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Fluid-Loss Control Technology: From Laboratory to Well Field

Shamil Islamov [1,*], Ravil Islamov [2], Grigory Shelukhov [1], Anar Sharifov [3], Radel Sultanbekov [4], Rustem Ismakov [5], Akhtyam Agliullin [6] and Radmir Ganiev [6]

[1] Department of Petroleum Engineering, Saint Petersburg Mining University, 199106 Saint Petersburg, Russia; gsheluhov@mail.ru
[2] Department of Technological Development, RusWellGroup JSC, 115409 Moscow, Russia; rrislamov@rosatom.ru
[3] Department of Product Development, Nedra—New Digital Resources for Assets, 190000 Saint Petersburg, Russia; anarshar@mail.ru
[4] Resource Management Centre, Gazpromneft Marine Bunker, 199106 Saint Petersburg, Russia; radelsultanbekov@mail.ru
[5] Department of Drilling Engineering, Ufa State Petroleum Technological University, 450064 Ufa, Russia; ismakovrustem@gmail.com
[6] World-Class Scientific Center, Ufa State Petroleum Technological University, 450064 Ufa, Russia; dir@et.center (A.A.); radmirganiev@mail.ru (R.G.)
* Correspondence: sh.islamoff@gmail.com

Abstract: Effective fluid-loss control in oil wells is a critical concern for the oil industry, particularly given the substantial reserves situated in carbonate reservoirs globally. The prevalence of such reservoirs is expected to rise with the slow depletion of hydrocarbons, intensifying the need to address challenges related to deteriorating reservoir properties post well-killing operations. This deterioration results in significant annual losses in hydrocarbon production at major oil enterprises, impacting key performance indicators. To tackle this issue, this study focuses on enhancing well-killing technology efficiency in carbonate reservoirs with abnormally low formation pressures. To address this issue, the authors propose the development of new blocking compositions that prevent the fluid loss of treatment fluids by the productive reservoir. The research tasks include a comprehensive analysis of global experience in well-killing technology; the development of blocking compositions; an investigation of their physico-chemical, rheological, and filtration properties; and an evaluation of their effectiveness in complicated conditions. The technology's application in the oil and gas condensate fields of the Volga-Ural province showcases its practical implementation. This study provides valuable insights and solutions for improved fluid-loss control in carbonate reservoirs, ultimately enhancing well performance and hydrocarbon recovery.

Keywords: well-killing technology; workover; hydrophobic emulsion composition; fluid-loss; abnormally low reservoir pressure; fractured carbonate reservoir; complicated conditions

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1. Introduction

The modern oil and gas industry is confronted with significant technological challenges associated with well killing before workover operations, especially in fields with carbonate reservoirs and abnormally low reservoir pressures. This issue has gained particular relevance, as 40% to 60% of the world's oil reserves are concentrated in carbonate reservoirs [1–5].

Fracturing is typical for carbonate reservoirs. Fractures in the rock can form as a result of tectonic processes during the formation of geological faults and folds in carbonate reservoirs. Such fractures are called natural. Fractures can also have technogenic origins, formed as a result of hydraulic fracturing and hydrochloric acid treatment [6,7].

Both natural and technogenic fracturing enhance the well productivity coefficient, ensuring an increase in the production rate of oil wells. Conversely, fractures can pose additional problems linked to breakthrough into an aquifer or into an overlying gas cap, attracting water and gas to the wells. They can also contribute to the fluid loss of technological fluid by the formation during workover operations [8,9].

It is anticipated that the proportion of such reservoirs will increase due to expanded geological and geophysical explorations. Equally important to note is that many developing fields with carbonate reservoirs face challenges related to the deterioration of reservoir properties after well killing before workover operations, resulting in significant losses in hydrocarbon production [10–12].

This article presents a study aimed at improving the efficiency of well-killing technology before workover operations in fields with fractured carbonate reservoirs and abnormally low reservoir pressures [13–15]. The focus of the work is on the development of blocking compositions capable of preventing complete fluid losses, thereby enhancing well operating conditions.

In practice, two main physical principles are employed to reduce fluid losses: increasing the viscosity of the blocking technological fluid and plugging the pores and filtration channels with solid particles [16–18]. A literature review revealed that the most preferable blocking compositions are structured hydrocarbon systems, such as inverse emulsions, which may additionally contain acid-soluble solid additives [19].

In the post-repair period, acid treatments may be required to remove the filler. However, when using gentle emulsion compositions, there is a possibility of creating a blocking screen with insufficient strength to retain the gas. An alternative in this case may be the use of gel-forming compositions [20] with fillers. One way to reduce the density and filtration of reverse emulsions and polymer solutions is by filling them with aluminosilicate microspheres with a concentration of up to 30% wt. [21].

The results of numerous observations of well-killing operations with various types of solutions enable their arrangement according to the decreasing negative impact on the bottomhole formation zone. The order is as follows: reverse emulsion; formation waters + surfactants; formation water + surfactant + polyacrylamide; and aqueous solutions of calcium or sodium chloride salts + surfactant and without surfactant. Field data from the results of well killing with reverse emulsions indicate the preservation of the filtration properties of the bottomhole formation zone in the post-repair period [22].

Regarding the characteristics of reverse emulsions, it is important to note their rheological properties and stability. These characteristics are influenced by the ratio of hydrocarbons to aqueous phases and their composition, as well as the type and concentration of the emulsifier reagent, methodology, and preparation procedure. In addition to sedimentation stability, stability under dynamic conditions must also be taken into account to justify the choice of composition. To maintain aggregative stability, it is necessary that inverse emulsions have a certain ultimate shear stress. In this case, the performance characteristics are determined based on the initial shear deformation. Inverse emulsions, especially highly concentrated ones, can be destroyed under various flow conditions, once again emphasizing the importance of their stability under dynamic conditions.

Thus, active research and development of existing and promising reverse emulsions for use as process fluids should play an important role in the introduction of new technologies [23].

One of the common trends in well killing is the use of viscoelastic compositions, polymers “cross-linked” with polyvalent metals, mainly the polysaccharide base. This ensures reliable shielding of the bottomhole formation zone from intensive penetration of water filtrates. At the same time, these technologies have a number of disadvantages, one of which is the complex sequence of technological operations and the complexity of their implementation [1].

Another disadvantage of polymer blocking compositions is the need to use destructors.

Destructors can be used in two ways: together with the technological fluid during workover or individually after workover operations.

The first method relies on the mixing of the destructor with the entire volume of the technological fluid, its complete destruction, and, consequently, a high permeability recovery coefficient. To effectively combat solution fluid loss, the study [24] describes polyglycolic acid as a moderator. The disadvantage of this method is the impossibility of slowing down the liquefaction of the technological fluid for more than 2 days [25].

The second method does not depend on the time of work and provides fluid-loss control throughout the entire workover [26]. However, the adsorption properties of polymers and the difference in rheological properties of technological fluids and destructors should be considered. This makes destruction impossible, and productivity is partially lost due to a decrease in the permeability coefficient [27].

When carrying out the well-killing process by operating two or more objects with high-permeability hydraulic fractures [28], or with a high degree of cavernousness of the productive reservoir and abnormally low reservoir pressure, in order to combat fluid loss and showings of gas, oil, and water, the common practice is the introduction of dispersed filtration reducers into the blocking composition. The most suitable reducer for this is particularly fractionated chalk [29]. According to research consensus, the average size of dispersed particles in technological fluids should be one third of the average pore size of the productive reservoir, with their concentration being at least 5% vol. Solid particles can be represented by NaCl, $Ca(OH)_2$, or $CaCO_3$. Water-soluble polymers are used as stabilizers: starch, hydroxypropyl guar, hydroxyethyl cellulose, etc. [30,31].

To prevent long-term blockage of formation pores, calcium carbonate ($CaCO_3$) is usually used, as carbonate particles can be removed by acid treatment [32]. Unfortunately, the rate of diffusion of the acid to the plugging particles is slow due to the thickened carbonate solution. Even after the acid diffuses through the thickened liquid, most of it is wasted on the dolomite formation before the particles are dissolved.

The paper [33] describes an experience where an emulsion suspension hydrophobic composition with a wide range of fillers is used for horizontal open holes.

From a technological point of view, an interesting method for well killing [34] consists of lowering polymer rods, in particular made of (composed of/containing) methylcellulose, which additionally include a crystalline gas-releasing agent: calcium carbide and sodium nitrite. This ensures rapid gelation of well water opposite the perforation interval, simultaneously simplifying and reducing the cost of the well-killing process. However, the dissolution time of the methylcellulose rod is long, which leads to a slower formation of gel-forming composition for well killing [35].

The analysis of the methods used in the oil and gas industry to increase the blocking properties of the process fluid has shown the prospects of using fillers with a multi-fractional composition. This occurs because solid filler particles that are too small can penetrate deeply into the formation and lead to a partial loss of its permeability, and larger filler particles, in turn, do not provide a reliable barrier preventing the technological fluid losses by the productive formation.

This work includes a review of global experience [36–45], the development of blocking compositions, the study of their physico-chemical, rheological, and filtration properties, as well as the modeling of well killing and oil well development processes. The research results will contribute to enhancing the efficiency of well-killing technology and reducing production losses in fields with carbonate reservoirs and abnormally low reservoir pressures.

Thus, this work is of significant importance for the oil and gas industry and has the potential to enhance hydrocarbon recovery conditions in fields with complex reservoirs and pressure conditions.

2. Materials and Methods

This chapter is dedicated to describing the laboratory equipment and methodologies used to study properties of the developed blocking composition to substantiate the well-killing technology for oil wells before workover operations in fractured carbonate reservoirs. Laboratory tests were carried out according to the program presented in Figure 1, which includes stages for determining the physico-chemical, rheological, and filtration properties of the technological fluid compositions.

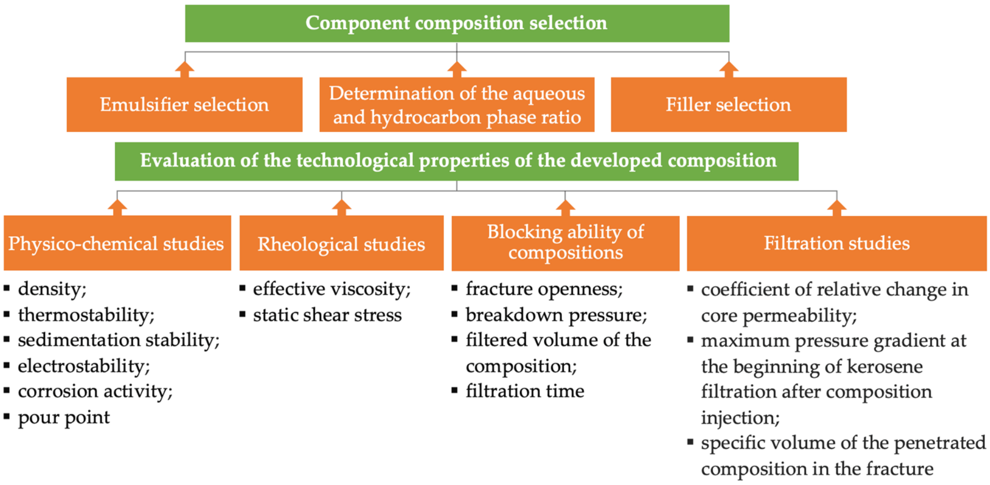


Figure 1. Laboratory test program.

2.1. Preparation of the Blocking Composition BHES-MC

The following chemical reagents were used for the preparation of the developed blocking hydrophobic emulsion solution with marble chips (BHEC-MC):

1. Emulsifier “Yalan-E-2” brand B2—a mixture of reaction products of amines and amino alcohols with fatty acids, produced according to TS 2458-012-22657427-2000 with amendment 1.
2. Oil prepared in accordance with GOST R 51858-2002.
3. Water phase mineralizer—calcium chloride, produced in accordance with GOST 450-77.
4. Fresh water.
5. Mineral filler—multi-fraction marble chips, produced according to TS 5716-001-56390243-2015 (fraction size and loading depend on the fracturing and fluid-loss capacity of the bottomhole formation zone).

The preparation of the blocking composition BHES-MC in laboratory conditions was carried out as follows: emulsifier “Yalan-E-2” brand B2 was dosed into the hydrocarbon phase (oil or diesel fuel), after which the resulting mixture was stirred using the laboratory overhead stirrer for 5 min at a frequency of 1000 rpm. Then, water, mineralized with calcium chloride or sodium chloride, was gradually introduced into the resultant substance equal, in amount, to the total volume of oil and emulsifier. After that, the mixing time was 5 min at 1000 rpm. Then, the remaining mineralized water was introduced in portions over 2 min at a stirrer rotation speed of 1000 rpm, followed by stirring for 13 min at 1500 rpm. After 15 min from the start of composition preparation, marble chips were added in portions, and the resulting mixture was stirred for 10 min at 1500 rpm.

Overall, the total preparation time for the blocking composition BHES-MC was 35 min.

The sequence of adding components of the developed blocking composition BHES-MC is schematically depicted in Figure 2.

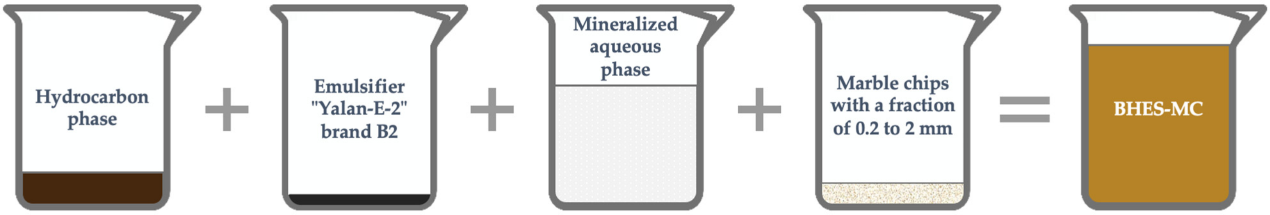


Figure 2. Blocking agent, BHES-MC, preparation procedure.

The prepared blocking composition BHES-MC is a stable, homogeneous, thickened liquid of light brown color. The components of the BHES-MC composition are available, inexpensive, and non-toxic reagents.

The preparation of the blocking composition BHES-MC is possible both on the basis of production support and in the field using mobile and stationary units and installations.

2.2. Determination of the Physico-Chemical Properties of Blocking Compositions BHES and BHES-MC

2.2.1. Determination of Density

The density of individual components constituting the BHES composition was measured using the DE 40 densitometer from Mettler Toledo (Zürich, Switzerland) at the standard temperature 20 °C. The principle of determining density with this device involved measuring the period of oscillation of a glass U-shaped tube filled with several milliliters of the test liquid. Density measurement was performed automatically with a precision of 0.0001 g/cm^3.

The density measurement process consisted of several stages:

1. The mass of the empty pycnometer was determined using precise scales, such as the XP 204 analytical balances from Mettler Toledo (Zürich, Switzerland) with a division value of 0.0001 g.
2. Distilled water was poured into the pycnometer up to the mark.
3. The mass of the pycnometer filled with distilled water was measured.
4. The water was poured out of the pycnometer, and its contents were dried in an oven.
5. The pycnometer was filled up to the mark with the tested technological fluid, and its mass was determined on the scales.
6. The density of the tested technological fluid (ρ_{tf}) was determined using Equation (1):

$$\rho_{tf} = \frac{m_2 - m}{m_1 - m} \cdot \rho_{dw}, \tag{1}$$

where m is the mass of the pycnometer, g; m_1 is the mass of the pycnometer with water, g; m_2 is the mass of the pycnometer with the tested technological fluid, g; and ρ_{dw} is the density of distilled water, g/cm^3.

The density measurement of the tested compositions was also conducted using hydrometers according to the methodology [46] at 20 °C. This is due to the density measurement of the technological fluid in field conditions before injection into the well being typically performed using a hydrometer. A 450 mL sample of the tested composition was poured into a 500 mL graduated cylinder volume at standard temperature. Subsequently, a hydrometer was placed in the cylinder, and readings were taken after one minute.

2.2.2. Thermal Stability Assessment

Determination of the thermal stability of the investigated compositions was conducted as follows: the prepared composition was placed in a thermostat, for example, in the VO 400 incubator by Memmert GmbH (Büchenbach, Germany), with a set temperature of 90 °C, and maintained for 7 days. Simultaneously, control samples were kept for 7 days at standard conditions. Every 24 h, the condition of the sample and the amount of separated hydrocarbons or the aqueous phase were visually assessed.

The criterion for evaluating the thermal stability of the investigated compositions was the absence of phase separation, clouding, color change, or sedimentation.

2.2.3. Sedimentation Stability Assessment

Evaluating sedimentation stability is crucial for determining the dispersed system's capacity to sustain an equilibrium state throughout the volume of the dispersion medium, as well as to identify the tendency of the composition towards phase separation.

Experiments to determine sedimentation stability were conducted according to the following procedure. The investigated compositions were poured into glass test tubes.

Then, the first half of the prepared samples were placed in an oven with the average reservoir temperature of the Volga-Ural oil and gas province (VUOGP) (37 °C), while the other half were kept at standard temperature for 7 days. The condition of the samples was visually assessed (photographed) after 1 h, 24 h, and 7 days.

In order to more reliably evaluate sedimentation stability, additional studies were conducted to determine the indicator of change in the density of the compositions by volume after thermostating. After the specified period (7 days), a sample of the investigated composition was taken from the upper and lower parts of the test tube to measure its density using a pycnometer or hydrometer (to simulate field conditions).

Sedimentation stability was calculated using Equation (2):

$$S_s = \frac{\rho_2}{\rho_1} \cdot 100, \tag{2}$$

where S_s is the sedimentation stability, %; ρ_1 is the density of the upper part of the sample of the investigated composition, kg/m^3; and ρ_2 is the density of the lower part of the sample of the investigated composition, kg/m^3.

Thus, the investigated composition was considered sedimentationally stable; the difference in densities between the lower layer and the upper layer did not exceed 20 kg/m^3, which was a change of 1.5%. In this case, the sedimentation stability range was found to lie within the 98.5% to 100% range.

2.2.4. Determination of Electrical Stability

Electrical stability is another parameter indirectly characterizing the stability of emulsions. This parameter describes the emulsion's resistance to the coalescence of dispersed phase particles and subsequent separation. The method for determining electrical stability involved measuring the voltage corresponding to the moment of emulsion destruction. In the present study, the TEE-01Ts device from LLC Analytica (Moscow, Russia) was used, with a measurement range from 0 to 750 V.

The device operates on the principle of measuring the electrical resistance of the liquid when a constant current of a certain magnitude passes through it. Thus, the breakdown voltage is determined, and its magnitude characterizes the electrical stability of the emulsion—the higher the breakdown voltage, the higher the stability of the emulsion.

The electrical stability indicator for reservoir temperatures up to 80 °C should not be less than 300 V.

It is important to note that the experience of using emulsions indicates that the absolute values of the electrical stability of the initial compositions stabilized by various emulsifiers may not correlate with the values of technological parameters such as thermal stability.

2.2.5. Determination of Corrosion Activity

The study of corrosion activity of blocking compositions involved exposing metal samples, made from steel grade St. 20, to the liquid medium of the acidizing composition for a specified period. Before testing, the surfaces of the metal plates were prepared according to [47].

The experimental studies were conducted at a temperature of 37 °C, within the limit not exceeding ±2 °C. No stirring of the liquid medium was applied. The corrosion rate was calculated based on the mass loss of the metal samples, and the exposure time of the metal samples in the test compositions was 24 h.

The corrosion rate V_c (g/m^2 × hour) was calculated using Equation (3):

$$V_c = \frac{m_2 - m_1}{S \cdot t}, \tag{3}$$

where m_1 and m_2 are the masses of the metal plates before and after testing, respectively, g; S is the surface area of the metal plate, m^2; and t is the test duration in hours.

The corrosion rate of the investigated compositions should not exceed the established standard (0.10–0.12 mm/year). In this case, their use is considered acceptable for conducting downhole operations in accordance with the requirements [47].

2.2.6. Determination of Pour Point

To determine the pour point, the method described in [48] was used. The test tube with a sample of the test composition was placed in the cooling thermostat Proline RP 890 by LAUDA GmbH (Lauda-Königshofen, Germany), where it was cooled down to −45 °C. The flowability of the investigated compositions was assessed visually every 3 °C.

2.2.7. Rheological Studies

To conduct rheological studies for determining the effective viscosity of the investigated compositions, a rotational automated viscometer from Messgerate Medingen GmbH (Ottendorf-Okrilla, Germany)—Rheotest RN 4.1, presented in Figure 3, was used.

Figure 3. Rotary viscometer Rheotest RN 4.1.

The methodology for conducting these studies involved constructing the initial rheological curves of the compositions and determining their static shear stress. The studies were conducted at a temperature of 37 °C.

The controlled shear rate mode was used to obtain the flow curve, representing the shear stress dependence on the shear rate with a gradual increase in the shear rate from 0 to 300 s^{-1}.

For a more informative analysis of the obtained results of flow curves for the investigated compositions, the dependence of effective viscosity (μ_{eff}) on the shear rate was plotted.

The procedure for determining the static shear stress was as follows:

1. Set the viscometer to the “constant shear rate” mode.
2. Pour the test liquid into the cylinder with a volume of 45 cm^3.
3. Set the number of revolutions of the cylinder D = 200 rpm.
4. Mix the test composition for 10 s.

5. After 1 and 10 min of rest of the composition, at a cylinder rotation speed of 3 rpm, readings were taken at which the structure of the test composition began to break down.

A high value of effective viscosity indicates good blocking capabilities of the compositions, contributing to the reduction of the penetration radius of technological fluids into the bottomhole formation zone. This is because an increase in effective viscosity leads to enhanced structural strength of the emulsion. During well development, such a reduction in the penetration radius of the liquid zone can lead to an increase in the permeability recovery coefficient in this zone.

Static shear stress is an indicator of the strength of the internal structure of well-killing fluids formed during the resting period. Its value is determined by the stress that is required to be exceeded in a fluid at rest to break its internal structure and set it in motion. The higher value of the static shear stress in well-killing fluids, the lower the probability of fluid loss of the composition in the reservoir zone is, especially when well killing with reservoir pressure gradients significantly below the potential capabilities of the well-killing fluids in terms of providing the required repression on the reservoir.

2.3. Determination of Interfacial Tension at the "Oil with Emulsifier—Aqueous Solution of Calcium Chloride" Boundary

To measure the interfacial tension of oil with varying emulsifier concentrations at the boundary with an aqueous solution of calcium chloride with a density of 1140 kg/m^3, the EasyDrop tensiometer from KRÜSS GmbH (Hamburg, Germany) was used.

The methodology for using the tensiometer and this software package involved determining the interfacial tension based on the shape and size of the oil droplet containing the emulsifier.

For each type of emulsifier, the dependence of the interfacial tension at the "oil with emulsifier—aqueous solution of calcium chloride" boundary with a density of 1140 kg/m^3 on the emulsifier concentration (at three points—0.05, 0.1, and 0.5%) was determined under standard conditions. Each data point was determined five times, after which the minimum and maximum values were discarded. Subsequently, the average interfacial tension value was calculated for each emulsifier concentration [49].

2.4. Study of the Blocking Ability of the BHES-MC Composition in Modeling a Fractured Reservoir

Studies to assess the blocking ability of compositions in modeling a fractured reservoir were conducted using the HPHT 500 filter press from FANN (Houston, TX, USA). This device is designed to measure the fluid loss characteristics of the studied compositions in the bottomhole formation zone, and it can also be applied to predict the potential for filter cake formation.

For laboratory investigations of the blocking ability of compositions concerning fractures of various degrees of openness, the design of the filter press was modified. A metal disk, simulating a fracture with different degrees of openness (0.1, 0.5, 1, 1.5, 2, 3, and 5 mm), was installed in the lower part of the cell (Figure 4). The lower part of the disk was machined in a "U"-shaped form to engage the entire working length of the fracture and allow unimpeded discharge of the blocking composition from the cylinder (Figure 5a). An enlarged 5 mm diameter exit hole was created in the lower plug of the cell to prevent its closure by the solid phase of the studied blocking agent (Figure 5b).

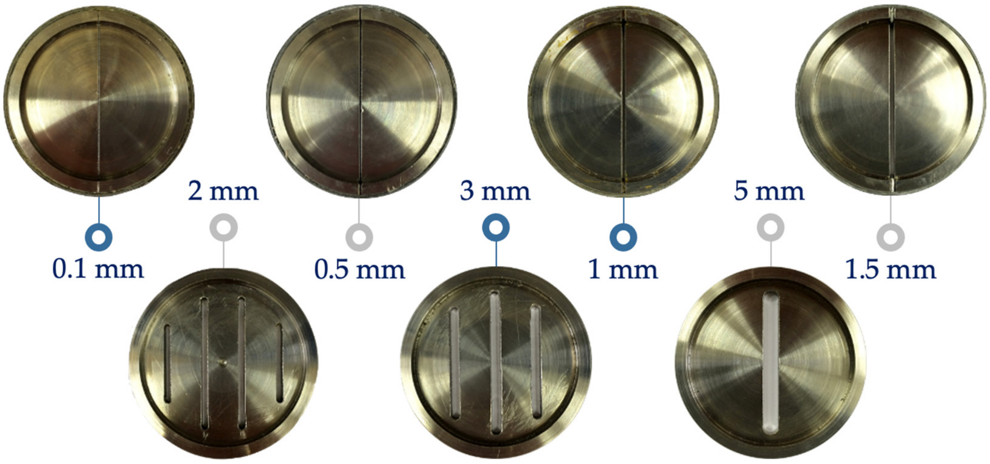


Figure 4. Metal disks simulating a fracture with varying degrees of openness.

(**a**) (**b**)

Figure 5. (**a**) Lower part of the filter press cell (exit point of the blocking composition from the crack); (**b**) Bottom plug of filter press (exit point of the blocking composition from the filter press).

The schematic diagram of the modified filter press construction is presented in Figure 6. The sequence of actions during the experiment consisted of the following stages:

1. In the lower part of the filter press, a metal disk with a crack of a specified degree of openness was placed between two rubber gaskets.
2. The lower plug was installed, and its outlet hole was closed.
3. The test composition was poured into the cell with a volume of 450 cm^3.
4. The filter press cell was closed with the upper plug, after which all clamping screws were tightened, and the top valve was closed.
5. The cell was placed in a thermo-jacket with a given temperature (37 °C) and left for the complete heating of the cell and the test composition (1–2 h).
6. The gas line from the nitrogen cylinder was connected and fixed to the upper valve.
7. The valve in the lower plug was opened.
8. A measuring capacity was placed under the cell to fix the volume of the filtered liquid.
9. The required pressure (1 MPa) was created in the gas line.
10. Along with the opening of the top valve, the stopwatch was started.
11. The test composition was maintained at each pressure (from 1 to 8 MPa, with a step of 1 MPa) for 5 min or until the filtration of liquid through the fracture ceased.
12. The volume of the filtered test composition was recorded for a certain period.

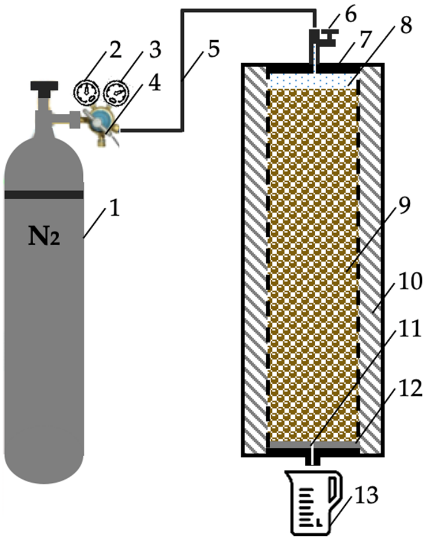


Figure 6. Schematic diagram of the stand for assessing the blocking ability of technological fluids during the well-killing process in a fractured reservoir: 1—gas cylinder; 2—regulator; 3—pressure gauge (pressure in the cylinder); 4—pressure gauge (pressure in the line); 5—gas line (gas supply); 6—top valve (open/close); 7—upper plug; 8—gas; 9—blocking composition; 10—thermo-jacket; 11—fracture; 12—metal disk with a fracture; 13—measuring capacity.

The main experimental conditions are summarized in Table 1.

Table 1. Experimental conditions on the filter press.

Parameter Name	Value
Simulated pressure drops, MPa	1–8
Temperature, °C	37
Fracture width, mm	0.1–5
Volume of blocking composition filtered, cm^3	450
Experiment duration, min	40

The studies used a minimum fracture width of 0.1 mm, corresponding to the minimum sizes of natural microfractures according to data [50] presented in Figure 7. A literature review indicated that in carbonate reservoirs of the VUOGP, microfractures are widely developed, with widths less than 0.5 mm. Among them, fractures developed along stylolites stand out, with widths not exceeding 1–2 mm [1].

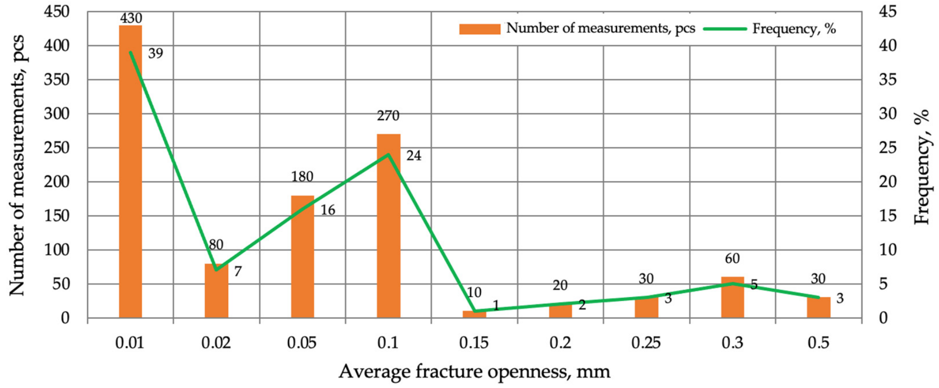


Figure 7. Assessment of fracture openness based on the analysis of 1110 carbonate core samples from one field of the VUOGP.

2.5. Conducting Filtration Tests of the Blocking Composition BHES-MC on Natural Core Samples with a Fracture

2.5.1. Preparation for Filtration Tests

For laboratory filtration tests, samples of natural core material from the VUOGP, measuring 6.45 cm in length and 3 cm in diameter, were prepared in accordance with regulatory documents [51–54], featuring artificially simulated fractures.

During filtration tests, three fracture widths were modeled: 0.1, 0.5, and 1.5 mm. A fracture width of 0.1 mm was simulated using two aluminum foil shims, each 0.1 mm thick and 5 mm wide, placed on the inner side of the natural core sample (Figure 8). Fracture widths of 0.5 and 1.5 mm were modeled using specially prepared metal plates of the corresponding thickness and 5 mm width, positioned on the inner side of the core.

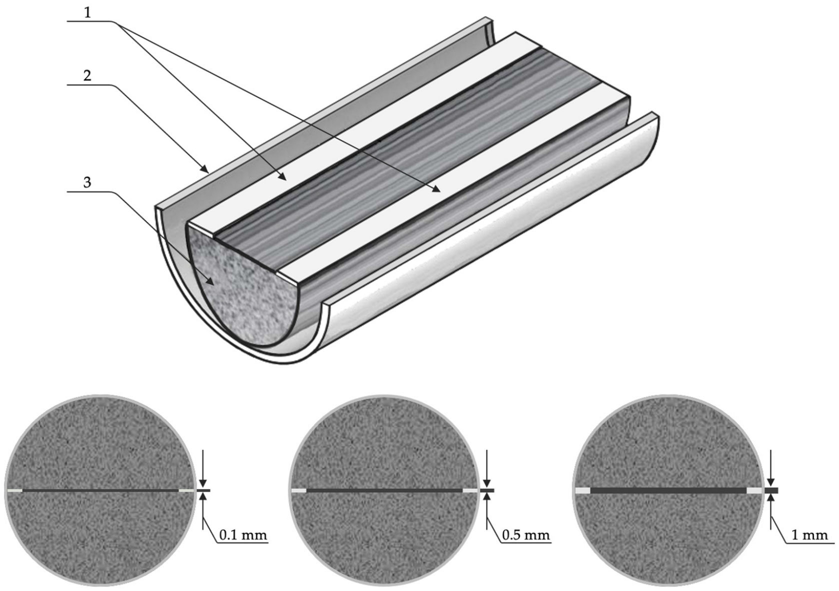


Figure 8. Appearance of a natural core in cross-section with fractures of varying degrees of width: 1—aluminum foil shims for 0.1 mm fractures or metal plates for 0.5 and 1.5 mm fractures; 2—plastic heat-shrink tubing; 3—natural carbonate core sample.

To conduct filtration studies with high-viscosity fluids containing a filler, modifications were made to the design of the standard core holder: tubes with an increased diameter of the flow passage (4.5 mm) were used in the "cylinder-reservoir—core holder—measuring capacity" system. Steel rings were placed before and after the core, facilitating unimpeded flushing of the core face even in the presence of a solid phase with a large particle size in the fluid. This core holder design also permits testing cross-linked polymer systems [10] without the risk of clogging the filtration tubes (Figure 9).

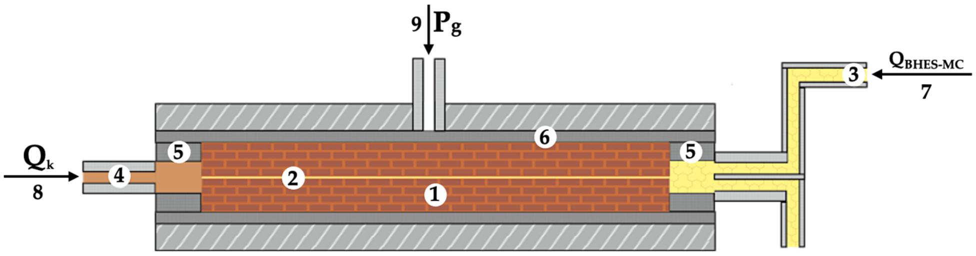


Figure 9. Core holder scheme: 1—natural core sample; 2—fracture; 3—blocking composition BHES-MC; 4—kerosene; 5—buffer metal rings; 6—rubber collar for core compression; 7—filtration direction of the composition BHES-MC; 8—filtration direction of kerosene; 9—fluid injection direction for creating compression pressure.

Prepared core samples were vacuum-saturated with kerosene and placed in the core holder (Figure 10) of the filtration unit FDES-645 (Formation Damage Evaluation System) by Coretest Systems (Morgan Hill, CA, USA). The tests were conducted under thermobaric conditions characteristic of the VUOGP.

Figure 10. Core holder and oven of the filtration unit FDES-645.

Figure 11 shows a schematic diagram of the operation of stand for conducting filtration tests in modeling the processes of well killing and oil well development using developed blocking compositions.

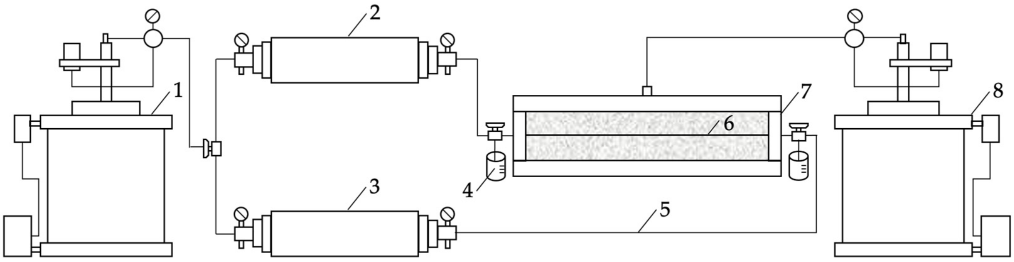


Figure 11. Schematic diagram of the stand for conducting filtration tests in the simulation of the well-killing processes and oil well development: 1—injection pump; 2—cylinder with kerosene; 3—cylinder with a developed blocking composition; 4—measuring capacity; 5—alloy steel tubes; 6—fracture; 7—core holder; 8—compression system.

2.5.2. Methodology for Conducting Filtration Tests

Assessment of the filtration properties' effectiveness of the investigated compositions is based on determining the permeability changes in reservoir models (cores) with fractures of varying degrees of width (0.1, 0.5, and 1.5 mm). The main objective of the research was to compare compositions "with" and "without" fillers in modeling the processes of well killing and oil well development.

Filtration tests were conducted using the FDES-645 unit in conditions closely approximating the average reservoir conditions of the VUOGP:

- repression during well-killing modeling—1 MPa (5% of reservoir pressure);
- reservoir pressure—20 MPa;

- average reservoir temperature of the VUOGP—37 °C.

The filtration rate (flow rate) value was set at 10 cm^3/min. Kerosene with a viscosity of 0.8 MPas was used as the saturating fluid.

The repression value of 1 MPa during well-killing modeling reproduced the condition of creating a liquid column of hydrostatic pressure at the wellhead in accordance with the requirements.

The result of the laboratory test is the determination of

- the volume of blocking composition filtered into the core at given pressure drop values during well-killing modeling;
- initial shear pressure gradient of the blocking composition in the fracture during well-development modeling;
- the coefficient of relative permeability changes in the core samples (with fractures) by kerosene after modeling well killing and well development processes.

The injection and filtration direction of the investigated compositions in the core corresponded to the direction of fluid and technological fluid movement in oil wells. Accordingly, direct filtration described the process of oil inflow from the reservoir into the well, i.e., the well development process. Reverse filtration modeled the well-killing process, which, in turn, involved creating a constant pressure drop.

Laboratory filtration tests were carried out in four stages:

1. Determination of the initial core permeability for kerosene. Prepared samples of natural core with a fracture saturated with kerosene were placed in a core holder FDES-645, where thermobaric conditions were created, closely approximating the averaged reservoir conditions of the VUOGP. Subsequently, filtration through the kerosene-saturated core was conducted. The initial phase permeability of the core to kerosene was measured in constant flow conditions until the pressure gradient stabilized at reservoir temperature. The filtration direction was "direct."
2. Modeling the process of closing the interval of perforations in the reservoir zone of an oil well with the investigated composition. In the constant pressure drop conditions, the investigated composition was injected into the core in the "reverse" filtration direction for 30 min, and the volume of the injected composition was recorded. Afterward, the core was left at rest for 1 h.
3. Modeling the process of treating the bottomhole formation zone with a hydrochloric acid composition. This stage of work was performed only for cases where the injection of the acid composition led to the formation damage of the bottomhole zone, resulting in a corresponding reduction in the coefficient of relative permeability change of the core after well development. The hydrochloric acid composition was injection at a constant volumetric flow rate of 0.5 cm^3/min. The amount of injected hydrochloric acid composition was set at 6 cm^3.
4. Modeling the process of oil well operation after the well killing and development operations. After the core had been at rest for a specified period, the restoration of kerosene filtration in the "direct" direction was carried out in constant flow conditions until the pressure gradient stabilized. The initial shear pressure gradient of the investigated composition in the core with kerosene was recorded.

The processing of the results involved the following steps:

- Pressure gradients were determined before and after the injection of the investigated composition into the core, based on which phase permeability coefficients for kerosene were calculated before and after injection.
- The initial shear pressure gradient of the investigated composition in the core with kerosene was recorded during the modeling of the well development process.
- The coefficient of relative change in the permeability of the core after its treatment with the investigated composition was calculated according to the following Equation (4):

$$\bar{k} = \frac{k_2 - k_1}{k_1} \cdot 100, \tag{4}$$

where k—permeability change coefficient, %; k_1—permeability of the core sample before the well-killing process, m^2; k_2—permeability of the core sample after the development process, m^2.

A positive value of the coefficient of relative permeability change indicated an increase in the permeability of the core sample after the well-killing process, while a negative value indicated a decrease in permeability.

The main controlled parameter during the experiments was the change in pressure drop, based on which the change in kerosene mobility as a result of pumping the investigated composition was determined.

In accordance with the key requirements imposed on fluids for well killing before workover operation in fractured carbonate reservoirs, a comprehensive research program on the technological properties of created blocking compositions was developed. The studies of blocking compositions, including physico-chemical, rheological, and filtration characteristics, were carried out using modern high-precision equipment.

To evaluate the blocking ability of the developed compositions, the author-designed stand was used, created for modeling a fractured reservoir using a high-pressure filter press. This equipment has been modernized and adapted for more precise investigation of the blocking screen quality.

Experiments to evaluate the filtration properties of the compositions were carried out on a special filtration unit in accordance with the developed methodology. They were based on the use of natural core samples with artificially simulated fractures, bringing the conditions as close as possible to reservoir conditions.

These results highlight not only the comprehensiveness of the research program but also the importance of the methods used for a more precise understanding of the properties of the developed blocking compositions in the context of fractured carbonate reservoirs.

3. Results

Based on the analysis of literary sources, the main requirements for blocking compositions can be formulated as follows:

- manufacturability in preparation, storage, and use;
- accessibility and low cost of composition components;
- non-toxicity of applied reagents;
- thermal and sedimentation stability in a wide range of reservoir temperatures;
- adjustable density and viscosity;
- low corrosive activity;
- effective blocking of pores and filtration channels;
- ability to prevent fluid loss of technological fluids into the productive formation;
- possibility of destruction;
- insignificant penetration into the bottomhole formation zone and maximum preservation of its reservoir properties.

The developed blocking composition is a thermally stable reverse oil-in-water emulsion stabilized by an emulsifier reagent, which is a mixture of reaction products of amines and amino alcohols with fatty acids. To enhance the blocking ability, a multi-fraction mineral filler based on calcium carbonate (marble chips) is introduced into the emulsion composition. The developed composition is conditionally named BHES (Blocking Hydrophobic Emulsion Solution), and when used with marble chips, it is called BHES-MC. After preparation, these compositions are stable, homogeneous, thickened liquids of light brown color with adjustable viscosity and density values.

The initial components used in formulating the developed emulsion technological fluids were as follows: hydrocarbon phase—light oil from the VUOGP; aqueous phase—aqueous solutions of calcium chloride; surfactant—emulsifiers of various brands and manufacturers; filler—marble chips. The components of the BHES-MC composition are available, cost-effective, and non-toxic reagents of domestic production.

The primary task in developing formulations for blocking compositions was the selection of the type and optimal concentration of the emulsifier reagent, which influences the thermal and aggregative stability of emulsions.

As a result of measuring interfacial tension at the interface "oil with emulsifier—aqueous solution of calcium chloride," the three (out of 20 investigated) most effective emulsifiers were selected (based on the lowest interfacial tension values at a surfactant concentration of 0.5% wt.) under the conditional numbers: E-09, E-14, and E-20. Subsequently, based on the selected emulsifiers, the value of their optimal concentration in oil was assessed in the range from 0.05 to 5% wt. The obtained results (Figure 12) showed that the critical micelle concentration (CMC) range is within the emulsifier content in oil from 0.5 to 1.5% wt. In comparison with other types of emulsifiers, their application, under equal conditions, will contribute to the creation of an emulsion with greater thermal and aggregative stability. This is due to the fact that low interfacial tension at the phase boundary is one of the most important conditions for obtaining a stable emulsion.

In selecting the optimal ratio of initial reagents for the developed emulsion blocking compositions, three selected emulsifier brands were used with concentrations of 0.5, 1, and 1.5% wt. During the series of laboratory experiments, the following ratios of aqueous and hydrocarbon phases in the emulsion were chosen: 70/30, 80/20, and 90/10% wt., respectively. Thermal and aggregate stability at 90 °C were determined for all prepared compositions.

The results of the thermal stability tests are shown in Figure 13. The volumes of the separated hydrocarbon phase in the emulsion solution after 7 days of thermostating are presented in the respective cells. All prepared compositions are thermally and aggregatively stable at 90 °C, indicating their potential application as blocking compositions for well killing under elevated reservoir temperatures (up to 90 °C).

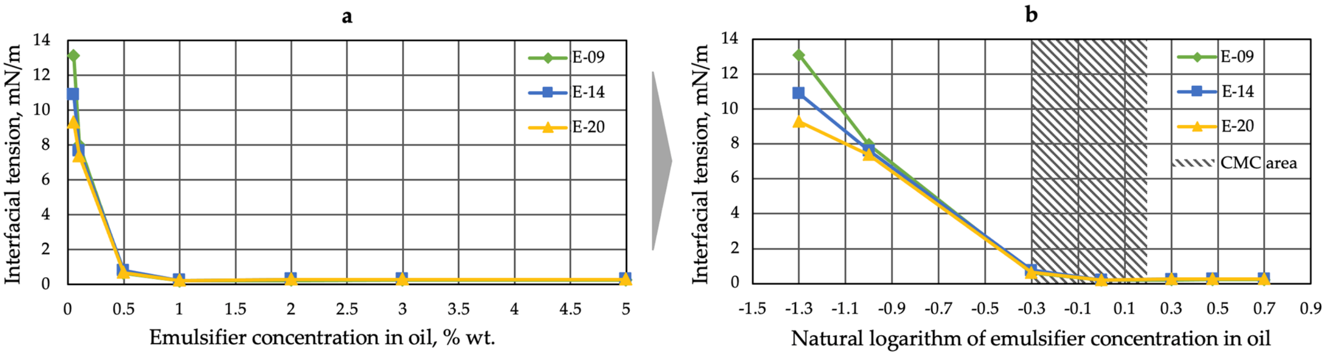
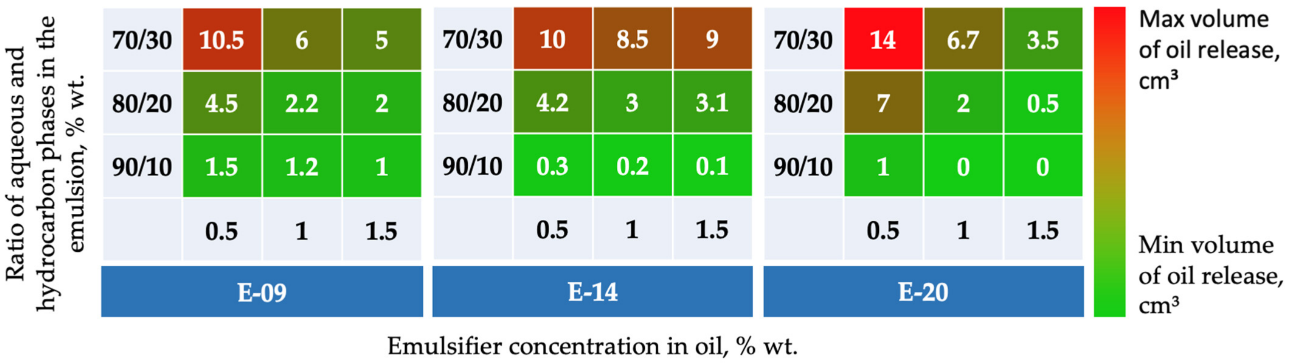


Figure 12. Results of measuring interfacial tension at the interface "oil with emulsifier—aqueous solution of calcium chloride" as a function of emulsifier concentration in oil: (**a**)—dependence of interfacial tension on emulsifier concentration in oil; (**b**)—dependence of interfacial tension on the natural logarithm of emulsifier concentration in oil.

Based on the results of the thermal stability tests at 90 °C, the optimal concentration and ratio of aqueous and hydrocarbon phases were determined for each emulsifier. A concentration of 1.5% wt. was selected for all three emulsifier types (considering the possibility of demulsifier content in oil, temperature increase, specific conditions of emulsion preparation in the field, etc.). With an aqueous-to-hydrophobic phase ratio of 80/20 and 90/10% wt., all emulsion compositions are stable. Variations in the phase ratio within these limits can be used to regulate the viscosity and density of the emulsion composition from less viscous (80/20% wt.) to more viscous (90/10% wt.).



Ratio of aqueous and hydrocarbon phases in the emulsion, % wt.	E-09			E-14			E-20		
	0.5	1	1.5	0.5	1	1.5	0.5	1	1.5
70/30	10.5	6	5	10	8.5	9	14	6.7	3.5
80/20	4.5	2.2	2	4.2	3	3.1	7	2	0.5
90/10	1.5	1.2	1	0.3	0.2	0.1	1	0	0

Figure 13. Thermal stability of emulsion compositions at different phase ratios at 90 °C after 7 days.

Based on the conducted research, it can be concluded that when using the emulsifier E-09 in the emulsion composition, the minimum amount of the hydrocarbon phase is separated compared to other emulsifier brands (E-14 and E-20).

Thus, for the preparation of BHES in further research, the emulsifier E-09 (“Yalan-E-2” brand B2) was used. This emulsifier is produced according to TS 2458-012-22657427-2000 and is a mixture of reaction products of amines and amino alcohols with fatty acids.

As a result, the following formulation for the BHES composition was recommended, % wt.: emulsifier “Yalan-E-2” brand B2—1.5; oil—18.5; aqueous solution of calcium chloride—80.

The use of the blocking composition BHES in its pure form (without a filler) does not prevent its fluid loss by the productive formation during well killing in conditions of fractured carbonate reservoirs and abnormally low reservoir pressures. Therefore, the next stage of research involved selecting the type and optimal concentration of the filler. To enhance the blocking ability of the BHES composition in practice, various types of plugging fillers are used to create a low-permeability blocking screen. An analysis of global experience in well killing with fluid-loss control has shown that the most promising fillers are marble chips, chalk, halite, and fibrous additives.

The assessment of the blocking ability of the compositions in modeling the fractured reservoir was carried out using a high-pressure filter press on the author’s developed stand, as described in the previous chapter. The studies covered the most common range of fracture width from 0.1 to 1.5 mm, corresponding to the data from numerous laboratory studies conducted using carbonate core materials.

During laboratory experiments conducted on the developed stand, it was established that for reliable blocking of fractures with a width of up to 0.5 mm, the most effective approach is to use MC-500 brand marble chips in the composition of BHES (BHES-MC-0.5). The distribution of the fractional composition ranges from 0 to 0.5 mm (Figure 14). At the same time, the most optimal concentration of marble chips in the BHES composition for this fracture width is 5% wt. It was found that at this concentration, a blocking screen is formed at the entrance to the fracture, withstanding a pressure drop of up to 8 MPa. At a lower content of marble chips in the BHES composition, breakthrough of the blocking screen occurs at a pressure drop of less than 1 MPa.

With an increase in fracture width to 1.5 mm, it is recommended to add to the BHES a mixture of marble chips MC-500 and MC-1000/1500 (BHES-MC-1.5), taken at the optimal concentration of 3% by mass for both brands, in a 1:1 ratio. The distribution of the fractional composition of this mixture ranges from 0 to 1.5 mm (Figure 14). In this case, the BHES-MC-1.5 composition contributes to the formation of a blocking screen at the entrance to the fracture, withstanding a pressure drop of up to 8 MPa.

As a result of the conducted research, the mechanism of forming a durable and impermeable blocking screen by the BHES-MC composition was established. This mechanism involves creating a hydrodynamic barrier at the entrance to the fracture in the carbonate reservoir through the correctly selected fractional composition of the filler, covering the entire range of fracture openness (from 0 to 1.5 mm). This allows withstanding high pressure drops (more than 8 MPa). The blocking screen is formed due to the arch effect of the multi-fraction marble chips. Thus, high particle compaction is achieved under the influence of frictional forces (Figure 15).

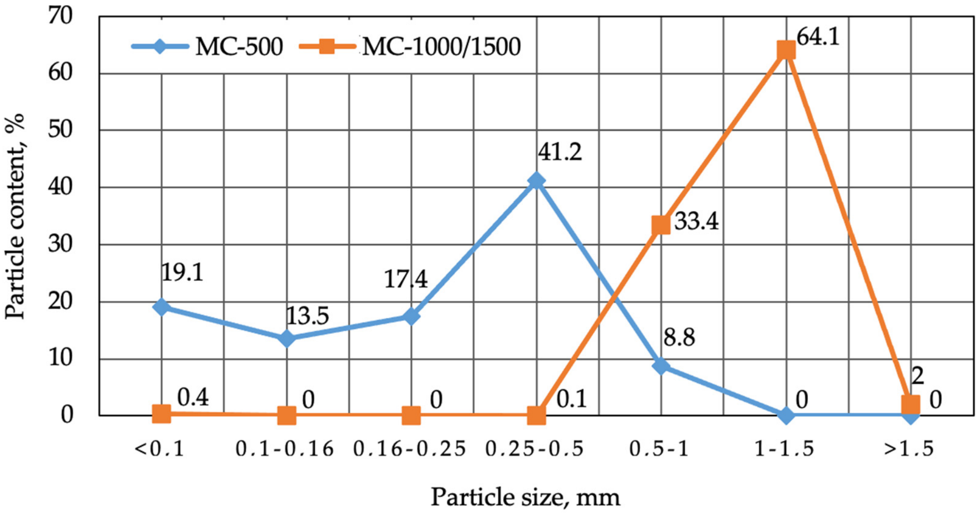


Figure 14. Distribution of the fractional composition of the filler (marble chips) in the BHES.

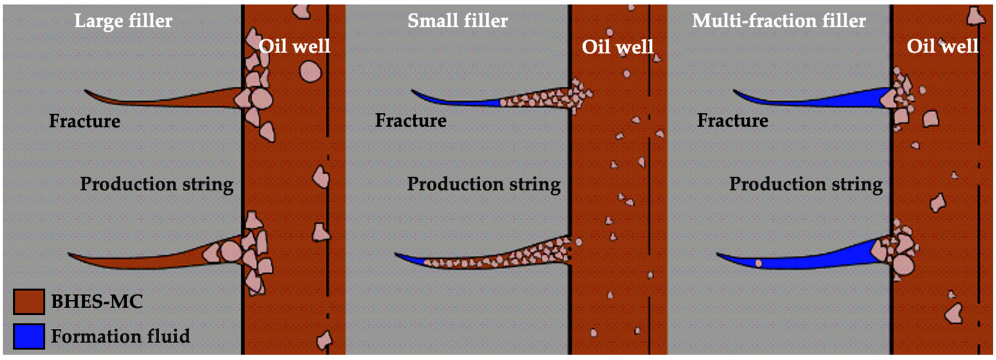


Figure 15. Mechanism of forming a blocking screen at the entrance to a fracture in the bottomhole formation zone when using fillers with various fractional compositions in process fluids.

The correct selection of the fractional composition of marble chips is important to reduce the penetration of small filler particles into the formation, which can lead to a decline in permeability and, consequently, complications during well development and production startup. On the other hand, larger filler particles are incapable of forming an impermeable blocking screen, which leads to the penetration of process fluid filtrate into the bottomhole formation zone (Figure 15).

The assessment of the sedimentation stability of BHES-MC is important to determine the ability of the dispersed system to maintain an equilibrium state throughout the volume of the dispersion medium and to identify the stability of the composition of a given phase separation. The sedimentation stability of BHES-MC was determined based on the changes in the density of the upper and lower layers of the composition after thermostating at 37 °C. After a specified time interval (7 days), a sample of the investigated composition was taken from the upper and lower parts of the test tube to measure its density using a pycnometer. The composition was considered sedimentationally stable if the difference in density between the lower and upper layers did not exceed 20 kg/m^3, representing a change of 1.5%. Thus, the sedimentation stability range was within 98.5 and 100%. In Figure 16, a dependence on changes in the sedimentation stability of the blocking composition BHES-MC is observed based on the ratio of the aqueous/hydrocarbon phases and the concentration of the filler. It was found that at an aqueous and hydrocarbon phases ratio of 80/20 and 90/10% wt., respectively, all compositions have similar sedimentation stability, indicating that the 80/20% wt. ratio is the most optimal. At this ratio, the solid particles of marble chips in the BHES composition are suspended due to the increased density and viscosity of the composition. Reducing the aqueous phase content in BHES to less than 80% wt. leads to a decrease in the density and viscosity of the solution, which impairs its sedimentation stability.

As a result, the main physico-chemical properties of the developed blocking composition BHES-MC were determined and are presented in Table 2. This composition meets the requirements imposed on technological fluids, indicating its applicability in the well-killing process in conditions of fractured carbonate reservoirs and abnormally low reservoir pressure, aiming to prevent the fluid loss of technological fluids and preserve the filtration characteristics of the bottomhole formation zone.

The study of the influence of the developed blocking compositions on the filtration characteristics of carbonate reservoirs was conducted on a specialized filtration unit, described in the previous chapter, using a modified core holder design that allows modeling fractures of different degrees of openness (0.1, 0.5, and 1.5 mm).

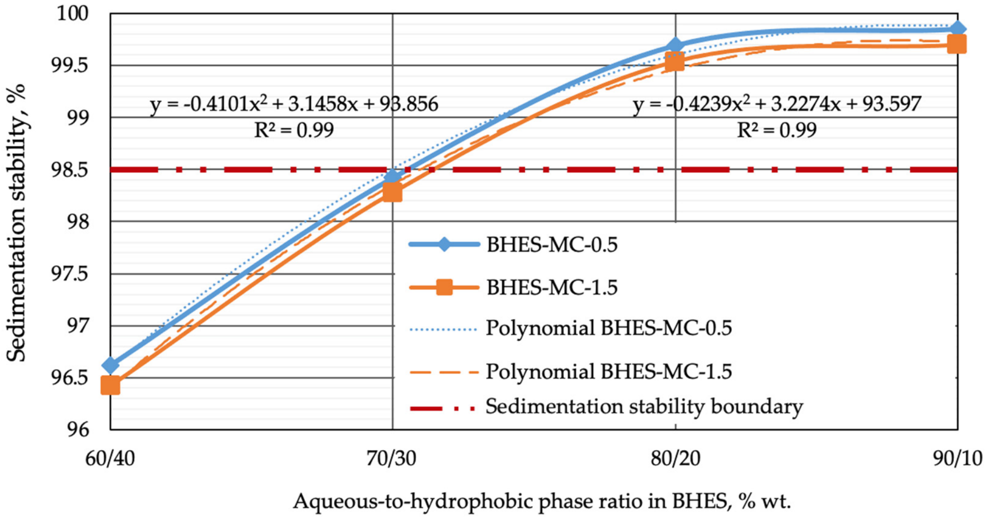


Figure 16. Dependence of sedimentation stability of BHES-MC on the ratio of aqueous/ hydrocarbon phases and filler concentration.

Table 2. The main physico-chemical properties of blocking compositions.

Parameter Name		BHES	BHES-MC-0.5	BHES-MC-1.5
Density at 20 °C, kg/m^3		1221	1287	1300
Thermal stability at 37 °C for 7 days		stable	stable	stable
Sedimentation stability after thermostating for 7 days, %		–	99.7	99.5
Electrical stability, V		367	355	351
Corrosion rate of steel grade St.20, mm/year		0.052	0.055	0.056
Pour point, °C		−21	−21	−21
Effective viscosity (D = 300 rpm), mPa·s		382	463	467
Static shear stress (D = 3 rpm), Pa	after 1 min	25.1	18.2	18.5
	after 10 min	36.6	39.4	42.1

The final results of the conducted filtration tests modeling the well-killing process and oil well development are presented in Table 3.

Table 3. Results of filtration tests of blocking compositions at a constant pressure drop (repression) of 1 MPa.

Fracture Width, mm	Composition Name	Coefficient of Relative Change in Core Permeability, %	Maximum Pressure Gradient at the Beginning of Kerosene Filtration after Composition Injection, MPa/m	Specific Volume of the Penetrated Composition in the Fracture, cm^3
0.1	BHES	−32	5.49	0.3
	BHES-MC-0.5	−2	5.07	<0.2
0.5	BHES	−93	20.9	14
	BHES-MC-0.5	−4	2.20	<0.2
1.5	BHES	−61	0.30	300
	BHES-MC-0.5	−56	0.27	300
	BHES-MC-1.5	−9	0.13	<0.2

The conducted filtration tests have shown that for conditions of a fractured carbonate reservoir, in order to prevent the fluid loss of technological fluid and maintain the filtration characteristics of the bottomhole formation zone, the use of a mineral filler (marble chips) is recommended. It is advisable to use a wider range of filler particle size with the increasing degree of natural fracture openness. Thus, with a moderate fracture openness of up to 0.5 mm, the use of the BHES-MC-0.5 is recommended. For a greater fracture openness of up to 1.5 mm, the use of the BHES-MC-1.5 is recommended. These compositions allow for the maximum preservation of the core's filtration characteristics by forming a fine-dispersed crust at its entrance from various-sized marble chips, which prevents further filtration into the fracture of the blocking composition BHES and the aqueous solution of inorganic salts. In addition to preserving filtration properties, a significant indicator of the effectiveness of the applied well-killing fluids is the low value of the pressure gradient at the beginning of kerosene filtration after well killing. This parameter characterizes the depression in the "well-bottomhole formation zone" system that needs to be created during well development to ensure the influx of reservoir fluids.

Negative values of the relative permeability change coefficient indicate the penetration of marble chips into the fracture until the formation of an impermeable blocking screen, which, in turn, leads to a deterioration of permeability. The restoration of permeability can be achieved by treating the bottomhole formation zone with a hydrochloric acid composition (12% HCl), as confirmed by the results of the filtration experiment presented in Figure 17.

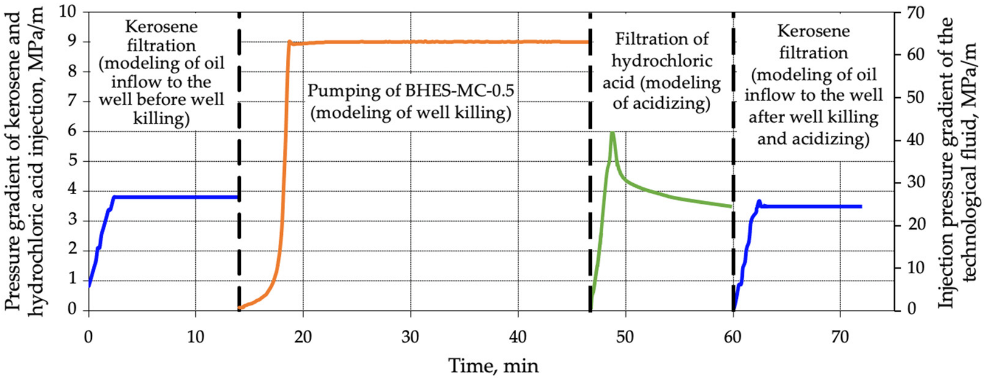


Figure 17. Results of filtration tests during modeling processes of well killing and oil well development (fracture width 0.1 mm).

Based on the presented results, it can be concluded that conducting acid treatment after the well-killing process using the BHES-MC-0.5 composition allows the following:

- improvement of the efficiency of well development by dissolving marble chips in the BHES composition and within the fracture (the maximum pressure gradient during kerosene injection after acid treatment reached 3.7 MPa/m);
- restoration and enhancement of the filtration characteristics of the bottomhole formation zone due to the impact on the fracture and matrix of the carbonate reservoir (the relative permeability change coefficient of the core was +8%).

Field tests of the blocking composition BHES-MC were carried out at the oil and gas condensate field in conditions of a fractured carbonate reservoir, abnormally low reservoir pressure, high gas factor, and long open-ended horizontal wells.

During the field test, the technology for preparing the blocking composition BHES-MC was also tested in field conditions. The following reagents and materials were used in the preparation of the blocking composition BHES-MC:

- mineralized water (saline solution) with the required density (according to the work plan)—aqueous solution of calcium or sodium chloride—78%;
- hydrocarbon phase (oil, gas condensate, diesel fuel)—10%;
- emulsifier reagent—2%;
- marble chips (ground or crushed marble), produced according to TS 5716-001-56390243-2015:
  - ground fractionated marble MC-500—3% wt.;
  - crushed marble MC-1000/1500—3% wt.

During the field testing, the following equipment was used:

- pump truck—1 unit;
- oilfield tanker—1 unit;
- on-board truck;
- 30 m^3 capacity tank—1 unit;
- crane—1 unit.

The process of preparing the blocking composition BHES-MC in field conditions, accompanying the well-killing process, and its discharge is presented in Figure 18.

Figure 18. The process of preparing the blocking composition BHES-MC in field conditions, accompanied by pilot test and well discharge.

The results of well killing using the blocking composition BHES-MC are presented in Table 4.

Analysis of the results of field tests of the BHES-MC technology on one of the oil and gas condensate fields with a carbonate reservoir showed that during the well-killing process, there is an increase in pressure when pumping the blocking composition into the bottomhole formation zone compared to a similar well treatment with a traditional emulsion composition without colmatant. This indicates the formation of a blocking screen in the fractures, contributing to preventing the fluid loss of technological fluid (Figure 19). The pressure at the pump truck at the end of the well-killing process was 12 MPa, which is 2 MPa higher compared to the previous well killing operation of the same well with an emulsion without a filler. This may indicate the formation of a filter cake of marble chips in the bottomhole formation zone.

Table 4. Results of well killing using blocking composition BHES-MC.

Parameter Name	Value
Bottomhole pressure, MPa	9.12
Vertical depth of the formation roof, m	1834
Calculated density of well killing fluid, kg/m^3	510
Current depth, m	2661
Open hole interval, m	2037–2661
Volume of blocking composition with colmatant, m^3	32
Volume of gas blocking screen (emulsion without colmatant), m^3	18
Pressure at the unit upon completion of well killing, MPa	12
Fluid level in the well after technical settling (pipe/annular), m	578/584
Presence of gas at the outlet after discharge	no
Transfer of the well to the workover team	yes

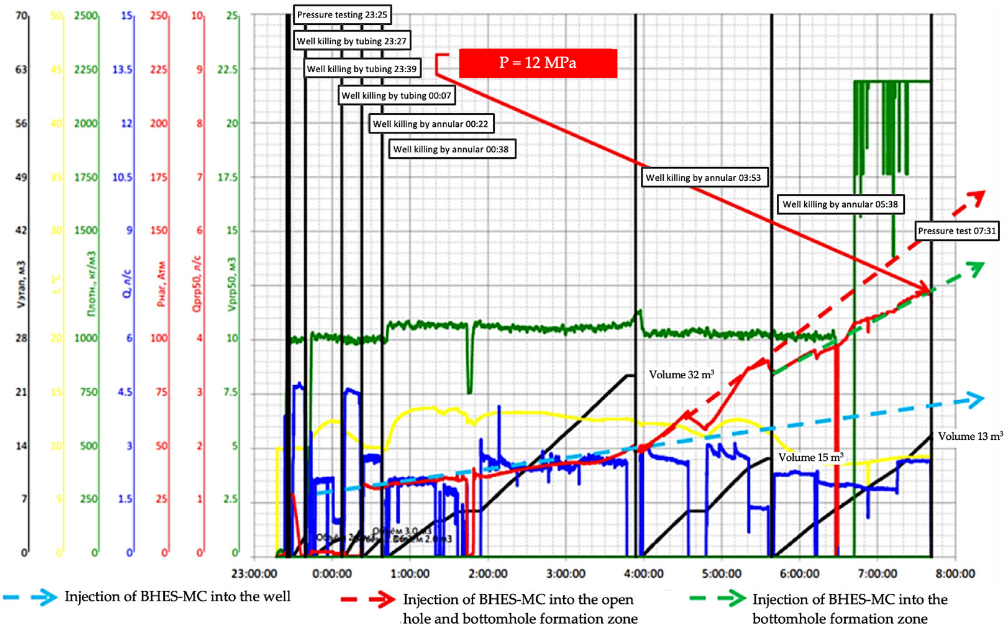


Figure 19. The dynamics of technological parameters during the well killing process using the blocking composition BHES-MC.

4. Discussion

In this section, the results of conducted investigations aimed at improving the efficiency of well-killing technology before workover operations in conditions of fractured carbonate reservoirs with abnormally low reservoir pressure are detailed and discussed. The discussion of the results is presented in the context of previous research and in consideration of the working hypotheses.

The obtained blocking composition, BHES-MC, possesses several key properties: high thermal stability up to 90 °C, sedimentation stability for 7 days at an average reservoir temperature (37 °C), adjustable density and viscosity over a wide range, and low corrosion activity.

The influence of the ratio of aqueous and hydrophobic phases, as well as the filler concentration, on the sedimentation stability of the blocking composition has been considered. At an aqueous-to-hydrophobic phase ratio of 80/20 and 90/10% wt., the composition demonstrates sedimentation stability, making it promising for use as a blocking technological fluid in the well killing before workover operations.

The mechanism of forming the blocking screen BHES-MC is described in detail, including the creation of a hydrodynamic barrier using different fractions of marble chips with fracture widths up to 1.5 mm. Such a screen can withstand pressure drops up to 8 MPa, which is extremely important in abnormally low reservoir pressure conditions.

In the context of filtration tests, the importance of the blocking composition BHES-MC in maintaining the filtration characteristics of reservoir rocks is emphasized, preventing the penetration of technological fluid into natural rocks. Particular attention is given to the possibility of restoring filtration characteristics after well killing, which is achieved using hydrochloric acid treatment.

The results of pilot tests of the BHES-MC technology on one of the oil and gas condensate fields in the VUOGP with a carbonate reservoir showed a pressure increase of 2 MPa when injecting the blocking composition into the bottomhole formation zone compared to a similar well treatment with a traditional emulsion composition without colmatant. This indicates the formation of a blocking screen in the fractures, contributing to preventing the fluid loss of the technological fluid.

The technical and economic analysis of the well-killing efficiency in complicated conditions using the blocking composition BHES-MC demonstrated a cumulative economic impact of 600 000.00 RUB (5555.00 USD) per well. Over a 4-year period for one of the oil and gas condensate fields, this amounted to a final accumulated net present value (NPV) of 80 million RUB (888,888.00 USD). The main factor of this economic effect is the prevented damage from oil production losses by reducing the number of well-killing cycles to 1, reducing the well development period, expediting its commissioning, and maintaining oil production rates.

The results obtained in this work provide a basis for further research and the development of innovative methods for well killing and workover operations, adapted to various reservoir conditions, ensuring long-term stability and operational effectiveness of oil wells.

5. Patents

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Abbreviations

BHES	Blocking hydrophobic emulsion solution
BHES-MC	Blocking hydrophobic emulsion solution with marble chips
GOST	Russian government standard
LLC	Limited liability company
TS	Technical specification
VUOGP	Volga-Ural oil and gas province

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