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Acidic Oil-Displacing System Based on Deep Eutectic Solvents and Surfactants: Development, Physical and Chemical Studies, Evaluation of Its Effect on the Composition and Properties of Oil

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Abstract. The paper presents the findings of a study of a ternary system of deep eutectic solvents (DES) 'boric acid– glycerol – carbamide', whose combination with surfactants for enhanced oil recovery forms the basis of an acidic oil-displacing composition. The results of laboratory investigation of the effect of a DES and surfactants-based composition on the filtration characteristics of a heterogeneous formation of a terrigenous reservoir of the Russkoye field are also reported. An oil-displacing composition characterized by physicochemical parameters controlled in a wide range has been developed on the basis of experimental phase equilibrium data for the components of, DES systems. The method of physical modeling is used to evaluate the effectiveness of the system as applicable to oilfields at the early and late stages of development. It has been established that the treatment of a heterogeneous reservoir model with a composition results in a significant increment in the oil displacement efficiency both at low and high temperatures due to the smoothing of filtration flows. The increase in the reservoir coverage and restoration of the initial permeability are also observed. The study of the composition and properties of heavy high-viscosity oil before and after its treatment with the system show that the use of an acid oil-displacing system based on DES and surfactants causes no significant changes in the qualitative composition of the oil but generally leads to a redistribution of the content of low and higher molecular structures. Hence, light occluded hydrocarbons are preferably displaced, while heavy oil components are adsorbed on the reservoir.

Keywords: deep eutectic solvents; methods of enhanced oil recovery; acid oil-displacing composition; surfactant; eutectics, oil displacement efficiency, heavy and high-viscosity oils.

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Кислотная нефтewытесняющая композиция на основе глубоких эвтектических растворителей и ПАВ: создание, физико-химические исследования, оценка влияния на состав и свойства нефти

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Аннотация. В работе представлены результаты исследования тройной системы глубоких эвтектических растворителей (ГЭР) «борная кислота – глицерин – карбамид», составляющих основу кислотной нефтewытесняющей композиции на основе ГЭР и ПАВ для увеличения нефтewотдачи пластов, а также лабораторных исследований влияния композиции на основе ГЭР и ПАВ на фильтрационные характеристики неоднородного пласта терригенного коллектора Русского месторождения. На основе результатов исследований фазовых равновесий компонентов систем ГЭР была создана нефтewытесняющая композиция, характеризующаяся регулируемыи в широком диапазоне физико-химическими параметрами. Методом физического моделирования оценена эффективность композиции применительно к условиям месторождений, находящихся на ранней и поздней стадиях разработки. Установлено, что обработка модели неоднородного пласта композицией приводит к существенному приросту коэффициента нефтewытеснения как при низких, так и при высоких температурах, за счет выравнивания фильтрационных потоков, увеличения охвата пласта и восстановления начальной проницаемости. В результате исследований состава и свойств тяжелой высоковязкой нефти до и после обработки композицией установлено, что применение кислотной нефтewытесняющей композиции на основе ГЭР и ПАВ не вызывает значимых изменений в качественном составе нефти, а приводит главным образом к перераспределению содержания низко- и более высокомолекулярных структур: вытесняются

преимущественно лёгкие окклюдированные углеводороды, в то время как тяжелые компоненты нефти адсорбируются на коллекторе.

Ключевые слова: глубокие эвтектические растворители; методы увеличения нефтеотдачи пластов; кислотная нефтewытесняющая композиция; ПАВ; эвтектика, коэффициент нефтewытеснения, тяжелые и высоковязкие нефти.

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Introduction

Currently, oil is the most important fossil fuel and the main source of energy used throughout the world. As a rule, only one third of the original oil can be produced in oil fields by flooding due to low pore or capillary space accessibility of the reservoir [1]. In order to extract the residual oil, physicochemical methods of oil recovery enhancement are used [1–11]. The use of physical and chemical methods of enhanced oil recovery allows additional oil recovering and maintaining or extending the profitability of field development. Some of these methods are aimed at reduction of residual oil saturation by increasing the capillary number and reducing interfacial and surface tension at the rock-water-oil interface. Other methods of enhanced oil recovery allow additional oil recovering due to the treatment of reservoir fluids and reservoir rock with chemical compositions causing an increase in rock permeability or redistribution of filtration flows of the hydrodynamic system of the reservoir. Injected chemicals change rock and fluid properties such as reservoir rock wettability, oil and water emulsification, etc. [11, 12]. Hence, increasing attention is paid to the development and optimization of physical and chemical methods for enhanced oil recovery (EOR methods). One of the newest approaches to the development of chemical compositions for complex physical and chemical methods of enhanced oil recovery is the principle of ‘green chemistry’ using deep eutectic solvents (DES). The DES are systems that include two or more compounds in the form of a mixture consisting of donor and acceptor of hydrogen bond. This mixture becomes eutectic, when components are mixed in a certain ratio, i.e. its melting point becomes much lower than that of any of the individual components [12–16].

Based on this principle, the researchers of the Institute of Petroleum Chemistry SB RAS have developed an acidic chemical composition (system) intended to increase oil recovery from reservoirs with heavy and high-viscosity oils [11, 17]. DES and surfactants based compositions (systems) for enhanced oil recovery have the following unique properties which are their advantages: high oil-displacing ability; bifunctionality, i.e. the system works as an acidic and oil-displacing composition; complex impact on

the reservoir, i.e. the system interacts with the reservoir rock and fluids; adjustable viscosity; evolution under reservoir conditions; during the hydrolysis of carbamide, which is component of the system, the pH value shifts to the range of alkaline values. This suggests a formation of a high-capacity alkaline buffer system capable to provide optimal conditions for surfactant operation; ability to smooth and redistribute filtration flows; low freezing point (important for use in the Arctic zones and northern regions of the Russian Federation); durable effect and low corrosivity.

This paper presents the findings of a study of a three-component DES system ‘boric acid-glycerol--carbamide’ with a view to developing an acidic DES and surfactants based oil-displacing composition and the results of its using in a physically modeled process of oil displacement.

It is known that as a result of the use of physical and chemical EOR methods, the state of the initial thermodynamic equilibrium between oil components and rock, formation water, etc. is disturbed, resulting in a change in the composition of mobile and residual reservoir oil. In this regard, it was of interest to study the composition and properties of the residual oil extracted from the cores of the Russkoye oilfield under laboratory conditions. This paper also presents the results of study of the effect of the system on the composition of displaced high-viscosity oil from the Russkoye field in the physically modeled oil displacement process.

Experimental

To create an acidic DES and surfactants based oil-displacing composition, experimental studies of phase equilibria have been conducted and a fusion diagram for a three-component DES system has been constructed. For this purpose, mixtures of components were prepared in a molar ratio of 1:10 ÷ 10:1, followed by heating or cooling and determining the point of melting or crystallization. A detailed description of the procedure for the preparation of DES is given in a previously published work [11].

The density of solutions of the systems was determined by the pycnometric method using an EASY D 40 density meter. The pH values were determined via the potentiometric method using a HANNA Instruments microprocessor-based laboratory pH meter with glass electrode.

Rheological properties of DES and acidic DES and surfactants based oil-displacing composition were investigated in the temperature range 20–150 °C via vibrational viscometry using a ‘Rheokinetika’ viscometer with a tuning fork sensor and rotational viscometry using HAAKE Viscotester iQ viscometers (CC25 DIN /Ti coaxial cylinder measuring system) and Reotest-2.1.M (S/S coaxial cylinder measuring system) at different shear rates (when the shear rate changes from 10 to 1200 s⁻¹ or at a shear rate of 3 s⁻¹).

Analysis of the composition of organic compounds was carried out using a Thermo Scientific DFS magnetic chromato-mass spectrometer (Germany). Separation was performed using an Agilent quartz capillary chromatographic column (an inner diameter of 0.25 mm, a thickness of 0.25 mm, a length of 30 m, and a DB-5MS stationary phase) with helium as a carrier gas.

The structural composition of oils was determined by IR spectroscopy using a Nikolet 5700 IR Fourier spectrometer with a Raman module.

The following compounds have been identified in the composition of organic compounds of oil via gas chromatography-mass spectrometry: n-alkanes, cyclohexanes, aromatic compounds, steranes, hopanes, sesquiterpanes and secohopanes.

Table 1. Characteristics of models of a heterogeneous reservoir

Column No.	Gas permeability, μm^2	Ratio of gas permeabilities of models	Oil viscosity, mPa·s/ oil density, kg/m ³	Pore volume, cm ³	Initial oil saturation, %
1	0.84	2.41:1	43.3 / 902.0	49.7	58.6
2	0.34			54.1	65.7

Laboratory investigation of an acidic DES and surfactants based oil-displacing system was performed using an installation for studying filtration characteristics (KATAKON LLC, Russia) as applied to heavy high-viscous oil from the Russkoye field. The procedure of physical modeling of the oil displacement process is reported in detail in the previously published work [11].

For filtration tests, a model of a heterogeneous reservoir was prepared, which consisted of two parallel columns filled with disintegrated core (terrigenous material) with different permeability. The columns were sequentially saturated with models of reservoir water (with pore volume determination) and oil (Table 1).

The gas permeability of the columns of the heterogeneous reservoir model was 0.34–0.84 μm^2 . The values of gas permeability were measured according to the method defined in the standard GOST 23409.6–78. The column permeability ratio was 2.41:1. The viscosity of oil was 43.3 mPa·s. The initial oil saturation was 58.6 and 65.7 % for the first and second columns, respectively.

Results and discussion

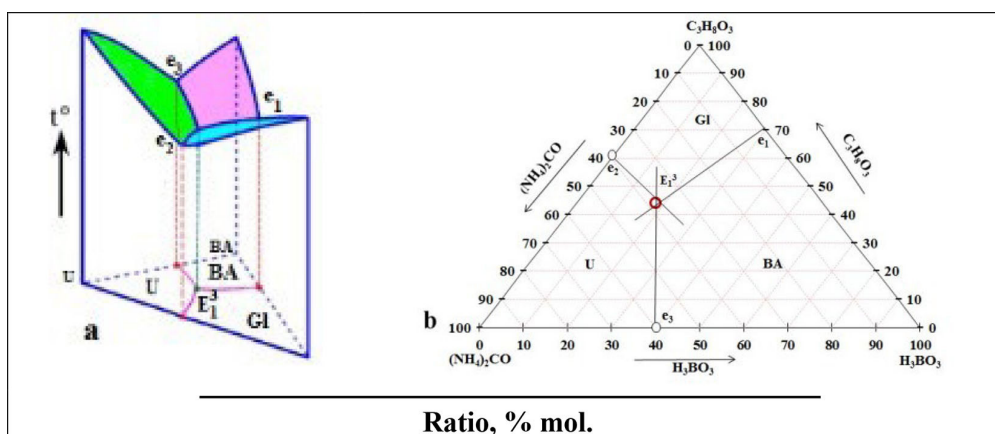
Creation of an acidic oil-displacing composition based on surfactants and the ternary DES “boric acid– glycerol – carbamide” system

To create an acidic oil-displacing composition based on surfactants and DES (glycerol, carbamide and boric acid), a phase diagram of a three-component “boric acid– glycerol – carbamide” system was constructed. The phase diagram was based on previously studied two-component “boric acid– glycerol”, “boric acid– carbamide” and “carbamide – glycerol” systems [18]. Fig. 1 shows the phase melting diagram of the ternary DES “boric acid– glycerol – carbamide” system.

Fig. 2 shows a pattern of the donor-acceptor interaction in the three-component system “boric acid– glycerol – carbamide”, where boric acid is an electron pair acceptor toward glycerol and carbamide, carbamide is a hydrogen bond donor toward boric acid and an acceptor toward glycerol, while glycerol is an electron pair donor for boric acid and carbamide. It should be noted that the ternary DES “boric acid– glycerol – carbamide” system leads to an increase in the acidity and buffer capacity of the composition. The system also promotes an expansion of the range of buffer action in the acidic pH region and an increase in the duration of its action in the reservoir.

Based on the eutectic composition of the three-component DES “boric acid– glycerol – carbamide”, an acidic surfactant based oil-displacing system has been created.

The concentration of water in the composition is 15 %. All considered oil-displacing compositions contain surfactants providing an increase in the displacement ability of the solutions under study and facilitating their access to the reservoir rock. Non-ionic surface acting agents (NSAA) were used as compatible with mineralized formation waters. In order to use the compositions of nonionic surfactants at reservoir temperatures above 100 °C, anionic surfactants (ASAA) increasing the cloud point of the nonionic surfactant were introduced into the compositions.



BA – H_3BO_3 , GI – $\text{C}_3\text{H}_8\text{O}_3$, U – $(\text{NH}_2)_2\text{CO}$; e_1 , e_2 and e_3 are monovariant cotectic lines of simultaneous crystallization of two phases, which begin in binary and converge in a triple eutectic. E_1^3 – triple (according to the number of components) eutectic of invariant simultaneous crystallization of three phases from the melt

Fig. 1. Space diagram (a) and planar diagram of ternary system (b)

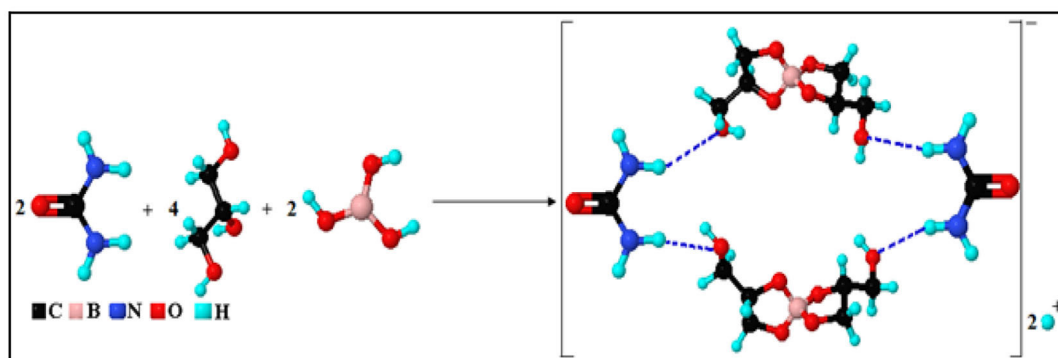


Fig. 2. Pattern of the donor-acceptor interaction of the three-component system “boric acid – glycerol – carbamide”

The composition is compatible with mineralized reservoir waters. It has a low freezing point (minus 20 ÷ minus 50 °C) and low interfacial tension at the boundary with oil. The density of the composition can be adjusted from 1100 to 1300 kg/m^3 and the viscosity from tens to hundreds of $\text{mPa}\cdot\text{s}$. The composition slowly reacts with carbonate rocks. High oil-displacing capacity, compatibility with saline reservoir waters, and a decrease in the swelling of clays promote an additional displacement of residual oil from both high-permeability and low-permeability reservoir zones.

Fig. 3 shows the results of the solubility of the disintegrated carbonate reservoir rock in an acid oil-displacing DES and surfactants based composition, the change in pH of the solution before and after thermostating for 6 hours at various temperatures.

Fig. 3(a) shows that the solubility of carbonate rocks in the composition increases depending on time. With an increase in temperature above 70 °C, the pH of the composition shifts to the alkaline region, therefore the composition becomes alkaline, Fig. 3(b). The evolution of an acidic DES and surfactants based oil-displacing system occurs due to the hydrolysis of carbamide, which is a component

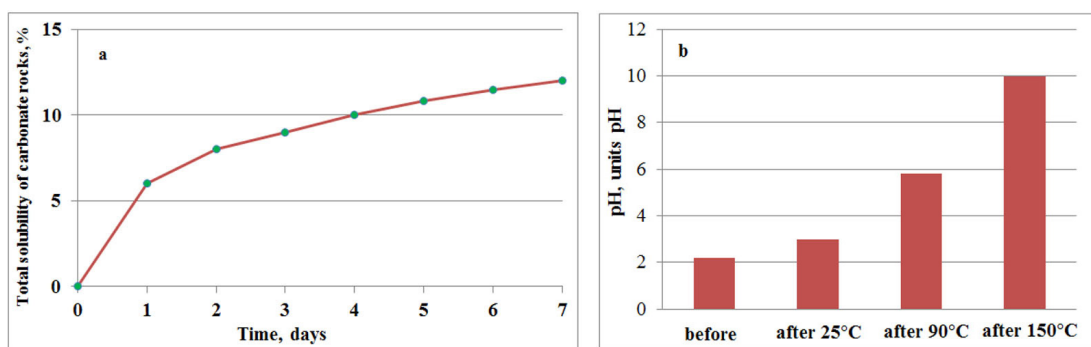


Fig. 3. Solubility of disintegrated carbonate reservoir rock in an acidic oil-displacing DES and surfactants-based composition depending on the duration of its treatment at 25 °C (a); change in pH before and after thermostating for 6 hours at various temperatures (b)

of the system. The reaction proceeds to form carbon dioxide and a borate-ammonia buffer system with a maximum buffer capacity in the pH range 9–10, which is optimal for oil displacement purposes. This results in a decrease in interfacial tension and liquefaction of highly viscous layers or films at the oil-water-rock interfaces.

The acidic oil-displacing system will have a dual effect on the carbonate reservoir and the carbonate cement of the terrigenous reservoir, which is as follows:

- At low reservoir temperatures, the interaction of an acidic DES and surfactants based oil-displacing system with carbonate rocks and carbonate cements of terrigenous rocks will restore the initial reservoir permeability to form water-soluble salts and reduce the swelling of clays, while the released CO_2 will dissolve in the oil reducing its viscosity. As a result, additional displacement of residual oil will occur.
- When the temperature in the reservoir rises above 70 °C, the rate of hydrolysis of carbamide, which is a component of the system, will increase. This will promote further release of CO_2 accompanied by a decrease in oil viscosity, while pH of the system will increase from 2.6–3.2 to 9.0–10.0 pH units. As a result, an alkaline oil-displacing system with a high buffer capacity will be formed directly in the reservoir, which ensures effective oil displacement and prolonged stimulation of the reservoir, Fig. 3(b).

Physical modeling of the oil displacement process using an acidic DES and surfactants based oil-displacing system

In order to draw conclusions about the applicability of the created acidic DES and surfactants based oil-displacing system under various geological and physical conditions and at different stages of field development and to evaluate its effect on the oil displacement and sweep efficiency of the reservoir in case of water flooding or thermal steam treatment, the filtration characteristics and oil-displacing ability of the system were investigated for the conditions of the carbonate reservoir of the Russkoye oilfield. Table 2 and Fig. 4 and 5 present the data on the effect of an acidic DES and surfactants based oil-displacing system on the filtration characteristics of the reservoir model of the Russkoye field at temperatures of 23 and 150 °C simulating the reservoir temperature and thermal steam treatment.

The model of reservoir water from the Russkoye oilfield was filtered with an injection rate of 1 cm^3/min at a temperature of 23 °C through a water-oil-saturated model of a heterogeneous reservoir in the direction ‘reservoir-well’. The oil displacement coefficient for the first and second columns when

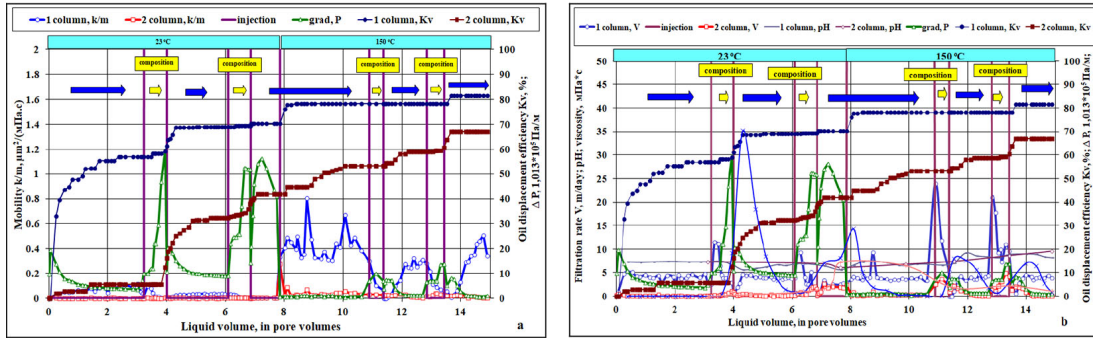


Fig. 4. Filtration characteristics of the Russkoye oilfield reservoir model: change in pressure gradient P , coefficient of oil displacement C_d , mobility ratio k/μ (a) and filtration rate V (b) at 23 and 150 °C after injection of an acidic DES and surfactants based oil-displacing system. Initial gas permeability of models: 1st column – 0.84 μm^2 , 2nd column – 0.34 μm^2

Table 2. Results of the study of the filtration characteristics of the reservoir model and the oil-displacing ability of the acidic DES and surfactant based oil-displacing composition

No. of column	Gas permeability of column, μm^2	Mobility ratio (before/after injection of composition)	Oil displacement efficiency		Maximum pressure gradient when pumping of composition, MPa/m
			Displacement with water / water and composition, %	Increment of the oil displacement coefficient due to the use of the composition, %	
1	0.84	48.9:1 / 1.24:1	56.9 / 81.5	24.6	5.625
2	0.34		5.6 / 66.9	61.3	

pumping 3.2 pore volumes of the reservoir water model was 56.9 and 5.6 %, respectively. After formation water filtration, at 23 °C in the “well-formation” direction, two rims of the composition were pumped in a volume equal to 0.5 of the pore volume of the heterogeneous reservoir model, and advanced to a predetermined distance with water. The increase in oil displacement coefficients at 23 °C due to two rims of the composition was 13.3 % for the first column and 35.9 % for the second column.

Table 2 shows the results obtained in the study of filtration characteristics of a heterogeneous reservoir and the oil-displacing ability of the composition.

Then the temperature was raised to 150 °C and the formation water model continued to be injected in the “reservoir-well” direction. Modeling of the steam-thermal effect on the formation resulted in additional oil washout, the increase in the oil displacement efficiency was 8.0 % for the first column and 11.3 % for the second column. Similarly, at a temperature of 150 °C in the “well-formation” direction, two rims of the composition were pumped in a volume equal to 0.5 of the pore volume of the heterogeneous reservoir model. The increase in the oil displacement efficiency due to two rims of the composition and subsequent filtration of the formation water model was 3.3 % for the first column and 13.1 % for the second column.

Analysis of the components of the composition in water samples taken at the outlet of the heterogeneous reservoir model revealed that after injection of the composition, the pH value of the

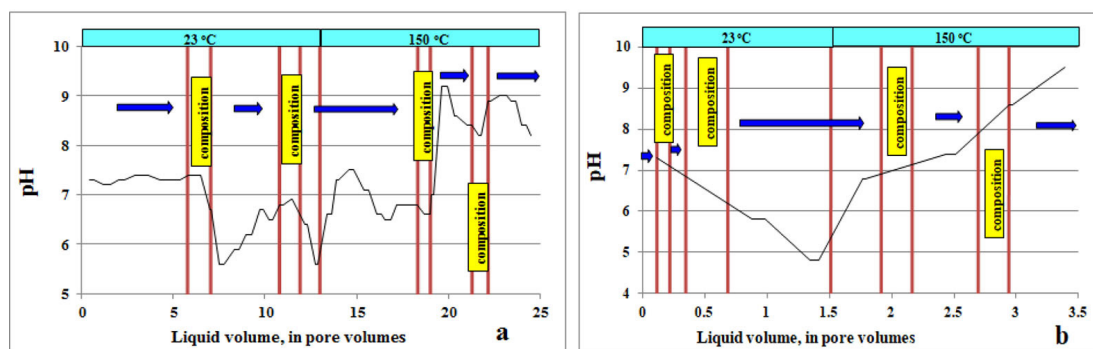


Fig. 5. Changes in pH before and after injection of the acidic DES and surfactant based oil-displacing composition into a reservoir model at 23 and 150 °C: a) with a gas permeability of $0.84 \mu\text{m}^2$, b) with a gas permeability of $0.34 \mu\text{m}^2$

samples decreases from 7.4 to 5.2 units. As a result of carbamide hydrolysis, it shifts after heating to 150 °C and thermostating to the alkaline region, reaching 9.5 pH units (Fig. 5).

Thus, it is experimentally confirmed that at high temperatures directly in the reservoir model the composition becomes alkaline due to the hydrolysis of carbamide, which is a part of the composition. In this case, carbon dioxide CO_2 and a borate-ammonia buffer system are formed, which creates optimal conditions for the effective washing ability of surfactants.

Effect of the acidic DES and surfactants based oil-displacing system on the composition and properties of heavy high-viscosity oil during physical modeling of the oil-displacement process

As a result of the study of the original oil and that displaced from the reservoir model by the methods of viscometry and pycnometry, it was found out that the viscosity and density of oil changes after contact with an acidic oil-displacing composition based on DES and surfactants. This is partly due to the formation of carbon dioxide and ammonia during the hydrolysis of carbamide. Carbon dioxide, dissolving in oil, leads to a decrease in its viscosity, while ammonia provides an optimal pH of the solution, which contributes to the displacement of mainly light and occluded hydrocarbons, while heavy oil components are adsorbed on the reservoir.

Fig. 6 shows the results of changes in the viscosity and density of oil from the Russkoye field during oil displacement.

An analysis of the spectral coefficients revealed quantitative differences in the IR spectra of the oils under study before and after treatment with the acidic DES and surfactants-based oil-displacing composition (Fig. 7).

Samples of oil displaced using the composition at the Russkoye oilfield are characterized by a decrease in the coefficient of the relative content of aromatic structures, including a decrease in the coefficient of the relative content of tricyclic arenes and alkanes (A_2). Slight changes are observed for C_2 , C_3 , and C_4 spectral coefficients, which characterize the conditional content of ether, sulfoxide, and $\text{C}=\text{O}$ groups (Table 3).

The study of the classes of compounds showed that the changes in the oil sample of the Russkoye field are insignificant: the content of *n*-alkanes decreases by 1.11 times, the content of naphthenes

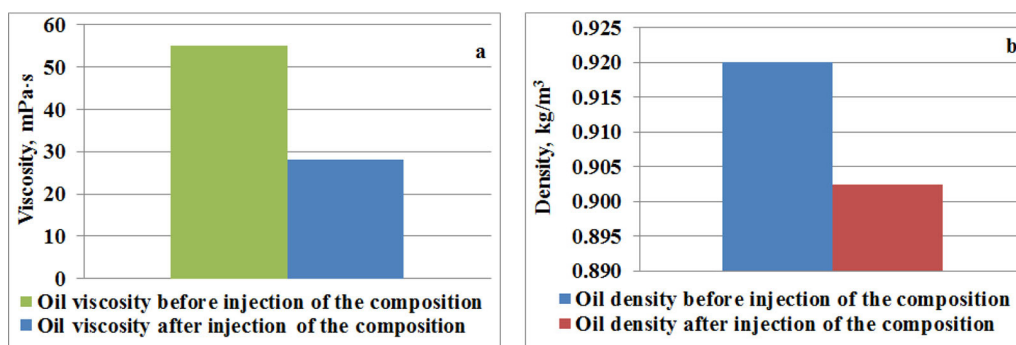


Fig. 6. Changes in viscosity (a) and density (b) of oil from the Russkoye field during oil displacement

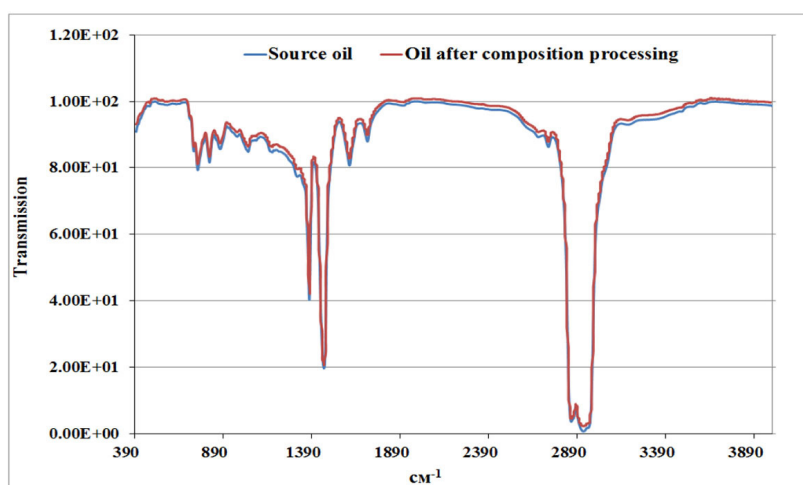


Fig. 7. IR spectrum of the original oil from the Russkoye field and that of the oil treated with the acidic DES and surfactants-based oil-displacing composition

Table 3. Spectral coefficients of oil sampled from the Russkoye oilfield before and after injection of the acidic DES and surfactants based oil-displacing composition

Spectral coefficients	Russkoye oilfield	
	Original oil	Oil after injection of the composition
$C_1 = D_{1603}/D_{724}$	1.31	1.269
$C_2 = D_{1167}/D_{1460}$	0.102	0.097
$C_3 = D_{1031}/D_{1460}$	0.101	0.096
$C_4 = D_{1705}/D_{1460}$	0.075	0.073
$A_1 = D_{813}/D_{747}$	0.874	0.978
$A_2 = D_{813}/D_{724}$	0.946	0.920

increases by 1.12 % rel., while that of arenes – by 1.03 % rel. The number of *n*-alkanes among the identified compounds prevails over that of aromatic and naphthenic hydrocarbons (Fig. 8).

The C₁₄–C₂₃ compounds have been identified among *n*-alkanes of the original and displaced oils of the Russkoye oilfield (Fig. 9).

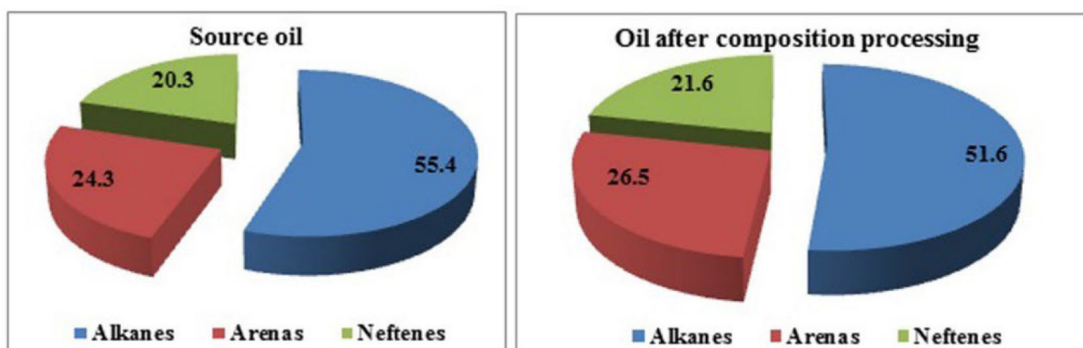


Fig. 8. Distribution of the main groups of organic compounds in the original oil from the Russkoye field and in oil treated with the acidic DES and surfactants-based oil-displacing composition, % rel.

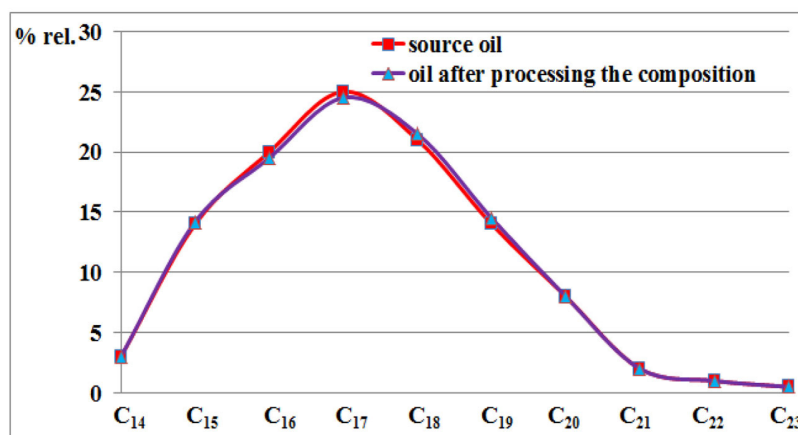


Fig. 9. Distribution of *n*-alkanes in the original oil of the Russkoye field and in oil treated with the acidic DES and surfactants based oil-displacing composition, % rel.

In both samples, low molecular weight *n*-alkanes predominate over high molecular weight ones. The maximum falls on C₁₇ *n*-alkane both in the initial sample and in that treated with acidic DES and surfactants based oil-displacing system. The composition of the original oil from the Russkoye field is similar to the composition of oil displaced using the acidic DES and surfactants based oil-displacing system, which suggests the absence of the influence of the composition on the oil.

Conclusion

Based on the results of the study of binary and ternary DES systems, an acidic DES and surfactants based oil-displacing composition with unique properties has been prepared. Its advantages are as follows: high oil-displacing ability, combined treatment of the reservoir (interaction with reservoir rock and fluids), adjustable viscosity, evolution under the reservoir conditions up to the formation of a high-capacity alkaline buffer system capable to provide optimal conditions for surfactant operation.

Treatment of a heterogeneous reservoir model with an acidic DES and surfactants based oil-displacing composition results in the smoothing of filtration flows in the model of heterogeneous reservoir and increase in its sweep efficiency. The surfactants included in the composition make it possible to increase the oil displacement efficiency due to the creation of optimal conditions for the evolution of composition. The acid components of the composition interact with the rock, which leads to the restoration of its permeability and well injectivity. The increment in the oil displacement efficiency due to the treatment of the reservoir model with the composition at 23 °C was 13.3 % for a high-permeability column and 36.3 % for a low-permeability column, while at 150 °C it was 11.3 % for a high-permeability column and 25.0 % for a low-permeability one.

As a result of the study of the composition and properties of heavy high-viscous oil before and after treatment with the acidic DES and surfactants based oil-displacing system, it was found out that its use cause no significant changes in the qualitative composition of the oil, but mainly leads to a redistribution of the content of low- and higher-molecular structures. Generally, light occluded hydrocarbons are displaced, while heavy oil components are adsorbed on the reservoir.

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