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Enhanced Oil Recovery in Carbonate Reservoirs of the Volga-Ural Petroleum Province: Challenges and Solutions

L.E. Lenchenkova[1], S.A. Vezhnin[2], F.E. Safarov[2,3], I.P. Novikov[4], R.N. Yakubov[1], M.A. Varfolomeev[5], S.A. Nazarychev[5], A.O. Malakhov[5], R.R. Asadullin[1], T.F. Khaernasov[1], A.S. Pavlik[1], A.E. Fetisov[1], A.G. Telin[2]*
[1]*Ufa State Petroleum Technological University, Ufa, Russian Federation*
[2]*Ufa Scientific and Technical Center, Ufa, Russian Federation*
[3]*Ufa Institute of Chemistry of the Ufa Federal Research Center of the Russian Academy of Sciences, Ufa, Russian Federation*
[4]*Nafta-Service LLC, Almetyevsk, Russian Federation*
[5]*Kazan Federal University, Kazan, Russian Federation*

Abstract. The development challenges of carbonate reservoirs in the Volga-Ural petroleum province are associated with their geological features (presence of fracturing, high formation water salinity, high oil viscosity, hydrophobic rock surface properties). While global practice employs well-established gas and physicochemical methods, Russia practically doesn't apply conventional EOR techniques in carbonate reservoirs. The article emphasizes that the main methods of carbonate formation stimulation in the Volga-Ural petroleum province include: injection profile conformance technologies (small-scale EOR) as well as production well stimulation through acid treatments. A comparative analysis of literature is provided, reflecting the main types of implemented geological and technical measures. The authors describe in detail their developed individual elements of systemic technology adapted for carbonate reservoirs, optimized for each type of impact based on the industry's achieved technical level – thereby demonstrating that all geological and technical measures for carbonate reservoirs have been refined and their effectiveness proven. The systematic approach to injection and production well treatments requires multiple expansions and replication, which should lead to profitable reserve recovery in carbonate reservoirs when applying best practices of geological and technical operations.

Keywords: systematic technology for carbonate reservoirs, flow-diverting technologies, injection profile equalization, small-scale EOR methods, polymer-disperse systems, gel-sediment-forming technologies, acid treatments with diverters, polyacrylate reagents

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Introduction

Development of carbonate formations of the Volga-Ural Oil Province is associated with certain difficulties related to their geological features. These include the presence of fracturing, high formation water salinity, high oil viscosity, and hydrophobic properties of the rock surface (Khissamutdinov, Astakhova, 2024). In global practice, proven gas and physiochemical methods are used to enhance oil recovery in carbonate reservoirs. For example, in the United States, the ratio of gas-based and chemical EOR methods for carbonate and terrigenous (clastic) reservoirs is almost the same. Thus, the most widespread methods are carbon dioxide flooding, high-pressure gas injection (primarily in Alaskan fields), as well as high-pressure air injection (known in Russian terminology as the thermogas method). Much less commonly used are surfactant flooding, polymer flooding, and alkaline-surfactant-polymer (ASP) flooding (Manrique et al., 2013). At the same time, enormous efforts are being expended in various countries around the world on optimizing chemical EOR methods

*Corresponding author: Aleksey G. Telin
e-mail: telinag@ufntc.ru

in carbonates (Yu-Qi et al., 2020). There are publications on the use of nanosilica with sodium dodecyl sulfate in experiments on physical models of carbonate reservoirs, in which the incremental displacement factor amounted to 15.1% when using nanosilica at a concentration of 0.03% and sodium dodecyl sulfate at 0.16% (Ali K. Alhurishawi et al., 2019). Relatively few field trials of conventional EOR methods are carefully analyzed and summarized with recommendations for expanding pilot field studies (Sreela et al., 2018; Sheng, 2013). Among the relatively young "EOR methods", growing interest in low-salinity water flooding should be noted, particularly in reservoirs with a pronounced block structure, with research in this direction continuing (Mohammad et al., 2018).

For a number of reasons, conventional EOR methods (polymer flooding, surfactant flooding, and gas injection) are practically not used in carbonate reservoirs in Russia. We will not analyze this circumstance within the framework of this paper; we will merely emphasize that the constraining factors are high capital and operating costs, as well as the absence of government preferences. In all fairness, it should be noted that a successful water-alternating-gas (WAG) injection project has been implemented at the Alekseevskoye field (subsoil user – Aloil, a small oil company, Republic of Tatarstan, Volga-Ural Oil Province). Injection is carried out into the fractured-vuggy (fractured-porous) carbonate reservoirs of the Kizelovsky horizon using a pump-booster unit manufactured by the Sinergia plant. Water-alternating-gas injection is applied to the near-edge parts of the reservoir with ten injection wells, under the influence of which 44 production wells are located. Offtake compensation is maintained at 150%. This project is profitable and, in addition to net profit, allows for the efficient utilization of associated petroleum gas (Muslimov et al., 2004; Vafin, 2008).

The main methods of reservoir stimulation in carbonate reservoirs of the Volga-Ural Oil Province are injectivity profile control (IPC) technologies, which according to the Russian classification are classified as low-volume EOR methods (Zemtsov, Mazaev, 2021), as well as production well stimulation by acid treatment (Kharisov et al., 2012). Among low-volume flow-diverting technologies, the most widespread are various modifications of well injectivity profile control using polymer-disperse systems and gel- and precipitate-forming technologies (Gazizov, 2002). The paper (Gafarov, 2005) describes the successful application of a combined reservoir stimulation technology: first a polymer-disperse system is injected, followed by a surfactant slug. It should be noted that this approach requires wider adoption. In the works of two research groups (Safarov et al., 2020; Ganiev et al., 2020), well treatments were carried out using the same methodology: first, tracer studies were conducted to identify the main water breakthrough pathways, followed by injection of gel- and precipitate-forming compositions based on acrylic polymers, which form a plugging barrier upon interaction with hardness salts present in the formation water. Also, in fractured reservoirs, well treatments using preformed gel particles have performed well (Ganiev et al., 2023; Rozhkova, 2021).

All of the noted technologies are successfully applied in various fields of the Volga-Ural Oil Province. Only one drawback of this approach can be pointed out – its lack of systematicity, which is associated with the fact that, as a rule, individual wells are treated in isolation from the field development concept. It should be noted that in the Soviet Union and in Russia, for terrigenous reservoirs of Western Siberia, the efforts of A.T. Gorbunov and A.N. Petrakov formulated and implemented a systematic technology of areal reservoir stimulation, encompassing such types of geological and engineering operations (GEOs) as IPC, water shut-off, and production well stimulation (Gumersky et al., 2000). For carbonate reservoirs, this approach, convincingly presented in the works (Raspopov, Zhigalov, 2023; Raspopov et al., 2016), is an effective tool for improving the efficiency of reservoir development under complex mining and geological conditions. The methodology adopted by A.V. Raspopov for the recovery of residual reserves is classical: based on development analysis and hydrodynamic simulation, recoverable reserves are localized, and a set of GEOs for their selective recovery is determined; moreover, the paper (Raspopov et al., 2016) examines physiochemical methods in combination with hydraulic fracturing and acid fracturing, together with sidetracking and drilling of horizontal wells. All these methods can significantly increase oil production, slow down the rate of water cut increase, and generally enhance the economic attractiveness of implementing systematic technology, as convincingly demonstrated on the example of the Bashkirian-Serpukhovian deposits of one of the fields in the Upper Kama region (Raspopov et al., 2016). Unfortunately, in the majority of carbonate targets, such an approach is absent. In fact, this logic is already partially implemented as one of the mandatory sections in field development and technological design documents submitted for expert review to the Central Commission for Oil Field Development. Currently, it is very important for design organizations to prepare technological design documents using best practices for oil production stimulation and water production limitation. In this case, the idea of economic feasibility should itself push subsoil users toward implementing systematic enhanced oil recovery technology.

The purpose of this work is to substantiate the effectiveness of GEOs for oil production stimulation and water influx limitation in carbonate reservoirs of the Ural-Volga region.

Materials and Methods

Determination of water-oil interfacial tension using surfactants

To conduct the experiment on determining interfacial tension for surfactant solutions, the spinning drop method was chosen, which allows measurements to be performed over a wide range of values, an important condition when analyzing the efficiency of surfactants that significantly reduce interfacial tension values relative to the initial level. The measurements were carried out using a SITE100 tensiometer (KRUSS GmbH, Germany).

This method is based on the rotation of a droplet under the action of centrifugal force. A droplet of the light phase (oil) with density ρ_1 is introduced via a capillary into the heavy phase (water from the reservoir pressure maintenance (RPM) system, surfactant solution) with density ρ_2, which is contained in a measuring cell. The cell rotates around its axis at a specified angular velocity ω. In this process, the molecules of the interfacial layer are subjected to centrifugal forces directed away from the rotation axis. At a certain rotation speed ω, the interfacial tension forces become equal to the centrifugal force, and the molecules in the interfacial layer begin to move along a specific trajectory with radius R. As a result, the droplet elongates along the rotation axis and, under equilibrium of forces, assumes a cylindrical shape (Fig. 1).

Using the difference between the density of the heavy phase and the density of the light phase, the droplet rotation radius (R), and the rotation speed (ω), the interfacial tension can be determined (k is the instrument constant, which depends on the optical magnification):

$$\sigma = k\, R^3\, \omega^2\, (\rho_2 - \rho_1).$$

Determination of Contact Angle (CA)

During the flow of a displacing fluid in a porous medium, the wetting ability has a significant influence on the recovery degree of capillary-trapped oil. Displacing oil from a hydrophobic reservoir requires higher pressure differentials or a greater reduction in surface tension than for a hydrophilic reservoir. A change in solid surface wettability from hydrophobic to hydrophilic due to surfactant action can promote improved detachment of oil films and droplets, increase their mobility, and activate capillary imbibition.

In common practice, the main criterion for evaluating the wetting ability of surfactants is their ability to reduce the contact angle (CA). Experiments to determine the contact angle were carried out using a Dataphysics OCA 15EC instrument (DataPhysics Instruments GmbH, Germany) (Fig. 2), which allows measurements to be performed with simultaneous photo-recording and analysis of the droplet contour shape.

Determination of Porosity and Permeability

Porosity and permeability were determined using a Plast-215ATM gas porosimeter-permeameter (Argosy Technologies Ltd., Russia) (Fig. 3).

Absolute air permeability measurements were carried out in accordance with GOST 26450.2-85, while open porosity measurements were conducted in accordance with GOST 26450.1-85.

Method for Determining Displacement Efficiency by Capillary Imbibition in Amott Cells

Core samples were prepared according to GOST 26450.0-85. Porosity and permeability were determined by the gas volumetric method according to GOST 26450.2-85. Cores were saturated with formation water or a model formation water under vacuum according

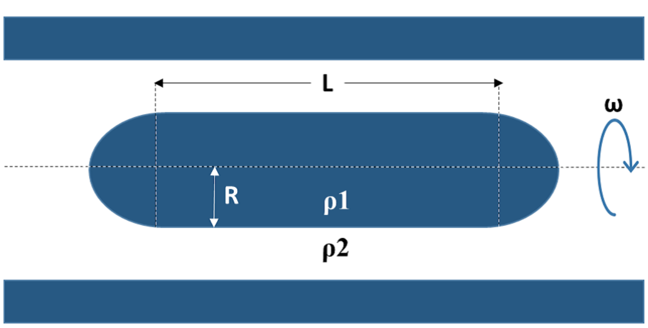

Fig. 1. Determination of interfacial tension by the spinning drop method

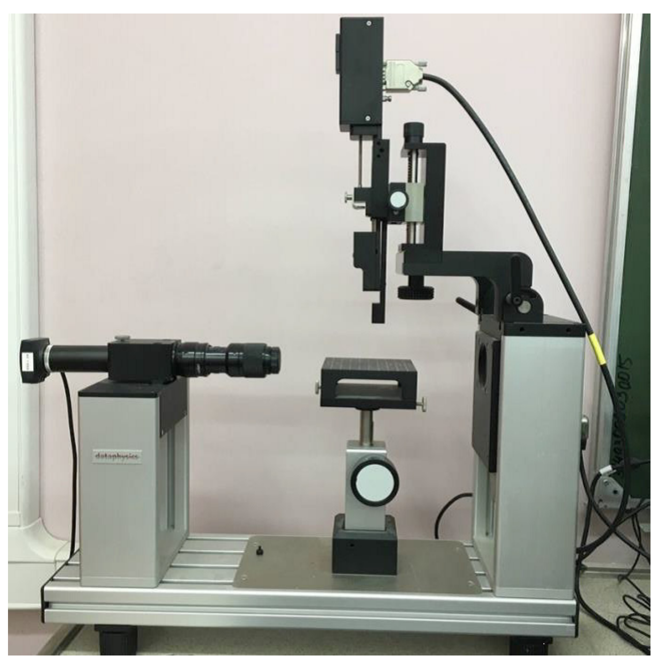

Fig. 2. External view of the Dataphysics OCA 15ES instrument

Fig. 3. Plast-215ATM gas porosimeter-permeameter

to GOST 26450.1-85. Residual water saturation was established by centrifugation, after which the cores were saturated with kerosene under vacuum. Initial oil saturation was created using an M-1 filtration unit (KFU, Russia) at reservoir temperature and pressure. To determine the initial oil saturation value, the samples were weighed. They were then placed in a container with oil under an oxygen-free atmosphere in a temperature-controlled oven for two weeks for aging.

Prior to being placed in the Amott cells, the oil-saturated cores were rolled and subjected to control weighing. The core was placed in the cell, and the surfactant solution, preheated to reservoir temperature, was poured into the cell.

The Amott cells containing the core immersed in the surfactant solution (or formation water) were stored in a temperature-controlled oven at reservoir temperature for 30 days. The volume of displaced oil was recorded at specific time intervals (2 hours, 4 hours, 8 hours, 12 hours, 1 day, and thereafter – every day).

The oil displacement efficiency (K_{dis}) was determined as the ratio of the sum of the volume of recovered oil (V_o) and the volume of recovered microemulsion (V_e) multiplied by the oil content coefficient in the microemulsion (C_{o_e}), to the initial oil volume in the sample ($V_{o.ini}$):

$$K_{dis} = \frac{V_o + V_e \cdot C_{o\_e}}{V_{o.ini}}.$$

Methodology of Coreflood Experiments for Oil Displacement

Coreflood experiments were conducted on an M-1 unit (Fig. 4) in accordance with OST 39-195-86.

In the experiments, prepared samples of formation water and oil were used. Cores with residual water saturation that had been saturated with kerosene under vacuum were stored fully immersed in kerosene prior to the experiment.

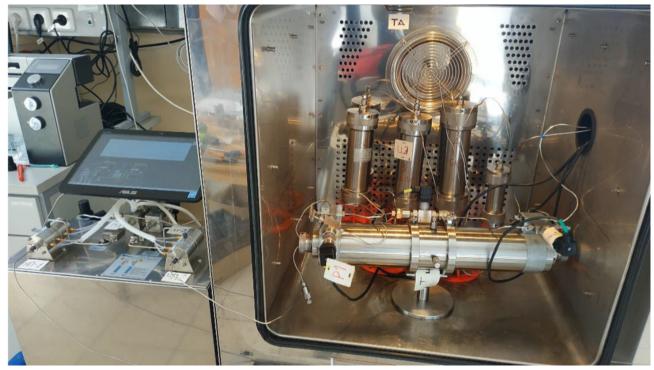

Fig. 4. General view of the M-1 flow unit

A schematic diagram of the flow unit is presented in Fig. 5.

The first stage of the flow experiment consisted of preparing the composite core model and fluids. The composite core model was placed in the core holder of the M-1 unit, resaturated with kerosene, and kept in the climatic chamber for no less than two hours at reservoir temperature and pressure. After this time, oil was filtered through the composite porous medium (no less than three pore volumes) at a filtration rate not exceeding 5 m/day (calculated rate of 0.2 mL/min). The saturated composite model was held at reservoir temperature and pressure for 16 hours to complete adsorption processes and restore wettability.

Second stage of the experiment: oil displacement from the sample by RPM water, determination of the oil displacement efficiency (K_{dis}) and post-displacement efficiency $K_{post\text{-}dis}$). The process of oil displacement by RPM water was carried out until the complete absence of oil at the core holder outlet (full watercut), but for no less than five pore volumes, with determination of K_{dis} and model permeability. Thereafter, a surfactant slug of 0.3 pore volume was injected, followed by RPM water flooding until full watercut of the effluent fluid, but for no less than five pore volumes, with determination of Kpost-dis and the change in model permeability.

The oil displacement and post-displacement efficiencies were calculated using the following formulas:

$$K_{dis} = V_{o.dis}/V_{o.ini},$$

$$K_{post\text{-}dis} = V_{o.post\text{-}dis}/V_{o.ini},$$

where K_{dis} and $K_{post\text{-}dis}$ are the oil displacement and post-displacement efficiencies by water, respectively; $V_{o.dis}$ and $V_{o.post\text{-}dis}$ are the oil volumes in the displacement and post-displacement experiments; Vo.ini is the volume of oil initially contained in the sample.

Method for Preparing Polymer-Gel Fibrous Composites

The base hydrogel was prepared from partially hydrolyzed polyacrylamide (1.7%) and a complex organic

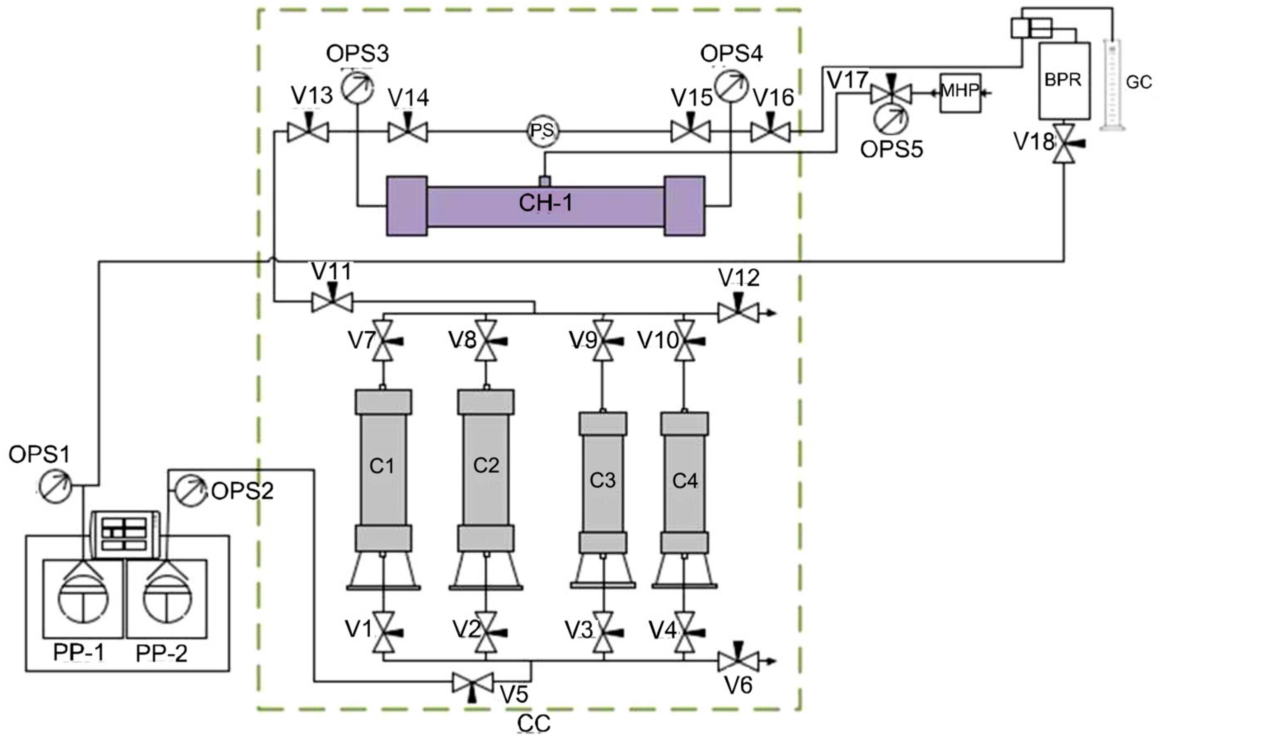

Fig. 5. Schematic diagram of the M-1 flow unit: PP-1, PP-2 – high-pressure plunger pumps; V1 – V18 – needle valves; C1 – C4 – piston-type fluid containers; OPS1 – OPS5 – overpressure sensors; PS – compact differential pressure sensor; CH-1 – core holder; CC – climatic chamber; GC – graduated cylinder; MHP – manual hydraulic pump; BPR – back pressure regulator

crosslinker (Mullagalin et al., 2015). Polypropylene fiber (Atren-Fibre), TU 2458-029-63121839-2011 (MKO LLC), with a length of 4 to 6 mm, was used as a fibrous filler; chrysotile, GOST 12871-2013 (Uralasbest PJSC), was used as a dispersed filler.

The preparation of the gel-forming compositions was carried out in fresh water using a magnetic stirrer: the dispersed and fibrous fillers were dissolved for 10–15 minutes, then the polymer and crosslinker were added. The resulting composition was stirred for 45 minutes or until complete dissolution of the polymer. After 16 hours, rheological and flow studies were conducted.

Method for Determining Yield Stress

Yield stress was determined using a Haake Viscotester iQ rotational viscometer (Thermo Fisher Scientific, USA) based on the shear rate versus shear stress relationship. To this end, rheological curves were recorded in controlled stress (CS) mode. A 4 mL aliquot of the gel was placed into a CC16 Din/Ti measuring cylinder; this cylinder was then placed into the temperature-controlled compartment of the rotational viscometer, the rotor was lowered, and the measurement was started. Upon reaching the yield stress, a sharp jump in shear rate occurs, and the measurement stops at that point. After the measurement was completed, data processing was performed, with the corresponding shear rate–shear stress curve being plotted. The yield stress value was determined from the sharp inflection point on this curve (Schramm, 2003).

Method for Determining Elastic Modulus, Viscous Modulus, and Complex Modulus

Oscillatory measurements were performed using a Rheotest RN 5.1 rotational viscometer (RHEOTEST Medingen GmbH, Germany) with a plate-plate measuring system at a temperature of 24 °C. The diameter of the measuring plate was D = 36 mm, and the gap between the plates was h = 1 mm (Fig. 6).

The required volume of hydrogel was applied to the plate using a syringe dispenser, and then a gap of 1 mm between the plates was set using a micrometer. Excess

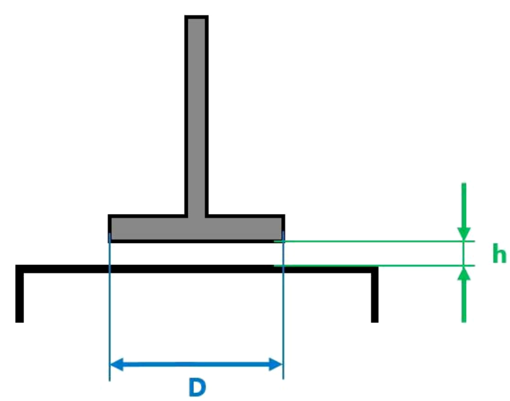

Fig. 6. Plate-plate measuring system

hydrogel was removed with special tweezers to ensure that the space between the plates was completely filled with the measured fluid.

Oscillatory measurements were performed with a shear stress τ sweep at an oscillation frequency ν of 1 Hz. The main measured parameters were the elastic modulus G’, the viscous modulus G’’, the crossover point (intersection point of G’ and G’’), which corresponds to the yield stress, as well as the linear viscoelastic range. Several measurements were taken during the experiments; the results were then averaged, and the standard deviation was calculated (Schramm, 2003).

Coreflood Testing on an Idealized Fracture Model

The studies were performed on a SMP FES-2R core flow testing unit (Kortech, Russia), the technical specifications of which are given in Table 1.

Core sample preparation and flow studies were carried out according to the requirements of OST 39-195-86. In creating the idealized fracture model (slit model – Fig. 7), natural core samples were used, which made it possible to satisfactorily reproduce the conditions of natural wettability.

Core samples were split lengthwise, and then the halves were matched so that the slit model had a cylindrical shape. After grinding the contacting surfaces of the slit model, foil strips of a specified thickness were attached onto one of the halves (to create a specified fracture aperture). The parameters of the manufactured idealized fracture model were as follows: length 11.2 cm; width 1.7 cm; orientation in space – horizontal.

Before each experiment, the surface of the slit model was carefully prepared, namely: it was cleaned of contaminants and washed with water and an alcohol solution. The idealized fracture model was placed into the core holder of the flow unit.

At the first stage of the experiment, water flow was carried out in the forward direction in a volume of at least 10 cm³ until the pressure gradient stabilized. Then, oil flow was carried out in the forward direction (at least 10 cm³) until the pressure gradient stabilized. At the next stage, water flow was also carried out in the forward direction at a specified constant flow rate, with a volume of at least 10 cm³, until the pressure gradient stabilized, and the water permeability was determined during this process. Thereafter, a plugging composition

No.	Parameter	Value
1	Linear length of the core model	100-300 mm
2	Core temperature control range	(+25) – (+150) °C
3	Maximum overburden pressure	70 MPa
4	Maximum reservoir pressure	55 MPa

Table 1. Technical specifications of the SMP FES-2R unit

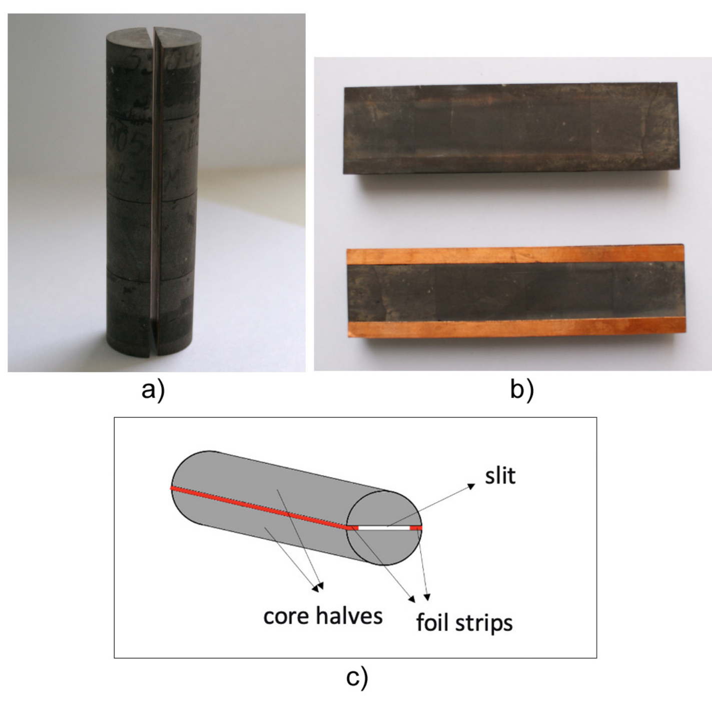

Fig. 7. Photograph of the idealized fracture model (a – photograph of the split core; b – photograph of the split core halves with attached foil strips; c – schematic of the idealized fracture model)

was injected into the idealized fracture model in the direction opposite to the initial one, in a volume not exceeding 10 cm³. After this, the system was allowed to statically soak for no less than 24 hours.

At the next stage, water was injected in the forward direction at a specified constant flow rate, with a volume of at least 10 cm³, until the pressure gradient stabilized, during which the water permeability and the maximum pressure gradient were determined. As a result, the residual resistance factor (RRF) was calculated – the ratio of the water/gas pressure differential after injection of the composition to the pressure differential before reagent treatment:

$$RRF = \frac{dP_i}{dP_1}$$

where dP_i is the fluid pressure differential after treatment at the corresponding flow stage; dP_1 is the fluid pressure differential before treatment with the composition.

Method for Preparing the Acid Composition

The acid composition was prepared from 12% hydrochloric acid, oleic acid, and neonol grade AF_9-12. The following were used: chemically pure 36% hydrochloric acid (AO Reakhim LLC, Russia), which was subsequently diluted to 12%; oleic acid (TU 6-09-5290-86); tall oil fatty acids (GOST 14845-79); and neonol grade AF_9-12 (TU 2483-077-05766801-98).

The acid composition was prepared by mixing all components in a 100 mL conical flask, followed by stirring with a paddle stirrer for 40 minutes until a direct emulsion was formed (Fig. 8).

Kinetic Studies

The determination of the reaction rate of carbonate rock with acid compositions based on studying the kinetics of CO_2 evolution under static conditions was carried out using a PIK-OSG volumetric unit (Geologika LLC, Russia), the external appearance of which is shown in Fig. 9.

During sample preparation, the core material was crushed and mechanically disintegrated in a mortar. Then, using a PGL-12 press (Labtools LLC, Russia), 2 g "tablets" were produced (Fig. 10). The disintegrated core was held under a pressure of 20–25 MPa for 5 minutes.

The "tablets" (rock samples) were placed into the reactor of the volumetric unit and covered with the tested acid composition (7.5 mL of acid solution per 1 g of carbonate rock) under ambient conditions. The volume of evolved CO_2 was recorded by a gas trap, and the data were automatically displayed on the software interface as a graph and a table.

Studies to determine the reaction rate of carbonate rock with acid compositions under dynamic conditions, based on measuring the amount of dissolved calcium in sampled aliquots of the acid composition, were conducted using a PIK-OSG rotating disk unit (Geologika LLC, Russia). The external view of the unit is shown in Fig. 11.

Sampling of the spent acid in a volume of 5 mL was performed at 0.5, 1, 1.5, 2, 3, 5, 7, 10, 15, 20, 25, 30, 40, 50, and 60 minutes after the immersion of the core sample into the acid. The obtained samples were titrated to determine their calcium ion content (titrant – Trilon B, indicator – murexide).

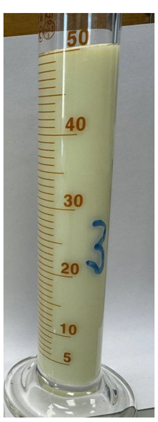

Fig. 8. External appearance of the acid composition after one hour of settling

Fig. 9. External view of the PIK-OSG volumetric unit

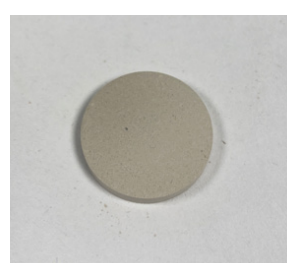

Fig. 10. A "tablet" of a carbonate rock sample

Fig. 11. External view of the rotating disk unit

Based on the dynamics of changes in calcium ion concentration, graphs were plotted, from which the reaction rate was determined.

Results

Let us consider in more detail the individual elements of the systematic technology as applied to carbonate reservoirs that we have developed, and for each type of treatment we have attempted to optimize the stimulation method, relying on the technical level achieved in the industry as well as on our own developments.

Since reservoirs of the fractured-vuggy (fractured-porous) type predominate in the section of carbonate development targets, IPC technologies based on gel- and precipitate-forming reagents that effectively reduce fracture permeability have proven most successful. From the standpoint of availability, cost, and processability, hydrolyzed polyacrylonitrile (PAN fiber) appears to be the most attractive reagent. Moreover, upon interaction with hardness salts present in formation water, it forms a gel-precipitate barrier, which is very convenient for field operations. Polyacrylate reagents, known under such trade names as Givpan, Geopan, NGT-Chem-2, etc., have been repeatedly tested in fields of Russia, the Republic of Kazakhstan, and the Republic of Belarus, consistently demonstrating high efficiency. Exceptionally important technological properties of reagents for IPC are phase selectivity and permeability selectivity. In the ideal case, the higher the permeability, the higher the RRF should be. However, for polyacrylate reagents, the RRF smoothly decreases with increasing permeability (Fig. 12).

An ideal dependence of the RRF on permeability is demonstrated by crosslinked polymer compositions (Telin et al., 2023), polymer-disperse compositions, and colloidal-disperse compositions (Gazizov et al., 1998; Lozin, Khlebnikov, 2003). We hypothesized that modifying polyacrylamide with functional groups that ensure the formation of a gel-precipitate upon interaction with di- and trivalent cations would impart to the reagent the properties required for selective reduction of fracture permeability. Guided by this concept, we developed the reagent NGT-Chem-6 (Karatseev et al., 2018), which exhibits the desired properties (Fig. 13).

Based on previously obtained experimental results (Safarov et al., 2017), in this work we tested the combination of a non-selective gel- and precipitate-forming reagent NGT-Chem-2 and a selective reagent NGT-Chem-6 in a fractured-porous reservoir. Well 820 (D_4, D_{4-0}) and well 856 (D_3) of the Garshinskoye field (Orenburg Oblast, Volga-Ural Oil Province) were selected as the targets for pilot field studies. Tracer studies confirmed good hydrodynamic connectivity between the injection wells and the responding production wells. Into well 820, 9.6 tonnes of NGT-Chem-2 and 11.8 tonnes of NGT-Chem-6, as well as 21.6 tonnes of crosslinker – aluminum oxychloride – were injected in batches. Into well 856, 52.2 tonnes of NGT-Chem-6 and 21.6 tonnes

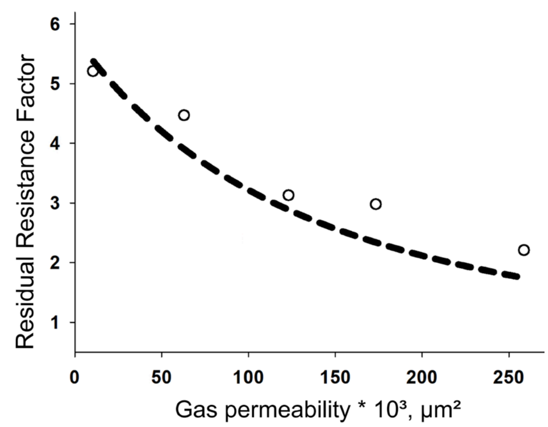

Fig. 12. Dependence of RRF for the reagent NGT-Chem-2, crosslinked with aluminum oxychloride mixed with lignosulfonate, on the gas permeability of a pore model with residual oil saturation (Safarov et al., 2017)

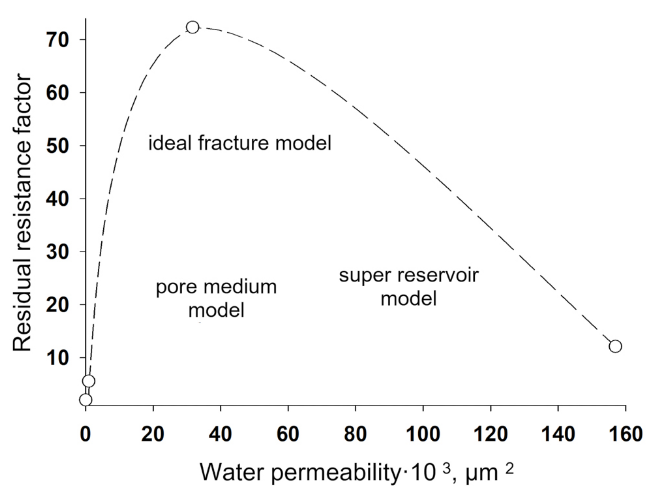

Fig. 13. Dependence of the RRF for the reagent NGT-Chem-6, crosslinked with aluminum oxychloride, on water permeability (Safarov et al., 2017)

of aluminum oxychloride were injected. As a result of the treatment, in the responding production wells within the influence zone of injection well 820, 1,190 tonnes of incremental oil were produced, and within the zone of well 856, 1,690 tonnes were produced. A comparison of the injectivity profiles of well 820 before and after treatment confirmed the redistribution of injected water flows (Fig. 14), with the main water-receiving interval being reliably isolated with only a minor reduction in injectivity.

Consequently, as a result of the treatment, the injectivity of low-injectivity layers increased: D_4^0 – from 11% to 27.2%, D_4^1 – from 0.3% to 16.2%, D_4^2 – from 16.9% to 56.6%; whereas the lower swept sublayer D_4^3 was completely isolated by the reagent.

It should be noted that a comparison of the treatment efficiency for the two injection wells showed that the injection of NGT-Chem-6 alone yielded a greater incremental oil production than the combined injection of NGT-Chem-2 and NGT-Chem-6. This is attributed to the molecular weight of the polyacrylate polymer (NGT-Chem-2) versus the polyacrylamide polymer (NGT-Chem-6): due to the higher molecular weight of the polyacrylamide, greater selectivity with respect to permeability and pore structure is achieved upon treatment with NGT-Chem-6, which is associated with the viscoelastic properties of the gel-precipitate.

Surfactant Slug Injection

Surfactant slug injection into carbonate reservoirs of the Ural-Volga region has not yet become widespread due to negative field experience from the Soviet period (Lozin, 2012), as well as due to the insufficient efficiency of commercially available surfactants. At the same time, by using compositions of anionic and nonionic surfactants (anionic surfactants and nonionic surfactants) that exhibit a synergistic effect in reducing interfacial tension (Pletnev, 1987; Holmberg et al., 2003), as well as satisfactory adsorption values that significantly increase the wettability of carbonate rock, economically viable well treatments can be achieved. It is most advisable to inject the surfactant slug after IPC, although a surfactant-only treatment option is also possible.

In this work, for the laboratory substantiation of surfactant slug injection methods, Composition 1 (TU 20.41.20-008-12726854-2021) based on Russian reagents was used, specifically, sulfonated fatty alcohol ethoxylates and neonol AF_9-6 with the addition of a mutual solvent, which had previously been tested for a Devonian formation of a field in the Volga-Ural Oil Province. The Ivinskoye field (Republic of Tatarstan, Volga-Ural Oil Province) was selected as the test reservoir, namely the Vereisky and Bashkirian horizons with oil viscosities of 143.42 mPa·s and 155.73 mPa·s, respectively, and with formation water salinity of 270–275 g/L. For such high values of oil viscosity and formation water salinity, selecting an appropriate surfactant composition is a challenging task, since anionic surfactants form calcium and magnesium soaps with hardness ions and precipitate out of the aqueous solution as a flocculent sediment. In this regard, a comprehensive set of studies was conducted to determine the CA, interfacial tension, capillary oil displacement (through spontaneous imbibition), and post-displacement oil recovery during injection of surfactant solutions in RPM water with a salinity of 267.98 g/L, belonging to the calcium-sodium chloride type.

The results of CA measurements carried out on oil-saturated core tablets showed that for the Bashkirian horizon, the CA with formation water is 111.3°. When a droplet of Composition 1 solution (0.1% concentration in RPM water) was applied to the tablet surface, the

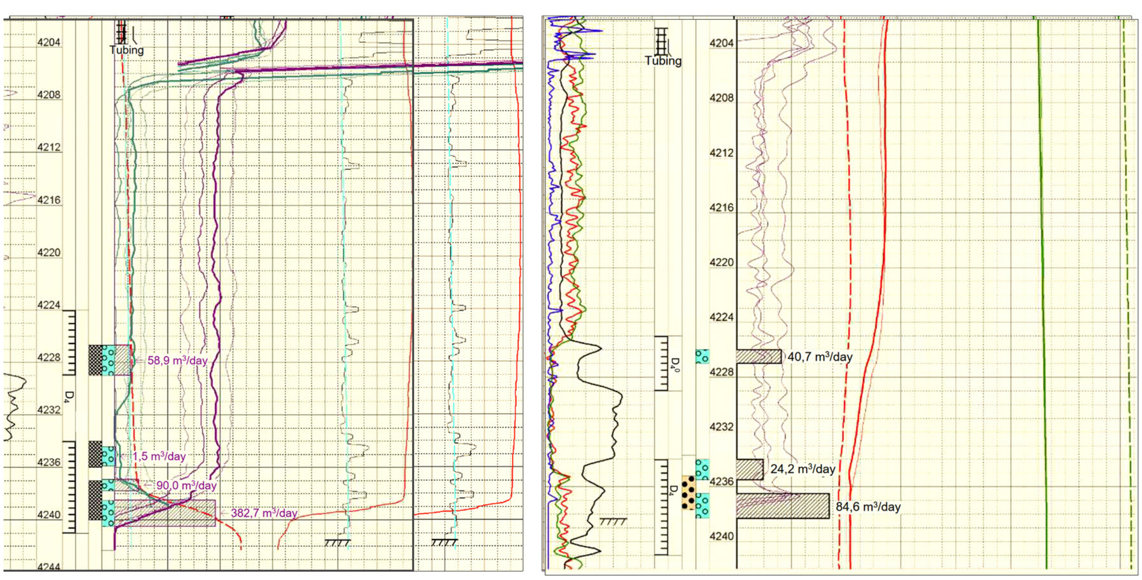

Fig. 14. Injectivity profile of well 820 before and after treatment

CA decreased to 86.9°, and after 4 hours, the value of this parameter was 54°. When the concentration of Composition 1 was increased to 0,3%, immediately after droplet application the CA was 99.2°, and after 4 hours – 38.9°. For the Vereisky horizon, the pattern was similar: at a concentration of 0.1% of Composition 1, immediately after droplet application the CA was 115.7°, and after 4 hours – 70°. For a 0.3% solution of Composition 1, the CA was 102.9° and 24°, respectively.

At the RPM water / oil interface with a concentration of 0.3% of Composition 1, the interfacial tension measurement yielded the following results: 0.123 N/m for the Vereisky horizon and 0.137 N/m for the Bashkirian horizon (Fig. 15).

When modeling waterflooding on a linear composite reservoir model with injection of 0.3 PV of Composition 1 (0.3% concentration in RPM system water), the incremental displacement efficiency was 16.98% for the Vereisky horizon model (permeability 718.8 mD) and 7.07% for the Bashkirian horizon model (permeability 544.2 mD). From the data presented, it is evident that the Vereisky horizon is the most preferable for field pilot testing. Thus, in August 2021, 4.5 tonnes of the commercial form of Composition 1 as a 0.3% solution in RPM system water was injected into injection well 4109 of the Vereisky horizon of the Ivinskoye field. Monitoring of the responding production wells was carried out for six months, and during this period, 231 tonnes of incremental oil were produced (51.3 tonnes per tonne of commercial surfactant form), which is an economically acceptable result.

Considering that cyclic waterflooding is implemented in many carbonate targets with viscous oils (Vladimirov et al., 2014), we evaluated the possibility of using surfactant solutions in one or several water injection cycles, which, by enhancing countercurrent capillary imbibition, can more effectively displace oil from the matrix blocks into the fracture network. Fresh water was used to prepare the surfactant solutions, which, according to Mohammad et al. (2018), is more actively imbibed into hydrophobic carbonate rock, as confirmed by a field project (Yousef et al., 2012). Core and oil from the Tournaisian horizon of the Shchelkanovskoye field (Republic of Bashkortostan, Volga-Ural Oil Province) were selected as the study targets, with topped crude oil having a viscosity of 234.3 mPa·s at 25 °C being used. Topped crude oil is the simplest model of residual oils (Fakhretdinov et al., 1992; Startseva et al., 1998), and its use in core experiments allows the surfactant solutions to manifest themselves more contrastingly. In the experiments, contact angle isotherms and the oil displacement efficiency were determined using Amott cells. Contact angle values were recorded immediately after applying a droplet of the surfactant solution onto the surface of a low-permeability hydrophobic oil-saturated tablet, as well as after a 4-hour exposure. Compositions of anionic and nonionic surfactants (1:2) with an addition of 8% mutual solvent by volume were studied. As anionic surfactants, sulfonated alcohol ethoxylates and sodium salts of sulfonated alkylaromatic compounds were selected; as nonionic surfactants, neonol AF_9-6 and OP-4 were used. It was found that for all compositions, as the concentration increased from 0.01% to 0.05%, a significant decrease in contact angle occurred, and when the concentration of the surfactant blends reached 0.07%, the contact angle decreased to nearly zero. The critical micelle concentration in all cases is estimated to be in the range of 0.05% to 0.06% (Fig. 16).

For the Amott cell experiment, the composition of the sodium salt of sulfonated alkylbenzene and AF_9-6, which exhibited the minimum contact angle at all concentrations (Fig. 16), was selected. It was found that capillary displacement of topped crude oil from an oil-saturated core with a permeability of 139 mD (irreducible water saturation – 8%) by water from the reservoir pressure maintenance system yielded a displacement efficiency of 16.6%. Capillary imbibition of a 1% solution of sulfonated alkylbenzene and nonionic surfactant made it possible to achieve a displacement efficiency of 41.9%. Thus, these experiments clearly

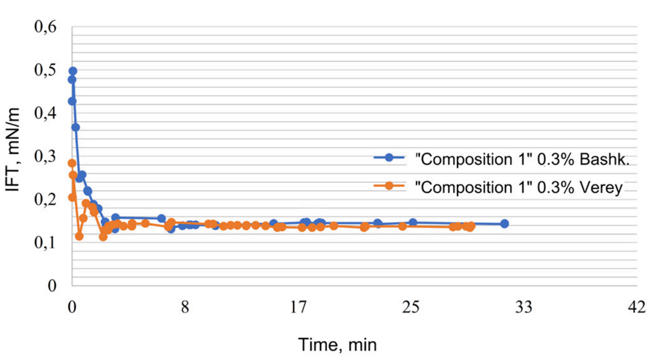

Fig. 15. Results of interfacial tension measurements for the Composition 1 solution

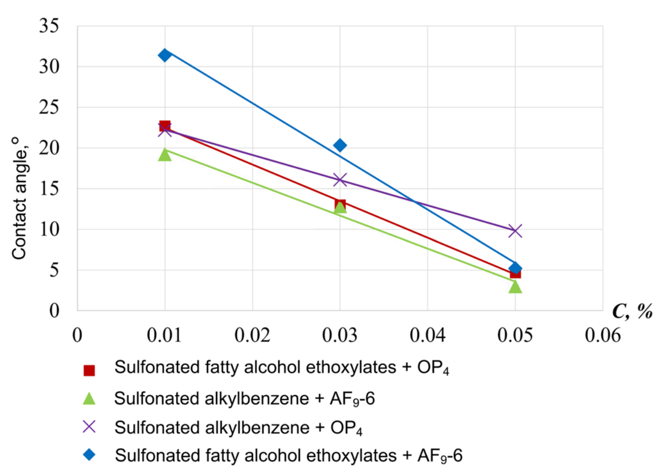

Fig. 16. Contact angle (CA) isotherms of surfactant solutions

confirm the potential of using surfactant solutions in fresh water for cyclic waterflooding in carbonate reservoirs with viscous oil.

Well Water Shut-Off

The fractured-vuggy structure of carbonate development targets facilitates water breakthroughs through fractures from injection wells to production wells and complicates water shut-off treatments, imposing increased requirements on plugging materials. The most important quality of water shut-off compositions in such cases is their high water-blocking ability in fracture structures and deep penetration into the formation. According to Bagrintseva (1999), fracture aperture in carbonate reservoirs varies from 20 to 60 μm. In a study of the Logovskoye field in Perm Krai (Martyushev et al., 2015), it was shown that the fracture aperture, determined by three methods, ranges from 5 to 12 μm. In all cases, when embarking on water shut-off operations in carbonate reservoirs, it is necessary to obtain information about the fracture density and aperture.

Previously, we showed that hydrogel compositions are easily filtered in fractures and, after gelation, exhibit sufficient water-blocking properties (Telin et al., 2023). Subsequently, we found that the combination of a foam gel with a hydrogel in a plugging slug contributes to an increased gas-shutoff effect in granular reservoirs (Telin et al., 2024). In fractured reservoirs, we decided to use a stiffer hydrogel by introducing polypropylene fiber (PPF) into the known plugging composition based on the reagent NGT-Chem-3 (Karatseev et al., 2024). It should be noted that in Tatneft PJSC, a similar approach has been well developed, wherein fibrous and dispersed additives are introduced into the gel, which enhances the efficiency of water shut-off in carbonate reservoirs (Baikova, Muslimov, 2016). At TatNIPIneft (Tatneft PJSC), there exists RD 153-39.01-821-13 entitled “Instruction on the Technology for Using Reinforced Polymer Systems to Enhance Oil Recovery and Water Shut-Off in a Production Well (APS Technology)”.

In this work, to substantiate the amount of fiber to be added to the gel, we conducted a series of rheological and coreflood experiments. The rheological experiments consisted of determining parameters such as the elastic modulus (G’), viscous modulus (G’’), and complex modulus (G*) using oscillatory rheometry, as well as the yield stress using shear rheometry. A gel based on the complex reagent NGT-Chem-3 with a polyacrylamide concentration of 1.7 wt.% in water was taken as the base matrix. To this, PPF was added in amounts ranging from 0.05 wt.% to 0.20 wt.% (Table 2).

From Table 2, it can be seen that the addition of fiber in all cases leads to an increase in all rheological parameters, with the elastic modulus increasing from 23.07 Pa to 27.52 Pa as the reinforcing fiber content increases from 0.00% to 0.20%. Similar changes occur in the viscous modulus, complex modulus, and yield stress. Flow studies conducted on the idealized fracture model showed that the RRF during flow in a fracture with an aperture of 50 μm at flow rates of 0.01 and 0.1 cm^3/min for the gel without filler was 24.72 and 7.23 units, respectively, whereas for the gel with the addition of 0.15% fiber, it amounted to 310.73 and 43.61 units, respectively.

Experimental water shut-off operations were carried out on production well 949 of the Zyuzeyevskoye field (Bashkirian horizon) (Republic of Tatarstan, Volga-Ural Oil Province). A sharp increase in watercut in this well from 40% (2017) to 96% occurred in July 2023. Moreover, it was found that shutdowns of injection in injection well 2363 rapidly reduced the watercut in production well 949, which indirectly indicates the breakthrough of injected water. Indeed, as can be seen

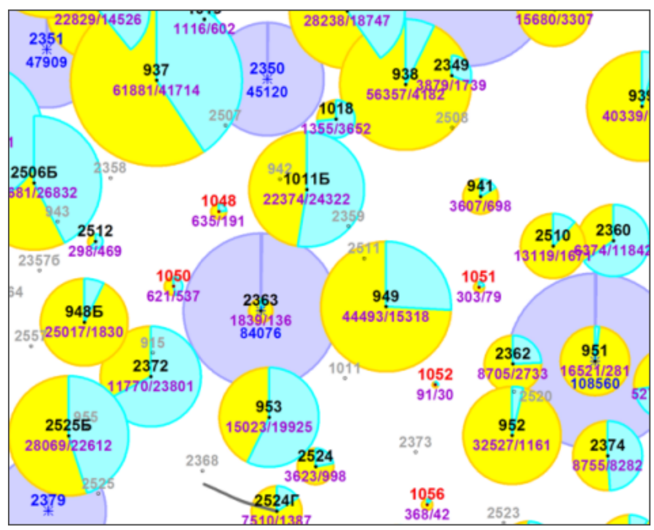

Fig. 17. Current production map: Zyuzeyevskoye field, well 949

Composition	G', Pa	G'', Pa	G*, Pa	Crossover, Pa	Linear viscoelastic range, Pa	Yield stress, Pa
BG	23.07	11.72	25.88	45.27	31.36	85.0
BG + 0.05% PPF	24.23	12.28	27.17	43.99	30.30	104.0
BG + 0.10% PPF	26.62	14.19	30.16	41.34	26.99	105.5
BG + 0.20% PPF	27.52	13.88	30.82	46.45	32.95	160.0

Table 2. Rheological parameters of the base gel (BG) with varying PPF content

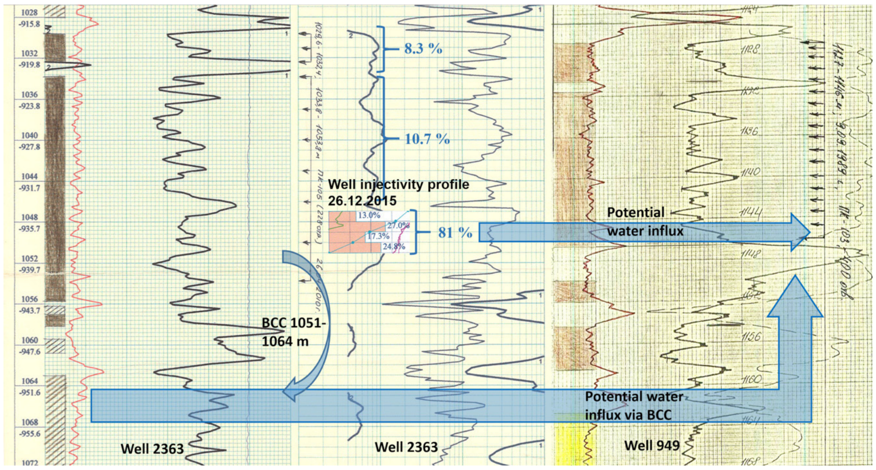

Fig. 18. Comparison of radioactive logging data and injectivity profile data of injection well 2363

from the current production map (Fig. 17), well 949 is located within the influence zone of injection well 2363.

Comparison of radioactive logging data and injectivity data of wells 2363 and 949 showed that excess water may enter both through the bottom of the perforated interval of the reservoir and from below via behind-casing circulation in injection well 2363 (Fig. 18).

Data from field geophysical surveys conducted on December 24, 2024, showed that in the interval 1146–1155 m, temperature logging in the shut-in well revealed behind-casing circulation (Fig. 19, marked with a red oval).

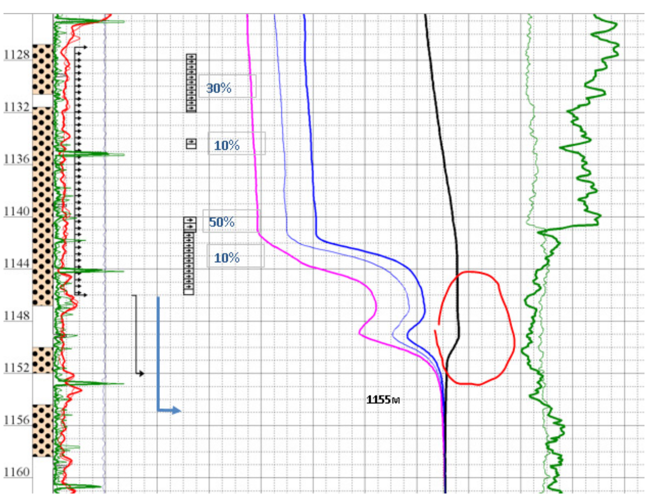

Fig. 19. Production log sheet dated December 24, 2024: well 949

Table 3 presents the operating parameters of well 949 before and after treatment, and it should be noted that the injectivity prior to the water influx limitation treatment was 576 m³/day at 0 atm.

The isolation work was carried out through the existing perforation interval. Into the reservoir, 30 m³ of a foam-polymer composition was injected at 0 atm, followed by 5.5 m³ of NGT-Chem-3 reagent with a polyacrylamide concentration of 1.7% and polypropylene fiber content of 0.15%. The pressure at the end of injection rose to 35 atm, and after displacement with technical water, to 100 atm. From Table 3, it can be seen that before the start-up of injection well 2363, the watercut decreased to 35%, and after the launch of the reservoir pressure maintenance system, it increased to 50%. Based on the results obtained, the subsoil user was recommended to perform IPC on well 2363, which would reduce the watercut of production to 35–40%.

Production Well Stimulation

Traditionally, acid treatments are used for production well stimulation in carbonate reservoirs. However, as reserves are depleted and well watercut increases, matrix acid treatments, which lead to the formation of dominant wormholes (Kharisov et al., 2012), are being replaced by targeted acid stimulation, in which a diverting agent slug is first injected, followed by the acid composition. Gels, emulsions, polymer solutions, and dispersion compositions are used as diverting agents (Magadova et al., 2009; Gavrilenko, 2023). In

Well operating mode	Q_1, m³/day	Q_0, tonnes/day	Watercut, %
Before workover (remedial cementing operations)	17	0.2	99
After workover as of January 1, 2025; RPM system not started	8	4.8	35
After workover as of January 20, 2025; RPM system started	7.5	3.5	50

Table 3. Technological parameters of well 949 operation before and after treatment

the work of Musabirov et al. (2019), a self-degrading fibrous material in a viscoelastic surfactant gel was proposed as an acid diverting agent. A similar technical solution is described in Polozov and Al-Rumaima (2018). However, we currently believe that there is no need to remove the fibrous polymer-gel composite from fractures; on the contrary, retaining water-blocking reagents in fractures will make it possible to reduce the watercut of production when inducing flow. In the early 2000s, we successfully used invert dispersion (disin) in targeted acid treatment technology (Telin et al., 2001) and achieved an increase in well productivity while simultaneously reducing watercut. Currently, disin is no longer produced; however, we consider the use of Pickering inverse emulsions and fibrous polymer-gel composites for fracture shutoff during acid stimulation to be a relevant task. It should be noted that the most widespread diverting agents in acid treatments are inverse emulsions (Glushchenko, 2008). In the present work, we propose using fibrous polymer-gel composites for targeted acid treatment. A composition based on NGT-Chem-3 reagent with a polyacrylamide concentration of 1.9% was selected as the base matrix for this composite. The rheological and flow properties of fibrous polymer-gel composites with the addition of chrysotile microfibers (1.5%) and polypropylene fiber 4–6 mm in length (0.15%) were studied. Thus, while the elastic modulus of the base matrix was 36.1 Pa and the complex modulus was 41.7 Pa, the addition of chrysotile and polypropylene fiber increased these parameters to 44.6 Pa and 50.9 Pa, respectively. Flow testing on an idealized fracture model with an aperture of 100 μm showed that the composition with two fibrous fillers yields an RRF of 167.3, 162.4, and 89.3 at flow rates of 0.1, 0.5, and 1.0 cm^3/min, respectively. In the case of a fracture aperture of 650 μm, at the same water flow rates, the RRF was 63.4, 8.72, and 5.38, respectively. As can be seen from the data presented, depending on the fracture aperture, it is possible to select a plugging composition for reliable fracture isolation during the injection of the acid composition.

The most effective for matrix treatment of carbonate reservoirs are acid compositions containing viscoelastic surfactants, which impart self-diversion properties to the acid (Kuryashov et al., 2009). The only drawback of such compositions is the high cost of the main component – zwitterionic compounds, the majority of which are alkylbetaines.

In order to create a cheaper and sufficiently effective acid composition, we imparted a self-diversion function to the composition by introducing oleic acid into it. The fact is that the resulting calcium oleate is soluble in oil but insoluble in water. In combination with a nonionic surfactant, the reaction of such an acid composition with carbonate rock in water leads to the formation of a colloidal dispersion, the viscosity of which is 2–4 times higher than the viscosity of formation water, depending on the nonionic surfactant – calcium oleate ratio. Of course, this effect is not comparable with the efficiency of known reagents such as Katol-40 (Mirriko); however, it should be noted that oleic acid introduces a self-diversion element into the acid composition and, in addition, ensures the equalization of its reaction rates with water-saturated and oil-saturated intervals. The acid factor (Kharisov et al., 2011) of the composition with oleic acid compared to inhibited hydrochloric acid is 3.38; therefore, this acid composition will penetrate the reservoir more than 3 times deeper than inhibited hydrochloric acid.

Kinetic studies were carried out using a rotating disk unit as well as a volumetric unit. In the first case, the reaction kinetics were monitored by the released calcium ion (Table 4), while in the second case, by the volume of released carbon dioxide. The processing of the kinetic curves obtained by the two experimental methods yielded close results. For example, while the acid factor for an oil-saturated core determined using the volumetric unit was 3.06, the value obtained with the rotating disk unit was 3.38. Given that the rotating disk method is preferred in the literature, we adopted the value obtained by this method (3.38).

Subsequent flow tests on an SMP-PS/FES-2R unit (Kortech, Russia) on single cores with a permeability of 7.9 mD confirmed the correctness of the chosen approach: it turned out that inhibited hydrochloric acid breaks through the core upon injection of 0.76 PV,

Stimulating composition	Reaction rate, mol/(cm^2·s)	Reaction order (dimensionless)	Rate constant, $(mol/cm^3)^{1-n}$(cm/s)	Acid factor
Water-saturated core				
12% HCl	$2.28 \cdot 10^{-5}$	0.3086	$1.42 \cdot 10^{-4}$	4.18
Acid composition	$5.45 \cdot 10^{-6}$	0.3799	$5.10 \cdot 10^{-5}$	
Oil-saturated core				
12% inhibited HCl	$1.68 \cdot 10^{-5}$	0.3799	$1.54 \cdot 10^{-4}$	3.38
Acid composition	$4.97 \cdot 10^{-6}$	0.3799	$4.58 \cdot 10^{-5}$	

Table 4. Results of reaction kinetics calculations (rotating disk method)

whereas with the addition of tall oil fatty acids (which contain 80% oleic acid), less than 0.55 PV of the acid composition was required. Thus, it can be argued that the use of targeted acid treatments with diverting agents having different degrees of softness-stiffness, as well as acid compositions with self-diversion elements, will make it possible to increase oil inflow and limit water inflow under different geological and mining conditions.

Discussion of Results

When selecting GEOs, the determining factors are always the geological and physical conditions of field development. For carbonate targets, this is primarily fracturing (Bagrintseva, 1999). According to Seright and Brattekas (2021), "strong gels" (based on polyacrylamide and crosslinkers), i.e., those formed at the wellhead, are used for fracture isolation in carbonates. Undoubtedly, this approach is effective, and it has been validated in Russia (Ismagilov, 2015); however, based on available data on fracture length, aperture, and density, the permeability of which can reach tens of Darcies (Belonovskaya et al., 2007), deep treatment of the fracture network is required in many cases. For these purposes, polyacrylate reagents are used, which, upon encountering formation water, form gel-precipitates directly in the fractures, where they create a water-shutoff barrier. The practice of carrying out IPC in many fields of the Volga-Ural Oil Province confirms the feasibility of using this type of stimulation.

During water shut-off operations, it is also necessary to ensure penetration of the plugging mass to a radius of at least 1.5–2.0 m from the well perforation interval by selecting reagent concentrations under reservoir conditions. In this regard, it is advisable to inject a gellant with fibrous fillers, which transforms into a reinforced gel at a programmable distance from the wellbore.

It is most advisable to carry out enhanced surfactant slug injection into injection wells after IPC, with the expectation that the fractures will be isolated by the gel- and precipitate-forming reagent, and the surfactant composition will be redirected into the pore matrix.

In the stimulation of production wells with acid compositions, targeted treatments have become firmly established in field practice. Technological improvements are possible through the optimization of the acid-diverting slug and the acid composition. It is preferable that the acid-diverting slug possesses the properties of a water-shutoff barrier, while the acid composition incorporates self-diversion functions.

Conclusion

Thus, as a result of the experimental and pilot field work carried out, it has been established that with the proper selection of surfactant compositions, even under conditions of high formation water salinity and high-viscosity oil, surfactant slug injection makes it possible to enhance oil production with a specific efficiency of 51.3 tonnes per tonne of injected commercial form of the domestic reagent composition.

The polyacrylate and modified polyacrylamide reagents used in injectivity profile control technologies, when injected into two injection wells, yielded 2880 tonnes of incremental oil from the responding production wells.

Water shut-off in a well in a fractured-vuggy Bashkirian formation using sequential injection of a foam-polymer composition and a fibrous polymer-gel composite made it possible to reduce the watercut from 99% to 50%.

For the stimulation and water shut-off of production wells, it is proposed to apply targeted acid treatment, wherein at the first stage a water-shutoff fibrous polymer-gel composite should be injected, and at the second stage an acid composition with self-diversion capability and a controlled reaction rate of the acid with the carbonate rock.

In conclusion, it should be noted that the use of a systematic approach to the treatment of injection and production wells, based on the analysis of reserve depletion as well as the determination of well influence and interaction, should lead to the cost-effective recovery of residual oil reserves in the carbonate reservoirs of the Volga-Ural Oil Province.

Acknowledgements

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About the Authors

Lyubov E. Lenchenkova – Doctor of Technical Sciences, Professor of Oil and Gas Fields Development and Operation Department, Ufa State Petroleum Technological University

1 Kosmonavtov St., Ufa, 450062, Russian Federation
e-mail: LenchenkovaL@mail.ru

Sergey A. Vezhnin – Head of the EOR Department, Ufa Scientific and Technical Center

59 Aksakov St., Ufa, 450076, Russian Federation
e-mail: Vezhninsa@ufntc.ru

Farit E. Safarov – Candidate of Chemical Sciences, Senior Researcher at the EOR Department, Ufa Scientific and Technical Center; Senior Engineer, Ufa Institute of Chemistry of the Ufa Federal Research Center of the Russian Academy of Sciences

59 Aksakov St., Ufa, 450076, Russian Federation
71 Oktyabrya ave., Ufa, 450054, Russian Federation
e-mail: SafarovFI@ufntc.ru

Igor P. Novikov – Chief geologist, Nafta-Service LLC
15 Lenin St., Almetyevsk, 423452, Russian Federation
e-mail: Novikovipnafta@mail.ru

Ravil N. Yakubov – Candidate of Technical Sciences, Associate Professor of Oil and Gas Fields Development and Operation Department, Ufa State Petroleum Technological University

1 Kosmonavtov St., Ufa, 450062, Russian Federation
e-mail: Rnyakubov@mail.ru

Mikhail A. Varfolomeev – Candidate of Chemical Sciences, Director of the Technology Park “Low-Tonnage Chemical Technologies”, Kazan Federal University

4/5 Kremlevskaya St., Kazan, 420111, Russian Federation

e-mail: Mikhail.Varfolomeev@kpfu.ru

Sergei A. Nazarychev – Research Scientist at the Laboratory of Enhanced Oil Recovery Methods, Kazan Federal University

1/29 Lobachevskogo St., Kazan, 420111, Russian Federation

e-mail: SeANazarychev@kpfu.ru

Aleksei O. Malakhov – Research Scientist at the Laboratory of Enhanced Oil Recovery Methods, Kazan Federal University

1/29 Lobachevskogo St., Kazan, 420111, Russian Federation

e-mail: AlOMalahov@kpfu.ru

Rustem R. Asadullin – Candidate of Technical Sciences, Associate Professor of Oil and Gas Fields Development and Operation Department, Ufa State Petroleum Technical University

1 Kosmonavtov St., Ufa, 450062, Russian Federation

e-mail: Yorkerbridge@mail.ru

Timur F. Khaernasov – Postgraduate student of Oil and Gas Fields Development and Operation Department, Ufa State Petroleum Technological University

1 Kosmonavtov St., Ufa, 450062, Russian Federation

e-mail: timur.fidailevich@mail.ru

Artem S. Pavlik – Postgraduate student of Oil and Gas Fields Development and Operation Department, Ufa State Petroleum Technological University

1 Kosmonavtov St., Ufa, 450062, Russian Federation

e-mail: Lnplay@mail.ru

Andrey E. Fetisov – Postgraduate student of Oil and Gas Fields Development and Operation Department, Ufa State Petroleum Technological University

1 Kosmonavtov St., Ufa, 450062, Russian Federation

e-mail: Fetisovae@ufntc.ru

Alexey G. Telin – Candidate of Chemical Sciences, Deputy Director for Scientific Work, Ufa Scientific and Technical Center

59 Aksakov St., Ufa, 450076, Russian Federation

e-mail: telinag@ufntc.ru

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Увеличение нефтеотдачи в карбонатных пластах Волго-Уральской нефтяной провинции: проблемы и решения

Л.Е. Ленченкова[1], С.А. Вежнин[2], Ф.Э. Сафаров[2,3], И.П. Новиков[4], Р.Н. Якубов[1], М.А. Варфоломеев[5], С.А. Назарычев[5], А.О. Малахов[5], Р.Р. Асадуллин[1], Т.Ф. Хаернасов[1], А.С. Павлик[1], А.Э. Фетисов[1], А.Г. Телин[2]*

[1]Уфимский государственный нефтяной технический университет, Уфа, Россия
[2]ООО «Уфимский Научно-Технический Центр», Уфа, Россия
[3]Уфимский институт химии Уфимского федерального исследовательского центра РАН, Уфа, Россия
[4]ООО «Нафта-Сервис», Альметьевск, Россия
[5]Казанский федеральный университет, Казань, Россия

Увеличение нефтеотдачи и интенсификация добычи нефти в карбонатных коллекторах Волго-Уральской нефтяной провинции осуществляется посредством планирования и проведения геолого-технических мероприятий (ГТМ), а классические многообъемные физико-химические и газовые методы практически не применяются. В этом отношении особое значение имеют технологическая эффективность отдельных видов ГТМ и применение их системным образом.

Опираясь на анализ российского опыта, а также на собственные экспериментальные и опытно-промысловые исследования, авторы данной статьи приводят результаты применения отдельных видов воздействия, которые успешно прошли апробацию в промысловых условиях и показали высокую эффективность. Представлены данные лабораторной проработки всех технологических операций, предшествующей опытно-промысловым испытаниям. Показано эффективное применение закачки оторочек поверхностно-активных веществ (ПАВ) в карбонатные пласты с высокой вязкостью нефти и высокой минерализацией пластовой воды. Обработки по выравниванию профиля приемистости осуществлены с помощью полиакрилатных и модифицированных полиакриламидных реагентов (NGT-Chem-2 и NGT-Chem-6), которые сшиваются ионами жесткости и алюминия. Приведены данные по изменению профиля приемистости и дополнительной добыче нефти на высокотемпературных карбонатных объектах разработки. Водоизоляция скважины в трещиновато-поровом коллекторе проведена с помощью самогенерирующейся пенополимерной системы и волокнистого полимерногелевого композита. Интенсификацию притока нефти одновременно с водоизоляцией предложено осуществлять с использованием волокнистого полимерногелевого композита и интенсифицирующей кислотной композиции с замедленным временем реакции с карбонатной горной породой и элементами самоотклонения – за счет добавок талловых масел и ПАВ. Цель работы – ознакомить с лучшими практиками проведения ГТМ проектировщиков, чтобы в проектные документы закладывались современные проверенные технологии, а при проектировании системного воздействия на карбонатные объекты разработки использовался передовой опыт сервисных предприятий.

Ключевые слова: системная технология, карбонатный коллектор, выравнивание профиля приемистости, малообъемные методы увеличения нефтеотдачи, гелеосадкообразующие реагенты, закачка оторочек ПАВ, направленные кислотные обработки с отклонителями

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