increased up to the value of γ_{max} (in seconds to the power of minus one degree) over e period of 300 s (forward move), then it was kept constant at the achieved value during 300 s (expectation of full destruction of internal structure of oil), and then the shear rate gradually decreased to a zero in 300 s (return move).

When studying the dependence of the viscosity of heavy oils and atmospheric residues from the temperature (Fig.1), a sharp decrease in viscosity was observed in a narrow temperature range (from 10 to 50 °C). It should be noted that there is a clear difference in the viscosity of the Yaregskoe and Usinskoe oil deposits at low temperatures, while the viscosity of fuel oil is similar in its indicators at temperatures above 30 °C, which is a consequence of the concentration of RAS in atmospheric residues of the Uinskaya and Yaregskaya oils, having a coagulation structure, resulting from direct distillation of oil under atmospheric pressure and separation of its light fractions.

When studying the rheological characteristics of a liquid, an important property is thixotropy, i.e. the ability of dispersed systems to reversibly liquefy under sufficiently intense mechanical action and solidify when staying at rest. Thixotropic media are the media whose structure deforms at a constant shear rate, which gradually leads to a decrease in the effective viscosity. Thixotropy is a reversible process, and after the removal of the loads, the structure of the liquid is gradually restored [3, 8]. The thixotropy energy is characterized by a hysteresis loop formed when the line of forward and return move does not coincide on

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the graph of the dependence of the shear rate on the shear stress. The area of the hysteresis loop, enclosed within a single measurement cycle, characterizes the amount of mechanical energy necessary to destroy thixotropic bonds per oil volume unit, so the larger the area of the «hysteresis loop», the more inclined the oil is to formation of structures under given temperature conditions.

As a result of laboratory studies, characteristic hysteresis loops for the Yaregskoe and Usinskoye oil deposits were obtained (Fig.2). The fact that the forward move does not repeat the line of the return move, indicates a thixotropic structure of the studied samples. In spite of the fact that both oils have a coagulation structure, 245 times more energy is needed to destroy the thixotropic ties of Yaregskaya oil than for Usinskaya one.

Thixotropic energy of heavy oils from Timano-Pechorkaya province:

Indicator	Yaregskaya oil	Usinskaya oil
The hysteresis loop area, Pa/s	320859.4	1307.76
Thixotropy energy, J/m^3	$711.01 \cdot 10^4$	$2.91 \cdot 10^4$

According to studies made by G.I.Fuks the decrease of viscosity with increase of shear rate gradient contradicts the Newton's law and is called viscosity anomality [3, 12].

When studying the dependecny of oils and atmoshperic residues vicosity from shea rate (Fig.3, 4) there was discovered an anomality of Yarhskaya and Usinskaya oils viscosity and their atmospheric residues at the whole range of investigated temperatures. Based on the behavior of the dependence of the samples under study, it can be concluded that they are viscoplastic liquids having a yield point, i.e. the limiting voltage below which the sample behaves as a solid. It should be noted that the dependency of shear yield point from temperature has a power-law form, which is confirmed by correlation coefficients from 0.9844 to 0.9987 (Fig.5). The structure of Yaregskaya fuel oil is more durable among all studied samples, that is indicated by high value of shear yield point at 20 °C.

When considering a viscous flow as a process whose velocity is determined by the energy necessary to overcome a potential barrier, the best results can be achieved by the joint application of the theory of absolute velocities and the statistical theory of a fluid based on the so-called free volume model [7, 8]. According to this theory, each molecule of a liquid is considered to be localized in a potential energy pit, i.e. in the region of the minimum of

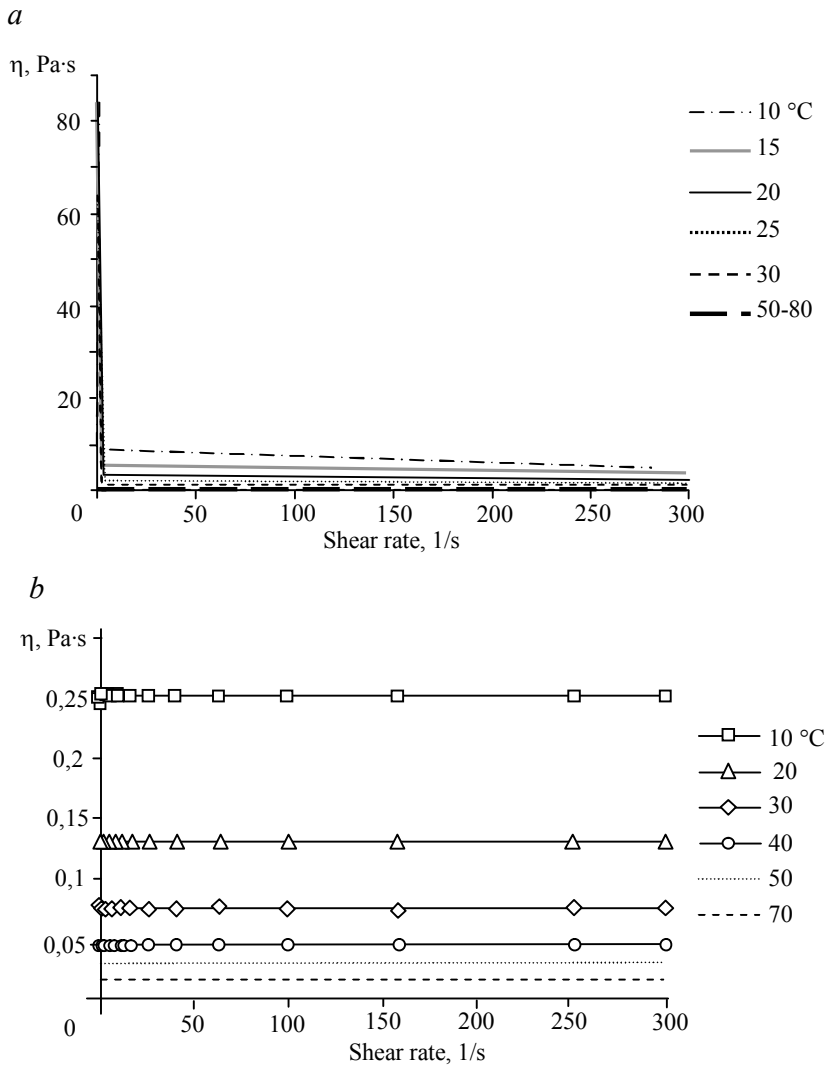


Fig.3. Dependency of viscosity from shear rate of Yaregskaya (*a*) and Usinskaya (*b*) oils

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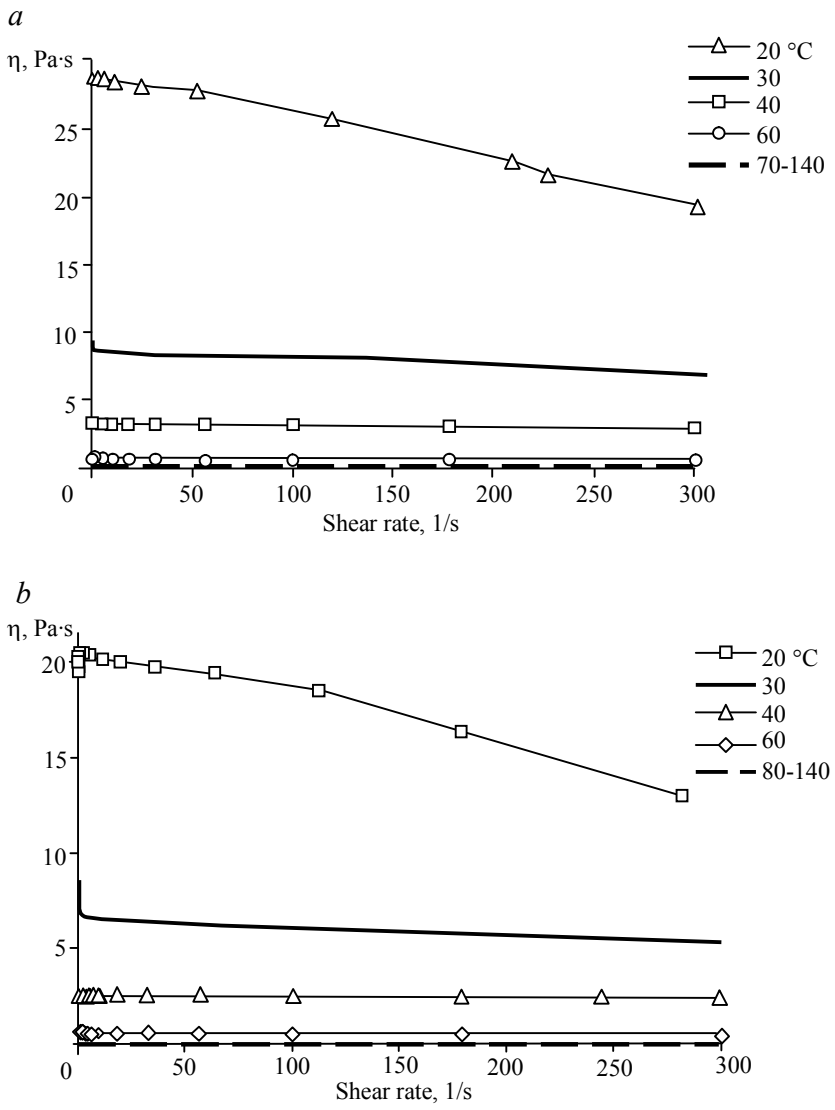


Fig.4. Dependency of viscosity from shear rate of atmospheric residue of Yaregskaya (*a*) and Usinskaya (*b*) oils

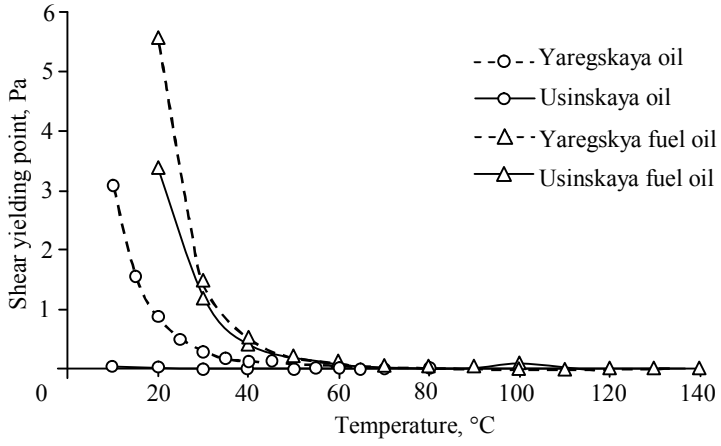


Fig.5. Dependency of yeild point from temperature

the potential energy, which is determined by the interaction of a given molecule with neighboring ones. This interaction leads to the establishment of short-range ordering, and long-range ordering almost does not exist [12]. According to H.Eyring theory the flow of liquids is accomplished by individual molecules jumping to a neighboring place, if it is free. These jumps always occur in a liquid and in the absence of flow only under the action of fluctuations in thermal energy. The presence of shear stress in the liquid during its flow makes it more likely that the molecules jump in the direction of the acting voltage. The probability of jumps is the higher, the greater is the thermal energy reserve in the system, i.e. higher temperature, and the weaker intermolecular interactions [11].

The frequency of molecular jumping is determined from a relation

$$J = \frac{1}{\tau}, \qquad (2)$$

where

$$\tau = \tau_0 e^{-E_a / RT} \qquad (3)$$

– residence time (Frenkel formula); E_a – activation energy, which defines the energy which «kinetic» (structural) unit has to receive as a result of thermal energy fluctuation to detach from its neighbors; characterizes the strength of bonds in associative complexes in each structural state of oil system for a given temperature value; R – gas constant; $\tau_0 = 10^{-13}$; T – temperature.

The patterns that determine the probability of jumping determine the regularities of the viscosity. By analogy with formula (3), we can write

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$$\eta = \eta_0 e^{E_a / RT}, \quad (4)$$

where η – dynamic viscosity; η_0 – pre-exponent of dynamic viscosity.

Taking logarithm of formula (2), we have

$$\ln \eta = \ln \eta_0 + \frac{E_a}{R}\frac{1}{T}. \quad (5)$$

As the temperature is raised, the value of E_a decreases, which leads to a gradual breakdown of supramolecular structures. Thus, the determination of the activation energy as a function of temperature gives an idea of the structural changes that are taking place in the substance.

It is known that the activation energy is equal to the work that must be spent to move the particles of the liquid, and is related to the energy of intermolecular interaction [8]. We have made the assumption that the free activation energy consists of two components:

$$\Delta G = \Delta G_0 + \Delta G_a, \quad (6)$$

where ΔG_0 – free energy associated with the process of momentum transfer in a gas; ΔG_a – the energy of hole formation in a liquid as a result of the displacement of structural elements.

According to the Frenkel-Eyring expression the change in viscosity is determined by the free energy for activation of viscous flow ΔG_a (5):

$$\eta = e^{\Delta G / RT} = e^{\Delta G_0 / RT} e^{\Delta G_a / RT} = \sigma_0 e^{\Delta G_a / RT}, \quad (7)$$

where $\sigma_0 = e^{\Delta G_0 / RT}$ – viscosity of a substance in a state of gas, Pa·s; according to Frost's formula

$$\sigma_0 = T(6{,}6 - 2{,}25 \lg M) \cdot 10^{-8}, \quad (8)$$

M – the average molecular mass of the substance at temperature T.

The free activation energy of the viscous flow is determined by the Gibbs equation:

$$\Delta G_a = E_a - T\Delta S, \quad (9)$$

where ΔS – activation entropy of viscos flow, J/(mol·K).

From relations (5) and (7) it follows:

$$\Delta S = (\ln \sigma_0 - \ln \eta_0) R. \quad (10)$$

The enthalpy of activation of the viscous flow is determined from the relation (9):

$$\Delta H = E_a. \quad (11)$$

The enthalpy and entropy energy values for activation of the viscous flow for multicomponent media are determined as the average over all components. Let us consider these averaged values.

When constructing the dependence of the logarithm of viscosity on the reciprocal temperature (Fig.6), it was found that the curve obtained for the Yaregsky and Usinskaya oils has a practically linear form. This is also confirmed by correlation coefficients, equal to 0.9949 and 0.9962, and perhaps, is determined by low content of solid par-

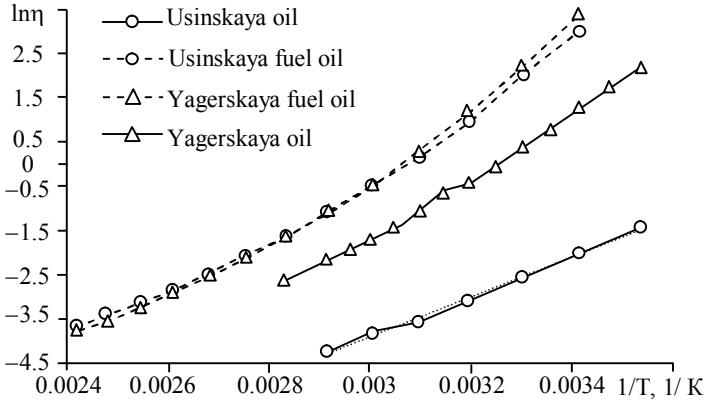


Fig.6. Dependency of Yaregskaya oil and atmospheric residues dynamic viscosity logarithm from reciprocal temperature

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affins in studied hydrocarbons, and values obtained for atmospheric residues of these oils are well approximated by two straight lines with an intersection point corresponding to 60 °C. On the basis of the obtained results, it is possible to assume a change in the structure of the atmospheric residues at a given temperature, as well as the precipitation of solid paraffins, i.e. at 60 °C, a phase transition of the media is observed. Thus, subsequent calculations for fuel oil should be carried out for two sections separately – before and after the temperature of the phase transition. As a result of the calculations, the activation energies of the viscous flow for oils and their fuel oils were obtained (Table 2).

Table 2

The activation energy of viscous flow

Indicator	Yaregskoe deposit			Usinskoe deposit		
	Oil	Fuel oil (20-60 °C)	Fuel oil (60-140 °C)	Oil	Fuel oil (20-60 °C)	Fuel oil (60-140 °C)
Activation energy, kJ/mol	57.92	77.31	47.95	37.84	70.41	44.74

It should be noted that the temperature of 60 °C corresponds to the melting point of n-alkane $C_{27}H_{56}$. It can be assumed that solid paraffins are concentrated in the high-boiling fraction of oil (fuel oil) and when this temperature is reached, they plate out, forming a phase transition of the system.

The concept of «activation energy» was first introduced by Arrhenius, who tried to explain the temperature dependence of the rate of chemical reactions. He put forward the idea of the existence of active molecules that are in equilibrium with the original (inactive) molecules. The physical meaning of the activation energy of a viscous flow corresponds to the energy of the transition of liquid atoms from the initial (equilibrium) to the intermediate (activated) state, from which they then transfer to a new equilibrium position [11].

To estimate structural transformations and intermolecular interaction in disperse systems, free energy, enthalpy and entropy of viscous flow activation are more often used. The applied shear stress τ can cause the destruction of these structures and the reorientation of macromolecules, which is accompanied by a change in their conformations. These processes must have different effects on the enthalpy and entropy of viscous flow activation, while the enthalpy of activation should reflect the strength of the structure, and the activation entropy – the degree of its ordering.

The activation energy of the viscous flow of Yaregskaya oil is 1.5 times higher than the activation energy of Usinskaya oil (57.92 and 37.84, respectively) (Table 2), which suggests stronger links of the structure in the Yaregskaya oil, when for atmospheric residues, this indicator is practically the same. Table 2 shows a sharp decrease in the activation energy of the viscous flow of atmospheric residues after a phase transition (almost 1.5 times), which is a consequence of the destruction of the oil structure and the decrease in the strength of the bonds as a result of heating. The energy of activation of the viscous oil flow is much higher than the oil enthalpy, on the basis of which it can be concluded that the bonds in the fuel oil harden to the phase transition temperature in comparison with the strength of the bonds in the original oils, which indicates the formation of a stronger structure in fuel oil as a result of atmospheric distillation and topping of its light diesel fractions.

The dependence of enthalpy and entropy of viscous flow activation on temperature was determined from formulas (9), (10) (Fig.7). It should be noted that for oil, the obtained values have a linear form, while for fuel oils at the temperature of the phase transition significant changes occur. The strength of the bonds of the studied systems decreases with increasing temperature, and the rate of decrease in the strength of the Yaregskaya oil bonds is higher than that of the Usinskaya oil. For atmospheric residues, the rate of decrease in bond strength decreases after heating above the phase transition temperature.

The entropy, being a function of the thermodynamic probability of a system, is related to the mutual arrangement of its elements, and changes in entropy reflect changes in the arrangement of these ele-

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ments, i.e. structure of a body. The entropy of viscous flow activation is the difference between the entropy of the activated and the initial state, and positive values of this value mean that the particles, molecules or other elements of the structure in the intermediate (active) state are less ordered than in the original state. This can occur if the flow is associated with the destruction of the structure. If during a flow the orientation processes dominate and are accompanied by the straightening of macromolecules and the formation of new ordered structures, the entropy of the activated state must be less than the initial state, hence the entropy of the activation of the viscous flow must be negative [13].

Since the obtained values of the studied systems are positive, a structure is destroyed for all media, which increases insignificantly with increasing temperature.

However, it should be noted that the structure of the Usinskaya oil and its fuel oil is more ordered than the structure of the Yaregskaya oil and its atmospheric residue, respectively. The entropy of activation of viscous fuel oil flow after the phase transition temperature decreases, which is a consequence of the ordering of the structure after the precipitation of solid paraffins from the system.

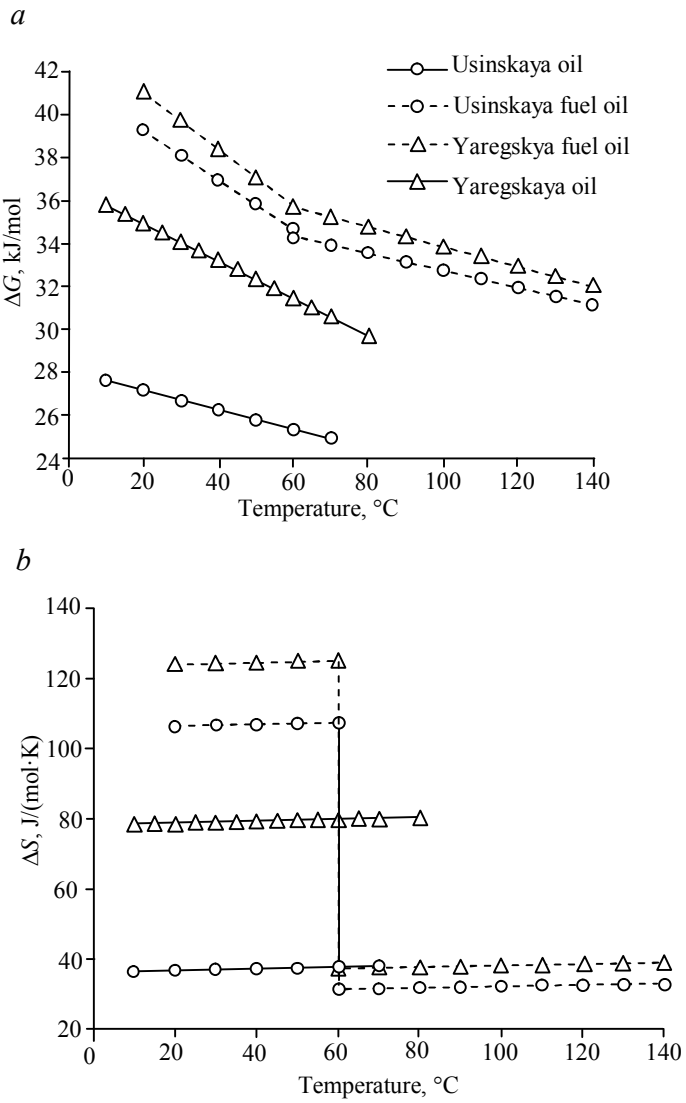


Fig.7. Dependencies of free energy activation (*a*) and entropy activation (*b*) of viscos flow of Yaregskaya and Usinskaya oils and atmospheric residues from temperature

High values of the energy of activation of oil and their rapid decrease with increasing temperature [1] were explained by the fact that in order to achieve the activated state of the liquid in the viscous flow, in addition to the work required for the formation of a "hole," it is necessary to expend energy for breaking strong intermolecular bonds.

The change in the free activation energy and the entropy of the viscous flow activation with temperature is associated with the flow mechanism itself, which is simultaneously occurring processes of destruction of the system structure and the orientation of macromolecules and elements of the destroyed structures.

Based on the research results and formula (2), the dependence of jumping frequency on viscosity for Yaregskoe and Usinskoye oil fields was determined:

$$J = 1589.2\,\mu^{-0.995}; \tag{12}$$

$$J = 165392\,\mu^{-1.018}, \tag{13}$$

and their atmospheric residues before and after the phase transition temperature, respectively:

$$J = 4.4014 + 7\mu^{-0.998}; \tag{14}$$

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$$J = 28.368\,\mu^{-1.005}; \quad (15)$$

$$J = 168056\,\mu^{-1.011}; \quad (16)$$

$$J = 348949\,\mu^{-1.012}. \quad (17)$$

It is determined that the dependence has a power-law character, which is confirmed by the correlation coefficients 0.9949; 0.9978; 0.9939 and 0.9964; 0.9901 and 0.9959, respectively.

Conclusions

1. Based on the rheological studies of the heavy oils from Yaregskoe and Usinskoye deposits and the residues (fuel oils) recovered at atmospheric pressure, there have been determined the temperatures at which the studied systems show an anomaly of viscosity and behave like non-Newtonian fluids.

2. Based on the dependence of the shear stress rate, it has been determined that examined oils have a thixotropy. The energy needed to destroy the thixotropic properties of Yaregskaya oil is 245 times more than the energy required to destroy the thixotropic properties of Usinskaya oil.

3. We have determined the presence of a phase transition of atmospheric residues of oils (fuel oils) at 60 ° C, which may correspond to the precipitation of solid paraffins from the system.

4. On the basis of the obtained viscous flow activation energies of oils and their atmospheric residues, it has been found that the activation energy of the Yaregskaya oil is 1.5 times higher than that of the Usinskaya oil, while for the fuel oil these indicators practically coincide.

5. The linear dependence of the enthalpy and entropy of viscous flow activation on temperature for the studied heavy oils of the Timano-Pechorskaya province has been found, while for oil residues at the phase transition temperature, the values of these parameters change drastically.

6. The strength of the bonds of the studied systems decreases with increasing temperature, and the rate of decrease in the strength of the Yaregskaya oil bonds is higher than that of the Usinskaya oil. For atmospheric residues, the rate of decrease in bond strength decreases after heating above the temperature of the phase transition.

7. Based on the positive values of the entropy of studied systems, structural destruction has been observed, which increases insignificantly with increasing temperature. The structure of the Usinskaya oil and resulting fuel oil is more ordered than the structure of Yaregskaya oil and its atmospheric residue, respectively. The entropy of activation of viscous fuel oil flow after the phase transition temperature decreases, which is a consequence of the structure ordering after the precipitation of solid paraffin from the system.

8. Dependences of the molecules jumping frequency on the viscosity of oils and their atmospheric residues before and after the phase transition temperature, which have a power-law character, have been determined.

9. The oils of Yaregskoe and Usinskoe deposits have different physical-chemical and structural-mechanical properties, while the atmospheric residues, which concentrate in themselves resinous-asphaltene components, are practically similar in their properties and have similar energy values of enthalpy and entropy of viscous flow activation. Thus, it can be concluded that the light fractions separated from the oils by direct atmospheric distillation cause them to differ significantly due to the unequal content of solid paraffinic, aromatic hydrocarbons and resins in them, and the high-boiling fractions of oil (fuel oil) – resins and asphaltenes.

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