

UDC 622.276.4

DOI: 10.15372/CSD2021271

Low-Temperature Composition with Two Gel-Forming Components for Water Shutoff and Enhanced Oil Recovery

L. K. ALTUNINA, L. A. STASYEVA, V. A. KUVSHINOV, V. V. KOZLOV, I. V. KUVSHINOV

*Institute of Petroleum Chemistry, Siberian Branch of the Russian Academy of Sciences, Tomsk, Russia**E-mail: alk@ipc.tsc.ru*

(Received April 21, 2020; revised July 03, 2020)

Abstract

To enhance oil recovery and limit water inflow in the fields with a low reservoir temperature (20–23 °C), a new low-temperature nanostructured composition with two gel-forming components (polymer and inorganic) with improved rheological characteristics was developed at the Institute of Petroleum Chemistry SB RAS. Laboratory studies of gelation kinetics, rheological and filtration characteristics in the system polyvinyl alcohol (PVA) – boric acid – aluminum salt – carbamide – hexamethylene tetramine (HMTA) – polyol – water are presented. At a temperature of 20–23 °C, this system forms coherently dispersed nanoscale structures of gel-in-gel type, with viscosity 2.4–6.8 times higher and elasticity 1.4–2.3 times higher than those of the gels with only one gel-forming component. The formation of gel directly in the reservoir leads to a selective water shutoff, a change in the direction of filtration flows, a decrease in water cut, and a limitation of breakthroughs of the injected working agent into production wells. Laboratory studies demonstrated the high efficiency of the composition for flow redistribution and additional displacement of oil from the channels with very high permeability. The composition is applicable to reduce water cut and to limit water inflow in cold wells, which allows us to recommend it for use on cold producing and injection wells with complicated geological structure of the formation, with highly permeable faults, cracks and washed channels, in particular, to limit water inflow and enhance oil recovery in the Permian-Carboniferous deposits of high-viscosity oil of the Usinsk field with the natural operating mode of development. Expected result: enhancement in the oil recovery factor (ORF), a decrease in the water cut of the product, and intensification of oil production.

Keywords: enhanced oil recovery, water shutoff, gels, kinetics, rheology, oil displacement

INTRODUCTION

Efficient development of oil reserves that are difficult to recover (including highly viscous oil) and a further increase in oil production need a wide-range application of the new integrated technologies to enhance oil recovery combining the basic impact on oil deposits by water or steam injection and physicochemical methods providing an increase in

formation coverage and oil recovery factor with simultaneous intensification of production [1–7].

At the Permian-Carboniferous deposit of highly viscous oil of the Usinsk field, the tests of integrated technologies combining thermal-steam and physicochemical treatment are carried out by the LC Lukoil-Komi together with the Institute of Petroleum Chemistry SB RAS (IPC SB RAS), LC Lukoil-Inzhiniring and

LC OSK since 2003 for the purpose to increase oil recovery, and the industrial application of the developed technologies is performed [7–10], as well as the cold physicochemical technologies for an increase in oil recovery and intensification of heavy oil production under natural conditions without thermal action.

To limit water inflow and to increase oil recovery from the reservoirs with the temperature of 40–220 °C during waterflooding, thermal-steam treatment and cyclic steam injection, thermotropic gel-forming compositions were developed at the IPC SB RAS, in particular, those containing one gel-forming component – GALKA® and METKA®, and two gel-forming components – MEGA®. These compositions form gels directly in the reservoir; these gels are bound dispersed nanometer-sized structures [7] blocking water or steam inflow from injection wells to producing wells; they redistribute the filtration flows of formation fluids in an oil reservoir, which leads either to stabilization or to a decrease of water cut in the products obtained from the surrounding producing oil wells or cyclic steam wells, thus causing an increase in oil production. The field trials of the technologies involving gel-forming compositions were successfully carried out in the Permian-Carboniferous deposit of highly viscous oil in the Usinsk oilfield with cyclic steam stimulation and in the zone of the continuous steam injection. The technologies were recommended for industrial application [7–10].

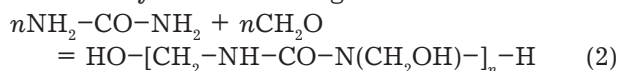
The GALKA® gel-forming composition (with one inorganic gel-forming component) is a system composed of aluminium salt, carbamide, and water [7–9]. At a temperature above 70 °C, hydrolysis of carbamide proceeds in this system, resulting in the formation of ammonia and carbon dioxide, which is accompanied by a gradual increase in solution pH. After a definite (threshold) pH value is achieved, practically instantaneous formation of aluminium hydroxide gel occurs within the entire solution volume. If this process occurs in the porous medium of oil reservoir, aluminium hydroxide gel would be able to decrease the phase permeability of the rock with respect to the liquid. To obtain a bound dispersed system at low formation temperature (20–23 °C) at pool regions not affected by thermal action, hexamethylene tetramine (HMTA) was introduced into the inorganic gel-forming system containing carbamide and aluminium salt. This additive promotes the formation of aluminium hydroxide gel at low temperatures. The sequence of gel

formation reactions in the system composed of carbamide, aluminium salt, HMTA and water at low temperature is as follows.

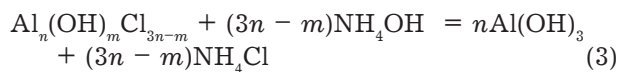
Hexamethylene tetramine ((CH₂)₆N₄) undergoes hydrolysis with the formation of formaldehyde (CH₂O) and ammonium hydroxide (NH₄OH):



Carbamide (NH₂–CO–NH₂) reacts with formaldehyde to form a soluble carbamido-formaldehyde resin having a linear structure:



As a result of formaldehyde binding by carbamide, the equilibrium of HMTA hydrolysis shifts to the right, that is, the degree of HMTA hydrolysis increases in the presence of carbamide. The formation of ammonium hydroxide increases; it reacts with the aluminium salt to form hydroxide aluminium gel and soluble ammonium chloride:



Depending on the concentration, HMTA decreases the acidity of composition solution after thermostating at 20–23 °C to the threshold pH 5–6, which leads to the formation of aluminium hydroxide gel. The soluble carbamidoformaldehyde resin increases the strength of aluminium hydroxide gel.

At the regions of the Permian-Carboniferous deposit of the Usinsk oilfield, an inorganic gel-forming composition GALKA®-NT with one gel-forming component is used under the natural mode of development (at 20–23 °C) to control filtration flows by pumping into injection wells, however, gels with improved rheological and strength characteristics are necessary to limit water inflow in producing wells. Because of this, it is urgent to develop a nanostructured composition with two gel-forming components (polymeric and inorganic), which would generate a more rigid bound dispersed nanosized gel-in-gel structure in the reservoir at 20–23 °C. Here we present the results of the development of this composition.

The goal of the work was to develop a new gel-forming nanostructured low-temperature composition (GNLC) with two gel-forming components (polymeric and inorganic) with improved rheological characteristics to enhance oil recovery and water shutoff in highly heterogeneous reservoirs of oil deposits with a temperature of 20–23 °C.

EXPERIMENTAL

To determine an optimal composition of GNLC with two gel-forming components (polymeric and inorganic), we studied the kinetics of gel formation and rheological properties of the solutions and resulting gels in the system polyvinyl alcohol (PVA) – boric acid – aluminium salt – carbamide – GNLC – polyol – water.

The reagents used in the work included aluminium salt (aluminium polyhydroxochloride AK-VA-AURAT 30 (AA-30)), PVA (13-99 grade, China), polyol (glycerol).

The kinetics of gel formation at 20–23 °C and rheological properties of different compositions of GNLC before and after thermostating were studied by means of vibrational and rotational viscosimetry using a Reokinetika vibrational viscosimeter (IPC SB RAS, Russia) with a tuning-fork sensor [11] and a HAAKE Viscotester iQ rotational viscosimeter (Thermo Scientific, USA, the measurement system of coaxial cylinders CC25 DIN/Ti) within the shear rate range of 10–1200 s⁻¹. The solutions of the gel-forming composition under investigation were placed in the thermostatic cell of the viscosimeter and thermostated at 20–23 °C under permanent measurement of solution viscosity at the shear rate of 3 s⁻¹. The time of gel formation in solutions was determined as the moment of viscosity increase due to gel formation.

The elastic modulus of gels was calculated on the basis of strain – deformation diagrams recorded in the quasi-static mode of the compression of cylindrical samples, as the tangent of the slope of the initial linear region of the dependence of strain on compression, where Hooke's law is fulfilled. The yield point of gels was determined with the help of the rotational viscosimeter (rheometer) HAAKE Viscotester iQ.

The acidity (pH) of the composition was measured using the potentiometric method with a glass electrode, with the help of a microprocessor laboratory pH meter (HANNA Instruments, Romania).

Laboratory investigation of the filtration characteristics and oil-displacing properties of different compositions of the gel-forming agent was carried out with a set-up (LC KATAKON, Russia) consisting of two parallel columns (1 and 2) 125 cm³ in volume. The models used in the work included the sand-packed reservoir models prepared from disintegrated core material of the Usinsk oilfield, the model of the reservoirs water of the Usinsk oilfield, with TDS 62.1–74.7 g/dm³, degassed oil

from the Usinsk oilfield (thermostabilized oil with the addition of 30 % kerosene). Permeability of the models was within the range of 0.447–3.343 μm², and permeability values for parallel columns differed from each other 1.02–2.84 times. Thermostating time was chosen taking into account the kinetics of gel formation in the “free volume” and was 12–24 h, counter-pressure was 2 MPa.

The efficiency of the use of different compositions as gel-forming agents was studied by replacing residual oil with water from two parallel columns with different permeability values. For this purpose, at first oil replacement with water was carried out until complete water cut the products from both columns was achieved at a temperature of 20–23 °C. Every 5–15 min we measured temperature, pressure at the column inlet and outlet, the volumes of replaced oil and water from each column. Thus obtained data were used to calculate pressure gradient (grad *p*, atm/m), filtration rate (*V*, m/day), mobility of the liquid (*k*/μ, μm²/(mPa · s), where *k* is the reservoir permeability coefficient, μ is the viscosity of the liquid) and oil recovery factor (*K_r*, %) with respect to water (OST 39-195-86 “Petroleum. Method to determine the factor of oil recovery with water under laboratory conditions”). After oil recovery with water, the slug of the gel-forming composition was pumped simultaneously in both columns, moved at the required distance using water, and thermostated for a definite time interval for the gel to be formed. Then water pumping was continued. The above-indicated parameters – temperature, pressure at the inlet and outlet, the volumes of displaced oil and water for each column – were measured permanently (every 5–15 min). The pH of liquid was also determined at the outlet of columns, along with the concentration of carbamide, a component of the composition. Using the obtained data, we calculated grad *p*, *V*, *k*/μ, absolute (total) oil recovery factor (by water, the composition, and again water), and an increment of recovery factor, which is equal to the difference of the absolute oil recovery factor and the factor of oil recovery by water.

RESULTS AND DISCUSSION

The method that has been developed at the IPC SB RAS to enhance oil recovery from highly heterogeneous low-temperature reservoirs involves regulation of filtration flows, enhancement of formation coverage by watering

with thermoreversible polymer gels formed from the solutions of polymers with the upper critical solution temperature (UCST), that is, the polymer with UCST serves as the gel-forming agent. Gel-forming compositions based on the system PVA – boric acid – glycerol – water with one polymer gel-forming component (PGC), in which gel is formed due to a reversible phase transition and complexing, were also obtained [7–9].

A new GNLC with two gel-forming components (polymeric and inorganic) with improved rheological characteristics was developed at the IPC SB RAS on the basis of laboratory studies of gel formation kinetics, rheological and filtration characteristics of the system PVA – boric acid – aluminium salt – carbamide – HMTA – polyol – water for the purpose of enhancing oil recovery and water shutoff at the deposits with low formation temperature. Gel-forming agents in the developed composition are inorganic solutions aluminium salt – carbamide – HMTA – water in which gels are formed due to hydroxopolycondensation of aluminium ions, and polymer solutions based on the system PVA – boric acid – polyol – water in which gels are formed due to reversible phase transition and complexing [7].

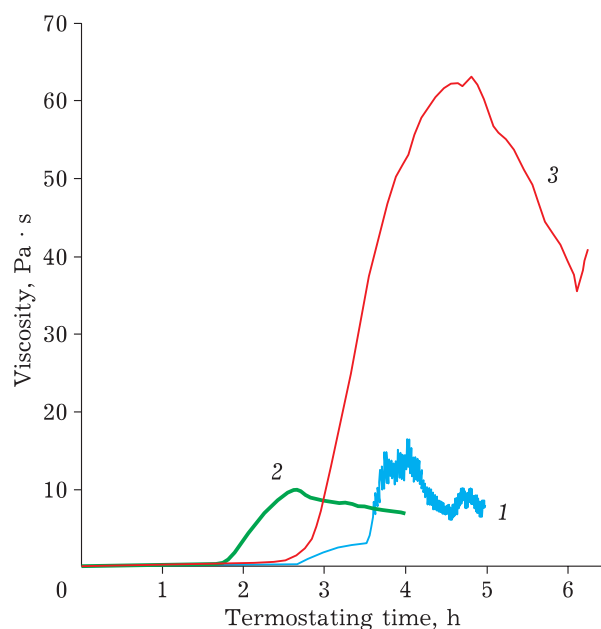


Fig. 1. Effect of PVA and boric acid on the kinetics of gel formation in the solutions of low-temperature gel-forming composition at 23 °C. Content of components in the composition (PVA – boric acid – AA-30 – carbamide – HMTA – glycerol), %: 0 – 0 – 6 – 8 – 6 – 20 (1), 3 – 0 – 6 – 8 – 6 – 20 (2), 3 – 1 – 6 – 8 – 6 – 20 (3). Here and in Fig. 2, 4: measurements were carried out by means of rotational viscosimetry at the shear rate of 3 s^{-1} .

To determine an optimal composition of GNLC with two gel-forming components (polymeric and inorganic), the effect of concentrations and component ratios on the kinetics of gel formation, physicochemical and rheological properties of solutions and resulting gels was studied.

Results of the studies of the effect of boric acid on the kinetics of gel formation in GNLC solutions show (Fig. 1) that it is necessary to add PVA together with boric acid to the inorganic gel-forming component of the composition in order to obtain the combined gel, which causes an increase in the viscosity of the resulting combined gel by a factor of 6.2–9.0 in comparison with the viscosity of the gel from the inorganic gel-forming system. The addition of PVA without boric acid does not lead to an improvement of the structural-mechanical properties of the gel (see Fig. 1).

An increase in the concentration of PVA causes an increase in the viscosity of the resulting gel (Fig. 2). With an increase in the concentrations of PVA and glycerol in the composition, the time of gel formation at 23 °C decreases (Fig. 2, 3). In addition to PVA concentration, the time of gel formation is most strongly affected by the presence of boric acid in the composition (see Fig. 3). Boric acid causes an increase in the acidity of the initial solution, so the threshold pH 5–6, at which the gel is formed, is achieved later, *i.e.* the time of gel formation increases.

Investigation of the effect of HMTA and carbamide concentrations on the kinetics of gel formation at 23 °C in the solutions of the composition, determined by means of rotational viscosimetry, showed that a decrease in HMTA concentration from 6 to 4 % caused an increase in the time of gel formation from 2.6 to 11–12 h, and viscosity decreased (Fig. 4). A similar dependence is also observed with an increase in carbamide concentration. This is connected with the fact that reaction rates in equations (1)–(3) increase with an increase in the concentrations of HMTA and carbamide, which leads to a decrease in the time of gel formation. In addition, with an increase in the concentrations of HMTA and carbamide, the equilibrium in the indicated reactions shifts to the right-hand side, to the formation of reaction products, which is depicted in an increase in gel viscosity (see Fig. 4).

The studies of rheological properties of the solutions of gel-forming composition with the varied component percentage by means of vibrational viscosimetry immediately after gel formation, 1 and 5 days later revealed that at a tem-

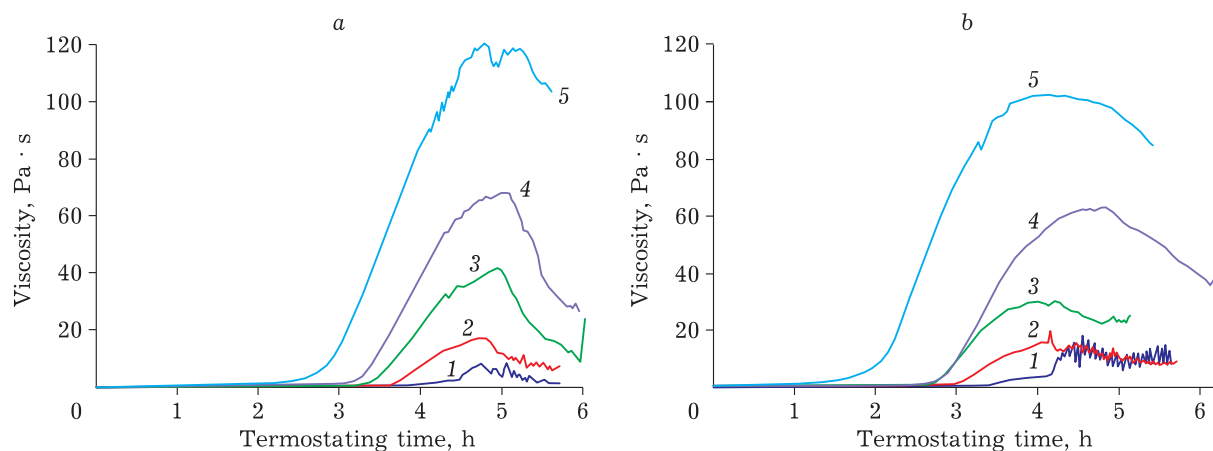


Fig. 2. Kinetics of gel formation in the solutions of composition PVA – boric acid – AA-30 – carbamide – HMTA – glycerol at 23 °C depending on PVA concentration: 0 (1), 1 (2), 2 (3), 3 (4) and 5 % (5) for glycerol concentration 0 (a) and 20 % (b). Concentrations of boric acid, AA-30, carbamide, HMTA are 1, 6, 8, 6 %, respectively. For designations, see Fig. 1.

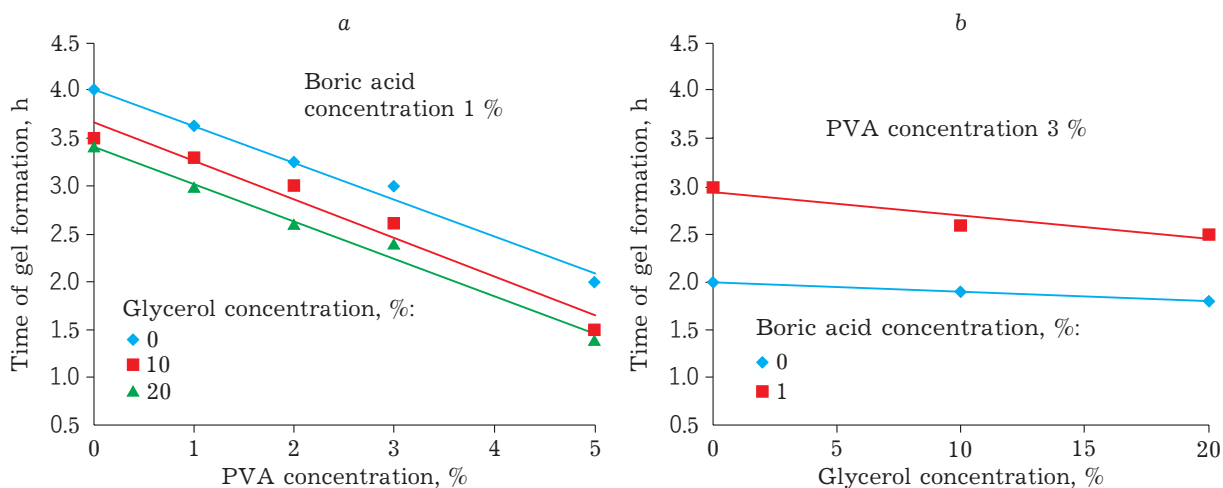


Fig. 3. Effect of the concentrations of components in the composition on the time of gel formation at 23 °C: boric acid – 1 % (a); PVA – 3 % (b).

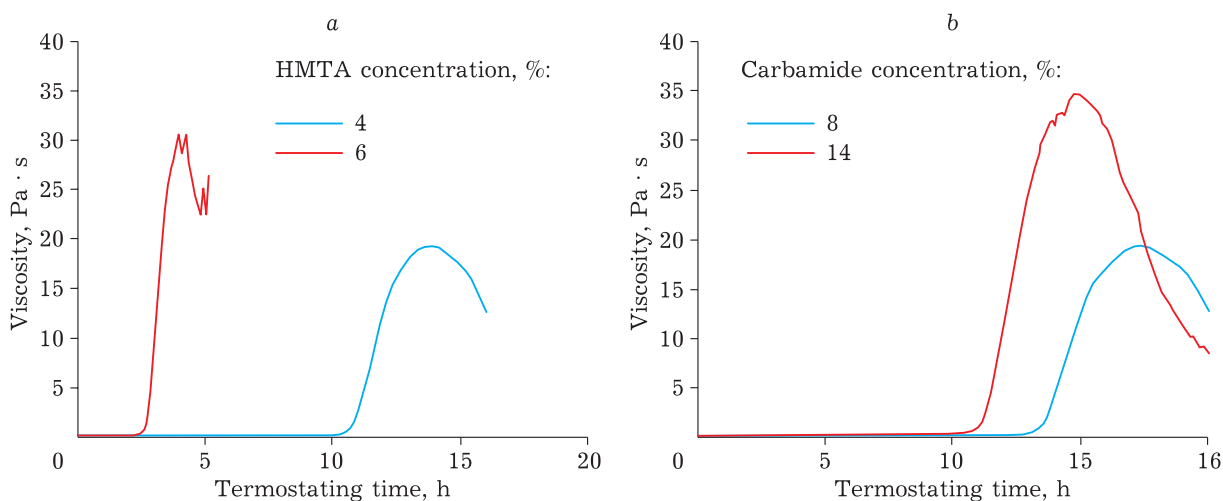


Fig. 4. Effect of the concentrations of HMTA (a) and carbamide (b) on the kinetics of gel formation at 23 °C in the solutions of composition PVA – boric acid – AA-30 – carbamide – HMTA – glycerol. The concentrations of PVA, boric acid, AA-30, glycerol are 2, 1, 6, 20 %, respectively. The concentration of carbamide is 8 % (a), HMTA 4 % (b). For designations, see Fig. 1.

perature of 20–23 °C, viscosity increases from 5.8–210 to 2215–2996, 2700–5927 and 2892–5080 mPa · s, respectively (for example, Fig. 5, *a*), that is, gels strengthen with time. The elastic modulus of the gels increases: immediately after gel formation (within 12–16 h after solution preparation) the elastic modulus of the gel was within the range 66 to 166 kPa, while after 5 days it was 140 to 315 kPa (see Fig. 5, *b* as an example).

Investigation of the elastic properties of gels obtained at 23 °C from the solutions of gel-forming compositions with different concentrations of the components revealed that the elastic modulus of the gels increases by a factor of 1.5–3 with an increase in the concentrations of PVA and glycerol, and by a factor of 1.7–2.3 as a result of the addition of boric acid into the composition.

So, experimental studies of the rheological and elastic properties of combined nanosized structures of the gel-in-gel type obtained from the gel-forming composition with two gel-forming components (polymeric and inorganic) showed that these compositions are characterized by higher viscosity (by a factor of 2.4–6.8) and elasticity (by a factor of 1.4–2.3) in comparison with the gels obtained from the compositions with one gel-forming component.

Experimental studies of the filtration characteristics and oil-displacing properties of GNLC with the models of heterogeneous reservoirs under the reservoir conditions imitating the Permian-Carboniferous deposit of the Usinsk oilfield

under the natural production regime (20–23 °C) confirm that the use of this composition causes redistribution (levelling) of filtration flows and an increase in oil recovery factor, which is more substantial in lower permeable reservoirs models – up to 50.8–52.4 % (Table 1). Redistribution (levelling) of filtration flows is evidenced by the change of the ratio of liquid mobility in columns (column 1 : column 2) for heterogeneous reservoir models maximally from 1240 : 1 to 1.0 : 1.2 (see Table 1).

Experimental results of the effect of GNLC pumping on filtration characteristics and oil recovery factor for the model of heterogeneous reservoir 2 (see Table 1) are shown in Fig. 6 as an example.

According to the results of laboratory studies, the experiments on oil displacement from two parallel columns with different permeability values (with the common inlet and separate outlets) involving the gel-forming composition revealed not only a general increase in oil recovery factor but also a connection between initial column permeability and an increment of the oil recovery factor (Fig. 7). The obtained dependence is linear, with the approximation confidence value 0.9214.

For more permeable models, an increment of oil recovery factor increases substantially, which allows one to assume the high efficiency of the composition if there are highly permeable fractures, cracks and washed-through channels. Because of this, the developed composition is intended to be applied in cold producing and injection wells with the complicated geological

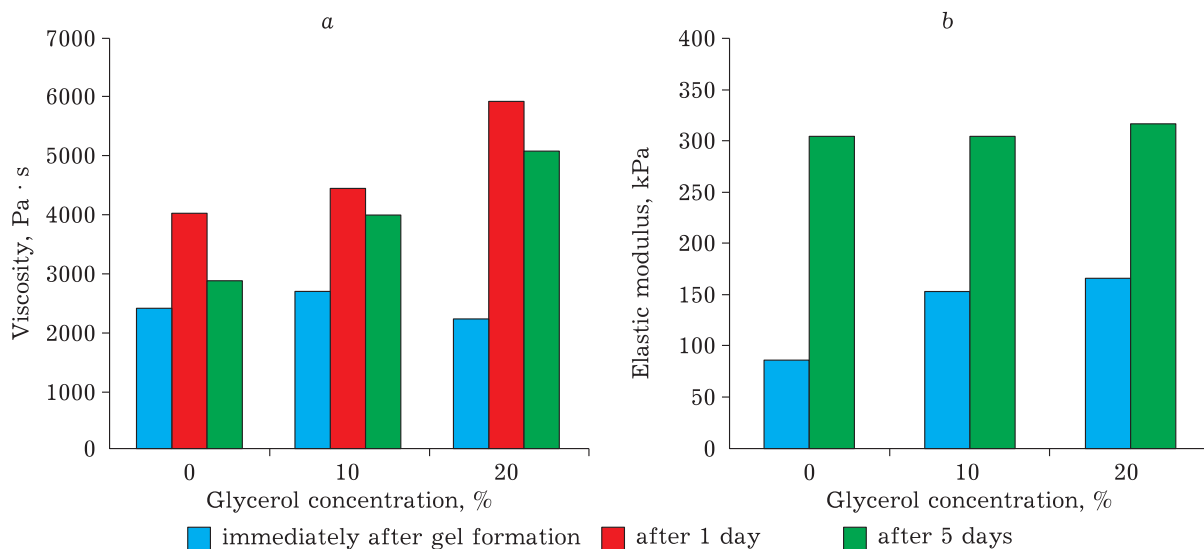


Fig. 5. Dependences of viscosity (*a*) and elastic modulus (*b*) of gels obtained from the composition PVA – boric acid – AA-30 carbamide – HMTA – glycerol with different glycerol concentrations on the time of gel formation. The concentrations of PVA, boric acid, AA-30, carbamide, HMTA are 3, 1, 6, 8, 6 %, respectively.

TABLE 1

Effect of the gel-forming composition on the characteristics of oil recovery from heterogeneous reservoir models under the conditions simulating the sheet Permian-Carboniferous deposit of the Usinsk oilfield under the natural producing conditions

Model	Gas permeability, μm^2		Oil recovery factor (by water), %		Increment of the oil recovery factor, %		Ratio of liquid mobilities (column 1 : column 2)	
	Column No.		1	2	1	2	Before injection	After injection
	1	2						
1	2.468	1.053	49.50	49.20	5.71	34.13	11.0 : 1.0	1.0 : 1.1
2	3.343	1.179	68.80	55.30	23.10	52.40	36.0 : 1.0	1.0 : 1.2
3	3.240	1.500	87.65	51.80	16.95	50.80	1240 : 1	1.0 : 1.2
4	0.844	0.447	47.10	37.70	9.72	4.27	5.2 : 1.0	3.0 : 1.0

structure of the reservoirs, in the presence of highly permeable fractures and cracks.

After pumping the composition with two gel-forming agents PVA – boric acid – aluminium salt – carbamide – HMTA – polyol – water, in the case of formation temperature 20–23 °C, at first an irreversible inorganic gel of aluminium hydroxide is formed, and then, after a definite time interval, the reversible polymer gel based on

PVA is formed inside the inorganic gel. As a result, the bound dispersed nanosized structure of gel-in-gel type is formed directly in the reservoir, the structural-mechanical properties of the gel are improved, its viscosity and elasticity are several times higher. Combined nanosized structures of the gel-in-gel type obtained from the gel-forming composition with two gel-forming agents (polymeric and inorganic) provide better ad-

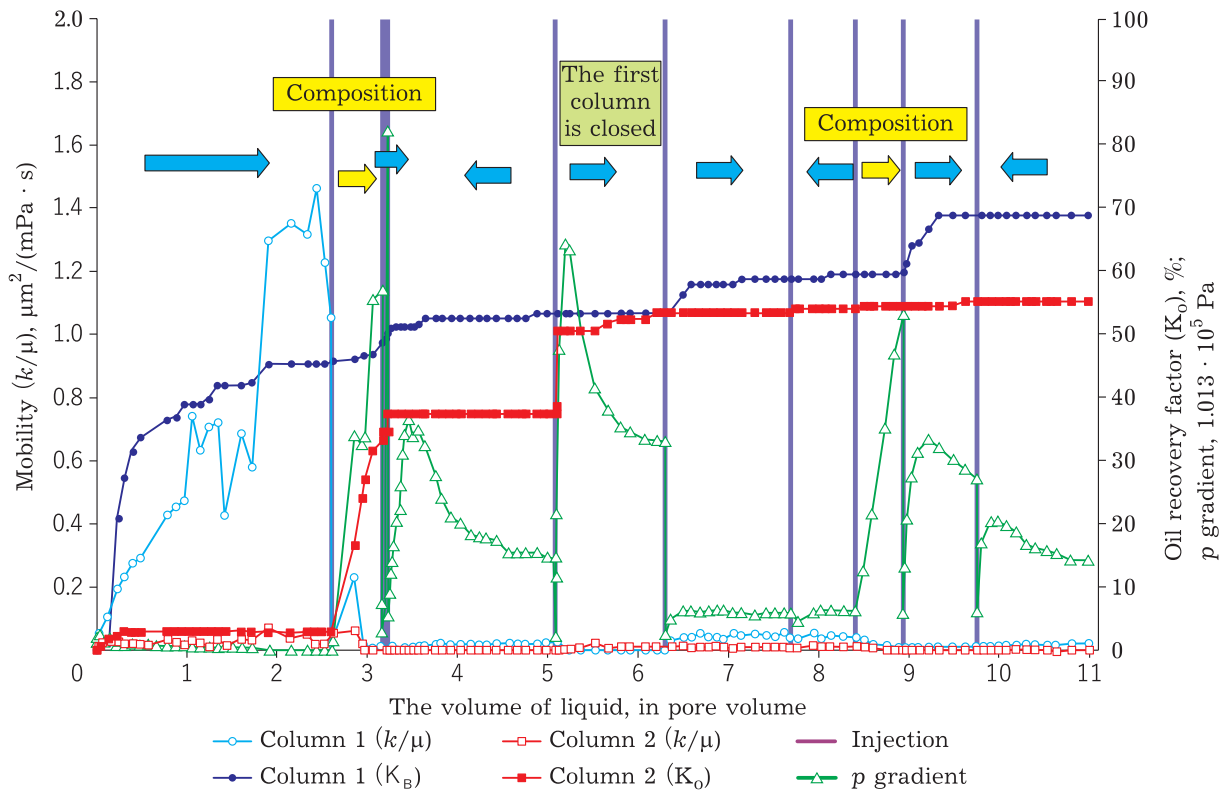


Fig. 6. Redistribution (levelling) of filtration flows and additional washing of residual oil after the injection of gel-forming low-temperature nanostructures composition in the model of the heterogeneous reservoir of Permian-Carboniferous deposit in the Usinsk oilfield from the disintegrated core material. The permeability of columns 1 and 2 is equal to 3.343 and 1.179 μm^2 , respectively. Temperature: 18 °C.

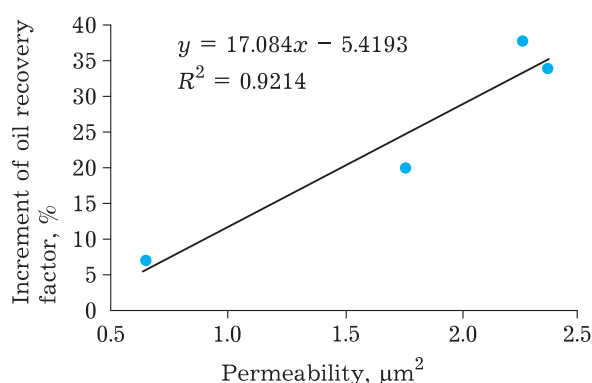


Fig. 7. Experimental dependence of the average increment of oil recovery factor for the reservoir model of the Permian-Carboniferous deposit of the Usinsk oilfield on the average permeability of models.

hesion with carbonate collector than aluminium hydroxide gels do; higher viscosity and elasticity in comparison with the gels formed from the composition with one gel-forming component. This composition is promising for the development of water-blocking barriers and screens in oil reservoir in order to enhance oil recovery and achieve water shutoff.

Gel formation directly in the reservoir leads to selective restriction of water inflow, changes in the directions of filtration flows, a decrease in water cut, limiting the breakthrough of the injected working agent into production wells. An expected result is an increase in oil recovery factor, a decrease in water cut in producing wells, and intensification of oil production.

CONCLUSION

As a result of experimental investigation of physicochemical, rheological and filtration characteristics in the system PVA – boric acid – aluminium salt – carbamide – HMTA – polyol – water, a low-temperature composition with two gel-forming components is developed. It generates a thermotropic nanostructured gel-in-gel directly in the oil reservoir, with improved rheological and structural-mechanical properties. The obtained synergic bound dispersed nanosized structure of gel-in-gel type at a temperature of 20–23 °C is characterized by increased viscosity (2.4–6.8 times) and elasticity (1.4–2.3 times) in comparison with gels containing only one gel-forming component. It is expected that gel formation directly in the reservoir will lead to selective limitation of water inflow, to changes in the directions of filtration flows, a decrease of water cut in produc-

ing wells, an increase in oil recovery coefficient and intensification of its production.

It was determined that the increment of oil recovery factor increases substantially with an increase in the permeability of the models, which allows us to anticipate the high efficiency of the use of composition in heterogeneous reservoirs containing highly permeable fractures, cracks and washed-through channels. Because of this, it is recommended to use this composition in cold producing and injection wells (20–23 °C) with the complicated geological structure of the reservoir, in the presence of highly permeable fractures and cracks.

Acknowledgements

The work was carried out within the State Assignment to the IPC SB RAS under financial support from the Ministry of Science and Higher Education of the Russian Federation.

REFERENCES

- Ruzin L. M., Morozuk O. A., Durkin S. M., Features and innovative directions for the development of highly viscous oil resources [in Russian], *Neftyanoe Khozyaystvo*, 2013, No. 8, P. 51–53.
- Romero-Zeron L., Chemical Enhanced Oil Recovery (cEOR), A Practical Overview, *InTech*, 2016. 200 p.
- Wang Y., Hou J., Song Z., Yuan D., Zhang J., Zhao T., A case study on simulation of *in-situ* CO₂ huff-‘n’-puff process, *SPE Res. Eval. & Eng.*, 2018, Vol. 21, No. 01, P. 109–121.
- Hascakir B., Introduction to thermal enhanced oil recovery (EOR) special issue, *Journal of Petroleum Science and Engineering*, 2017, Vol. 154, P. 438–441.
- Sheng J. J., *Modern Chemical Enhanced Oil Recovery*, Gulf Publishing, 2011. 617 p.
- Xiaohu Donga, Huiqing Liua, Zhangxin Chena, Keliu Wua, Ning Lua, Qichen Zhanga., Enhanced oil recovery techniques for heavy oil and oilsands reservoirs after steam injection, *Applied Energy*, 2019, Vol. 239, Issue C, P. 1190–1211.
- Altunina L. K., Kuvshinov V. A., Stasyeva L. A., Kuvshinov I. V., Trends and outlooks for the development of physicochemical methods to enhance oil recovery from heavy oil deposits, *Chemistry for Sustainable Development*, 2018, Vol. 26, No. 3, P. 261–277.
- Altunina L. K., Kuvshinov V. A., Stasyeva L. A., Chertenkov M. V., Shkrabyuk L. S., Andreev D. V., Physicochemical and integrated technologies for enhancement of oil recovery from the Permian-Carboniferous deposit of highly viscous oil from the Usinsk oilfield [in Russian], *Neftyanoe Khozyaystvo*, 2017, No. 7., P. 26–29.
- Altunina L. K., Kuvshinov V. A., Kuvshinov I. V., Chertenkov M. V., Ursegov S. O., Pilot tests of new EOR technologies for heavy oil reservoirs, *Proceedings of SPE Russian Petroleum Conference*, Moscow, October 26–28, 2015, Paper 176703-MS.
- Kuvshinov I. V., Altunina L. K., Kuvshinov V. A., Combined wells treatment with various chemical compositions and thermal methods [in Russian], *Zhurn. SFU. Khimiya*, 2019, Vol. 12, No. 4, P. 473–482.
- RF Pat. RU 1229647 G 01, 2016.