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# The Effect of Petroleum Resins and Ultrasonic Treatment on the Properties of a Petroleum-Like System

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# Abstract

Complications in the technological processes of hydrocarbon transport (an increase in viscosity, the loss of fluidity, the formation of asphalt-resin-paraffin deposits on the surface of the equipment) arising with a decrease in the temperature of the feed stream cause an increase in energy consumption and reduce the efficiency of petroleum transportation. During the recent decade, the physical methods and their combinations with chemical methods allowing a substantial improvement of the structural-mechanical characteristics of problematic petroleum have been under active development. One of these methods is ultrasonic treatment. The effect of ultrasonic treatment and the addition of petroleum resins on the viscosity, temperature and energy-related parameters of a 6 mass % solution of petroleum paraffin in decane (PP-d) simulating a high-paraffin petroleum system was investigated. Ultrasonic treatment (field frequency 22 kHz, intensity 18 W/cm<sup>2</sup>, duration 10 min) of the PP-d solution stimulates the crystallization of the molecules of petroleum paraffin after the treatment, which causes an increase in viscosity, pour point temperature and the amount of paraffin deposits. The introduction of benzene and benzene-alcohol resins extracted from highly resinous petroleum into the PP-d solution suppresses the crystallization of petroleum paraffin molecules thus promoting a decrease in viscosity and temperature characteristics, the activation energy of viscous flow, the specific fracture energy of the disperse system, the amount of paraffin deposits, changes in the shape and structure of petroleum paraffin crystals. It is demonstrated that alcohol-benzene resins containing a larger amount of heteroatomic functional groups and longer alkyl substituents in aromatic and naphthene cycles are the best depressants of viscosity and pour point. The formation of deposits in the solution under investigation is stronger inhibited by benzene resins containing a larger amount of aromatic structures.

Keywords: petroleum resins, ultrasound, viscosity, crystallization, pour point

## INTRODUCTION

The development of new technologies of production and transportation of problematic petroleum is urgent for Russia and other petroleum-producing countries because the fraction of this hydrocarbon raw material in the total petroleum balance is permanently increasing [1, 2]. In this connection, numerous studies aimed at the improvement of the structural-mechanical parameters of petroleum dispersed systems (PDS) are carried out. A decrease in viscosity and pour point temperature of mined and transported petroleum may be achieved using the following methods: thermal treatment, physical and chemical action, dilution with light-weight petroleum fractions of solvents of various kinds [3-10]. Combined action is often applied to enhance the efficiency of treatment.

At present, the effect of ultrasound on the properties of PDS is investigated extensively. Theoretical and experimental studies in the area of ultrasonic cavitation and acoustic flows have been successfully carried out, and this allowed the development of new technological processes [11-16]. A review of works [8, 10, 11-16] demonstrated that the structural-mechanical properties of petroleum with different component compositions after ultrasonic treatment (UST) change rather ambiguously. The majority of the studies are applied as the very fact of the changes in the properties of petroleum systems is recorded without the revelation of the physicochemical regularities of the process. In this connection, for the prediction of the properties of hydrocarbon raw material after UST, it is necessary to evaluate the effect of different components on the characteristics of a PDS. Previous studies showed [17] that UST of a 6 mass % solution of petroleum paraffin in decane (PP-d) imitating a high-paraffin petroleum system leads to an increase in the temperatures of phase transitions, energy-related and rheological parameters of the system, while the introduction of silica gel resins in the concentration of 0.3 mass % into the initial solution promotes a substantial decrease in these parameters. It appears important to establish the structural units that have larger effects on the viscositytemperature and energy-related characteristics parameters of petroleum-like systems after UST.

The goal of the present work was to study the effect of UST and the addition of benzene resins (BR) or alcohol-benzene resins (ABR) on the structural-mechanical properties of a 6 mass % PP-d solution.

#### EXPERIMENTAL

The initial 6 mass % PP-d model solution imitating a high-paraffin petroleum system was prepared using petroleum paraffin (GOST 23683-89) and decane (TU 6-09-3614-74). Ultrasonic treatment of the solution was carried out with the help of a UZDN-2T ultrasonic disintegrator (NPP Ukrrospribor, Ukraine). The set-up included a magnetostrictive transducer (the working frequency 22 kHz) and a rod waveguide with the diameter of the working end of the tool equal to 1.72 cm. The efficiency factor for the transducers of this type is 30-40 % [18]. Calorimetry was used to estimate the acoustic power of oscillations absorbed by water [19]. The power of ultrasonic action was 42 W. The treatment was carried out in the technological chamber 0.1 dm<sup>3</sup> in volume, so the acoustic power for the implementation of processes under study was not less than 420 W/dm<sup>3</sup>. For the transducer end radius equal to 0.86 cm, the intensity of ultrasound is 18 W/cm<sup>2</sup>. The duration of treatment of solutions under study, 0.1 dm<sup>3</sup> in volume, was 10 min at the ambient temperature equal to 20 °C.

Benzene and alcohol-benzene resins (BR and ABR, respectively) were isolated from high-resin petroleum from the deposit in the Republic of Komi using standard procedures [20]. The relative content of structural fragments (structural group composition) of resins was determined using the data of IR spectroscopy. IR spectra were recorded with a NICOLET 5700 FTIR spectrometer (Thermo Electron, USA) in the region of 400-4000 cm<sup>-1</sup>. The spectra were processed and optical density was determined with the help of OMNIC 7.2 software. To determine the relative content of structural fragments, we used characteristic absorption bands at 1730, 1700, 1650 cm<sup>-1</sup> related to - C=O-group in esters, acids and amides, respectively, at 1600 cm<sup>-1</sup> - C=C-bonds of the aromatic ring, 850, 805, 760  $\text{cm}^{-1}$  – the aromatic triplet of benzene derivatives, 1465 cm<sup>-1</sup> aliphatic C-H-bonds, 1380 cm<sup>-1</sup> - CH<sub>2</sub>-groups, 1030 cm<sup>-1</sup> - S=O-groups, 720 cm<sup>-1</sup> -  $(CH_{2})_{n}$ groups in aliphatic structures with n > 4 [21]. The relative content of a structural fragment was estimated from the ratio  $D_i/D_{1465}$ , where  $D_i$ ,  $D_{1465}$ are integral optical densities of the characteristic band and the band at 1465 cm<sup>-1</sup>, which was used as an internal standard [22]. According to the obtained values of normalized optical densities (Table 1), BR contain larger amount of aromatic structures, while the content of sulphoxide substituents, C=O-groups (in particular in amides), aliphaticity degree (( $D_{\rm _{720}}$  +  $D_{\rm _{1380}})/D_{\rm _{1600}}$ ) and branching degree  $(D_{1380}/D_{1465})$  are lower than in ABR.

The solution of petroleum resins in chloroform (1:2) was introduced into the PP-d solution immediately after UST with the help of pipette aid. The added amount of chloroform (0.6 g per 100 g of solution) does not affect the viscosity-temperature properties of the PP-d solution.

The rheological parameters of the solutions of petroleum paraffin were determined using a ro-

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Data	of	IR	spectroscopy	of	petroleum	resins

Resins	$f_{\rm a}$	Positions of absorption bands, cm <sup>-1</sup>									
		1730	1700	1650	1600	1380	1030	850	805	760	725
		Norma	Normalized optical densities, $D_i/D_{1465}$								
BR	2.214	0.217	0.268	0.182	0.328	0.597	0.224	0.166	0.186	0.196	0.131
ABR	2.383	0.415	0.292	0.302	0.279	0.631	0.515	0.151	0.177	0.162	0.136

*Note.* Aliphaticity degree  $f_a = (D_{720} + D_{1380})/D_{1600}$ ; branching degree  $= D_{1380}/D_{1465}$ .

tary viscosimeter HAAKE Viscotester iQ (Thermo Scientific, USA). Viscosity-temperature dependences were recorded under the permanent decrease of temperature (T) at a rate of 0.3  $^{\circ}C/min$ with the shear rate  $1 \text{ s}^{-1}$ , at which the destruction of the structure of the thixotropic system is minimal. The activation energy of viscous flow  $(E_{a})$ was determined from the tangent of the straight regions of  $\ln \mu = f(1/T)$  dependences, where  $\mu$  is the effective viscosity of the solution, mPa/s; T is temperature, K [23]. The energy of the destruction of PDS supramolecular structure was calculated from the area of the loop formed by the flow curves of the forward and back motion recorded at a temperature of 10 °C according to the procedure described in [24]. Rheological measurements were carried out after 20 s at a shear rate up to 85 s<sup>-1</sup>. The equation for the energy of the system is written as

 $dW_{u} = dQ + dA + dZ \tag{1}$ 

where  $W_u$  is the internal energy of the system; Q is heat; A is work; Z is the energy of mass transfer. At a constant temperature and the absence of

At a constant temperature and the absence of mass transfer, the change of the internal energy  $(dW_u)$  will be due to work (dA).

The area limited by the curves of forward and back motion is proportional to the energy  $(\Delta W)$  of the destruction of the supramolecular structure of the dispersed system the steady value of rheological parameters. To calculate  $\Delta W$ , it was necessary to pass from shear rates ( $\gamma_i$ ) and shear strain ( $\tau_i$ ) to the moment of resistance to the rotation of the cylinder of rotary viscosimeter ( $M_i$ , N · m) and to the angular frequencies of revolution ( $\omega_i$ , s<sup>-1</sup>), which were determined using equations

$$M_i = 2\pi H r 2\tau_i$$
(2)  

$$\omega_i = (h/r)\gamma_i$$
(3)

where H is cylinder height; r is cylinder radius; h is the gap between the walls of cylinders in the viscosimeter.

The flow curves were recalculated over steps using equations (2) and (3). A smooth change of shear rate from 0 to 85  $\rm s^{-1}$  and back proceeds within 720 s.

Then the mechanical power  $(P_i)$  of the viscosimeter was calculated using the equation

 $P_i = M_i \omega_i \tag{4}$ 

Energy  $(W_i)$  at every step of the time interval was calculated for the forward and back motion from the dependences of mechanical power on time  $P_i(t)$ :

$$W_{i} = \Delta P \Delta t N \tag{5}$$

where  $\Delta P$  is the graduation value of the power axis;  $\Delta t$  is the time interval; *N* is the number of steps.

The energy of destruction ( $\Delta W$ ) of the dispersed system is defined as

 $\Delta W = W_i$ (forward motion) –  $W_i$ (back motion)

For energy values to be independent of the parameters of cylinders, we introduced the specific energy ( $\Delta W_{sp}$ , J) of the destruction of the dispersed system for 1 m<sup>3</sup> of the sample:

 $\Delta W_{\rm sp} = \Delta W / v$ 

where v is the volume of liquid retained in the gap between the cylinders, m<sup>3</sup>.

The cloud point  $(T_c)$  and the pour point  $(T_p)$  of PP-d solutions was determined with the help of INPN Kristall SX-800 instrument (Russia). The temperature of the maximal crystallization rate was estimated during a gradual decrease in temperature relying on the differential dependences of light transmission on temperature.

Precipitation was studied using a set-up operating on the basis of the cold rod principle under the following conditions: rod temperature 8 °C, sample temperature 30 °C, time of experiment 1 h, sample portion 40 g. The mass of the precipitate formed on the rod was determined gravimetrically. The obtained values were calculated for 100 g of the solution. The inhibiting capacity (I, %) of resins was calculated using the equation:  $I = (m_0 - m_c) \cdot 100/m_0$ 

where  $m_0$  is the mass of the precipitate in the initial solution;  $m_c$  is the mass of the precipitate in the solution with resins.

Investigation of the microstructure of the precipitates of petroleum paraffin was carried out by means of optical microscopy in transmitted light with the help of a Zeiss AXIO LAB.A1 microscope (Carl Zeiss, Germany). The microphotographs were processed using Zen and Axio Vision software (Zeiss).

# **RESULTS AND DISCUSSION**

Structural transformations that are observed in the PP-d solution during cooling were studied using the dependences of the viscosity of the system on temperature (Fig. 1). The viscosity-temperature curve for the initial PP-d solution is represented by several regions (see Fig. 1). With a decrease in temperature to 13 °C, the viscosity of the solution under study increases slowly, which is connected with the nucleation of paraffin particles in the initial dispersing medium (decane). During further cooling of this dispersed system, a sharp increase in viscosity is observed due to the aggregation of primary crystals. At a temperature of about 2.5 °C, a sol-gel transition is observed, and the system loses the liquid behavior.

After UST, the viscosity of the initial solution within temperature range 6-15 °C decreases substantially, however, at 5 °C it reaches the initial value, and then, with a further decrease in temperature, exceeds it. This viscosity-temperature

dependence may be due to intense relaxation processes taking place in the system.

After UST of PP-d solution followed by the introduction of either BR or ABR, viscosity decreases substantially, especially in the temperature region below 13 °C. Aggregation of primary crystals after the integrated action is decelerated, and viscosity-temperature curves are smoothed. The temperatures of sol-gel transition in the treated samples shift to negative temperatures: -6.7 and -8.3 °C after the introduction of BR and ABR, respectively.

Viscosity-temperature dependences are used to calculate the activation energy of viscous flow  $(E_{\perp})$  of PDS from the tangent of the slope of linear regions on the curve  $\ln \mu = f(1/T)$  [23]. For the initial PP-d solution and the sample after UST, the dependence of the logarithm of viscosity on reciprocal temperature (Fig. 2) is expressed by two linear functions with an inflection at 2.5 °C, so the rheological properties of these samples are characterized by two values of activation energy within the temperature range under study:  $E_{\circ}$  within the range 3-15 °C is 55 and 174 kJ/mol, while below 3  $^{\circ}\mathrm{C}$  – 340 and 520 kJ/mol, respectively. For samples with the addition of resins, straight-line dependences are observed within a broader temperature range (from -8 to 15 °C). The values of  $E_{\circ}$  for PP-d solutions after the joint action of UST and the addition of petro-



Fig. 1. Viscosity - temperature curves for PP-d solution flow.



Fig. 2. The logarithmic dependence of efficient viscosity of  $\mbox{PP-d}$  solution on reciprocal temperature.



Fig. 3. Flow curves of straight and back run for PP-d solutions at 10  $^{\circ}\mathrm{C}.$ 

leum resins are lower: 112 and 60 kJ/mol in the presence of BR and ABR, respectively.

The degree of structuring of dispersed systems may be described with the help of specific energy of the destruction of supramolecular structure ( $\Delta W_{sn}$ ). The ascending and descending curves of the flow of the initial PP-d solution are presented in Fig. 3; they form a hysteresis loop. Hysteresis phenomena in thixotropic systems are usually linked with retardation of the processes of structure recovery or insufficient destruction of initial structure [25]. The  $\Delta W_{_{\rm SP}}$  value calculated from the area of the hysteresis loop for initial sample is 54 J/m³. After UST, the  $\Delta W_{_{\rm SP}}$  value increases (100  $J/m^3$ ), which is connected with the formation of stronger structures. The mergence of the flow curves of the straight and back run and consequent approach of  $\Delta W_{\rm sp}$  value to zero for PP-d solutions after UST with the addition of either BR or ABR provide evidence of Newton flow of a non-structured system.



Fig. 4. Microphotographs of the crystal structure of deposits isolated from the solutions: PP-d (a), PP-d + UST (b), PP-d + UST + BR (c), PP-d + UST + ABR (d).

Sample	Temperature, °C					
	Cloud	Maximal crystallization	Pour			
	point $(T_{\rm c})$	rate	point $(T_p)$			
PP-d	17.6	16.0	10.0			
PP-d + UST	18.3	17.4	13.0			
PP-d + UST + ABR	19.4	18.7	1.2			
PP-d + UST + BR	20.7	19.8	3.5			

TABLE 2 Effect of processing conditions on the temperature parameters of PP-d solution

The low-temperature properties of hydrocarbon systems may be characterized by such parameters as cloud point, maximal rate of crystallization and pour point (Table 2). For the systems under investigation, a trend to increase the cloud point from 17.6 to 20.7 °C may be traced in the sequence PP-d  $\rightarrow$  PP-d + UST  $\rightarrow$  PP-d +  $UST + ABR \rightarrow PP-d + UST + BR$  (see Table 2). The same sequence is observed also for the temperature of the maximal crystallization rate. Quite the contrary, the pour point  $(T_p)$  in solutions with the addition of resins, is lower by 8–10 °C than in the initial system ( $T_p = 10$  °C). The introduction of 0.3 mass % BR into the treated PP-d solution promotes a decrease in  $T_p$  by 6.5 °C. The maximal depression of  $T_{p}$  (by 8.8 °C) in PP-d solution is observed in the presence of 0.3 mass % ABR (see Table 2).

Changes in the structural-mechanical properties of PP-d solutions after UST and the introduction of BR and ABR are depicted in precipitation. The addition of resins into solution treated with ultrasound promotes a decrease in the amount of precipitate; in the presence of BR, precipitation is inhibited in the solution under study to a higher extent (74 %) than after the addition of ABR (37 %).

The structure of the formed deposits was studied by means of optical microscopy. One can see in Fig. 4, *a* that the precipitate formed in the initial PP-d solution is composed of radial ray-like crystal aggregates up to 250  $\mu$ m in diameter and fibrillar crystals 30–240  $\mu$ m long. After the UST of the solution under investigation, the degree of dispersity of aggregates increases (see Fig. 4, *b*). The joint action of UST and petroleum resins leads to a substantial change in the structure of crystallized petroleum paraffin: a fibrous precipitate with the elements of dendrites is formed in the presence of BR (see Fig. 4, *c*), while after the addition of ABR the precipitate is a system of de-oriented rod-like particles (see Fig. 4, *d*).

#### CONCLUSION

The effect of the action involving UST and the addition of BR or ABR on the structural-mechanical properties of the PP-d solution is demonstrated. Independently of structure, petroleum resins added into preliminarily treated PP-d solution promote the improvement of viscosity-temperature and energy-related parameters of the system and decrease the amount of precipitate. The addition of petroleum resins into solution after UST shifts the first stage of crystallization (nucleation of centres) into a higher temperature region and at the same time stabilizes the primary crystal formations changing the shape and size of paraffin crystals. The introduction of BR and ABR promotes the formation of a disordered structure, which decreases the expenses of energy for its destruction.

According to the obtained results, ABR are stronger depressors of viscosity-temperature characteristics. This may be explained by the content of heteroatoms and longer alkyl substituents in aromatic and naphthene cycles of ABR molecules in comparison with BR. So, ABR may be more readily sorbed on the surface of nucleating crystals of petroleum paraffin and prevent their growth with the formation of a three-dimensional network.

The formation of the deposit is slowed down to a higher extent in the case if BR is added, which may be due to the formation of paraffin crystals containing a higher amount of voluminous condensed aromatic structural fragments at the periphery, which prevents the aggregation of primary crystal formations.

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