



Article Well Killing Technology before Workover Operation in Complicated Conditions

Dmitry V. Mardashov^{1,*}, Mikhail K. Rogachev¹, Yury V. Zeigman² and Vyacheslav V. Mukhametshin²

- ¹ Department of Development and Operation of Oil and Gas Fields, Saint Petersburg Mining University, 21 Line, 2, 199106 St. Petersburg, Russia; Rogachev_MK@pers.spmi.ru
- ² Department of Development and Operation of Oil and Gas Fields, Ufa State Petroleum Technological University, Kosmonavtov St., 1, 450062 Ufa, Russia; jvzeigman@mail.ru (Y.V.Z.); vv@of.ugntu.ru (V.V.M.)
- Correspondence: Mardashov_DV@pers.spmi.ru

Abstract: Well killing is an important technological stage before conducting workover operation, one of the tasks of which is to preserve and restore the natural filtration characteristics of the bottomhole formation zone (BFZ). Special attention should be paid to the choice of well killing technologies and development of wells in complicated conditions, which include abnormally low reservoir pressure, high oil-gas ratio and carbonate reservoir type. To preserve the filtration characteristics of the productive formation and prevent fluid losses in producing wells during well killing operation, blocking compositions are used. At the same time, an informed choice of the most effective well killing technologies is required. Consequently, there is a need to conduct laboratory physicochemical and coreflood experiments simulating geological, physical, and technological conditions of field development, as similar as possible to actual reservoir conditions. The article presents the results of experimental studies on the development well killing technologies of producing wells during workover operation in various geological, physical, and technological conditions of oil field development. Physicochemical and coreflood laboratory experiments were carried out with the simulation of the processes of well killing and development of wells in reservoir conditions with the use of modern high-technology equipment in the Enhanced Oil Recovery Laboratory of the Department of Development and Operation of Oil and Gas Fields at St. Petersburg Mining University. As a result of the experimental studies, new compositions of well killing and stimulation fluids were developed, which ensure to prevent fluid loss, gas breakthrough, as well as the preservation, restoration and improvement of the filtration characteristics of the BFZ in the conditions of terrigenous and carbonate reservoirs at different stages of oil field development. It is determined that the developed process fluids, which include surfactants (YALAN-E2 and NG-1), have a hydrophobic effect on the porous medium of reservoir rocks, which ultimately contributes to the preservation, restoration and improvement of the filtration characteristics of the BFZ. The value of the presented research results is relevant for practice and confirmed by the fact that, as a result of field tests of the technology for blocking the BFZ with the composition of inverse water-oil emulsion during well killing before workover operation, an improvement in the efficiency of wells operation was obtained in the form of an increase in their oil production rate by an average of $5-10 \text{ m}^3/\text{day}$, reducing the time required for the well to start operating up to 1–3 days and reducing the water cut of formation fluid by 20–30%.

Keywords: well killing; workover operation; well killing fluid; hydrophobic emulsion; emulsifier; well stimulation

1. Introduction

At the current stage of the oil industry's development, it is generally known that the productivity of production wells is significantly influenced by physical and chemical processes that occur when various technological operations affect the productive reservoir and the fluid that saturates it. Such processes include well killing for the period of workover operation, well completion and stimulation of production.



Citation: Mardashov, D.V.; Rogachev, M.K.; Zeigman, Y.V.; Mukhametshin, V.V. Well Killing Technology before Workover Operation in Complicated Conditions. *Energies* **2021**, *14*, 654. https://doi.org/10.3390/en14030654

Received: 10 November 2020 Accepted: 20 January 2021 Published: 28 January 2021

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). Analysis of numerous works and experience of field research carried out by leading scientists around the world (Zeigman Yu.V., Glushchenko V.N., Musabirov M.Kh., Silin M.A., Binks BP, Bridges KL, Caenn R., Chesser BG, Meehan DN, Hisham N.-E.-D., Rylance M., Warren FP and others) showed that the most promising methods for saving and improving the filtration properties of the bottomhole formation zone (BFZ) during the well killing operation are chemical. At the same time, the authors do not focus enough attention on the areas associated with the development of effective well killing technologies in conditions of fractured carbonate reservoirs with abnormally low reservoir pressure. Additionally, the mechanisms of action of various well killing fluids on the porous medium of reservoir rocks have not been fully studied. Besides, in the work carried out, there is no data on the development of highly effective thermally stable emulsifiers of inverse water–oil emulsion (IWOE), which can be used for the preparation of hydrophobic blocking compositions for well killing operations [1–8].

In this regard, the development of blocking fluids for well killing operation, ensuring the preservation and improvement of the filtration characteristics of the BFZ, and the study of the mechanism of their action on reservoir rocks, is the main focus of this work.

The process of well killing is an important technological stage preceding workover operations, one of the tasks of which is to preserve and restore the natural filtration characteristics of the BFZ. Special attention should be paid to substantiation and selection of well killing technologies and development of wells with carbonate reservoirs, as the complex structure of the fracture-porous volume with low formation reservoir properties is highly influenced by process fluids due to intensive fluid losses, which is especially relevant at abnormally low formation pressures. In addition, during the development of oil and gas fields, there are complications associated with hydrocarbon gas breakthroughs into production wells from overlying gas-saturated pay zones ("gas caps"); this often leads to the need for repeated well killing due to the low gas-holding ability of the blocking composition.

Besides geological factors complicating the process of well killing during workover operation, there are also technical and technological factors, which include:

- Open and extended horizontal section of the wellbore in the pay zone;
- Being well equipped with downhole packers;
- Acid fracturing and acid treatments.

The process of well killing before workover operation is marked by two main problems:

- Losses of process fluids in the BFZ, which leads to their increased consumption;
- Hydrocarbon gas breakthroughs into the wellbore, which leads to saturation of process fluids with gas and, as a result, to a decrease in back pressure on the formation.

In this regard, an urgent task is to select and adapt well killing technologies to difficult geological and technological conditions of oil field development. These technologies should ensure minimum penetration of well killing fluids into the pay zone, have a blocking effect on the BFZ and prevent gas filtration into the wellbore [1,3,4,9].

Analysis of existing well killing technologies [6,10–14] showed that most blocking emulsion compositions have low thermal stability at formation temperatures of more than 60–80 °C, low sedimentation stability and low blocking properties, which, in conditions of high formation temperatures and abnormally low formation pressure, leads to the loss of blocking compositions.

The analysis of oil field development shows deterioration reservoir properties of BFZ due to the negative influence of well killing fluids used in the processes of workover and operation of oil wells. The use of traditional compositions of water-based process fluids leads to a significant decrease in the permeability of the productive formation for the hydrocarbon phase and, as a consequence, to a decrease in the rate of oil production [1,10,15,16].

There are two main ways to prevent the negative impact of traditionally used process fluids on the productive reservoir:

 Mechanical, i.e., the use of technologies excluding contact of aqueous compositions of technological process fluids with BFZ rocks (downhole shut-off valves); Physical and chemical, i.e., the use of polymeric and hydrophobic chemical compositions of process fluids.

The second method is the most perspective in oil production. Reduction of filtration abilities of process fluids is most often provided by increasing their viscosity. At present, the following types of nonfilterable process fluids are widely used [1,6,17–19]:

- Polymer-based;
- Polysaccharide;
- Invert emulsions;
- Invert dispersions.

These types of process fluids are mainly used as blocking compositions located in the BFZ, preventing the penetration into the reservoir of traditional water-based well killing compositions. The use of nonfilterable process fluids also provides for an increase in the reliability of well killing in horizontal wells with high oil–gas ratio by keeping the pop-up gas bubbles in the horizontal section [10,13,20].

The experience of application of hydrophobic emulsion compositions indicates that they have the following regulated properties that favorably distinguish them from traditionally used water-based well-killing compositions: density, viscosity, thermal stability, filterability, etc. [1,10,21,22] These parameters can be regulated due to the content, mineralizatio, and type of aqueous (dispersed) phase; as a rule, aqueous solutions of inorganic salts (CaCl₂, KCl, NaCl, ZnCl₂) are used. However, when aqueous solutions of acids (for example, hydrochloric acid HCl) are used as the disperse phase, hydrophobic emulsions can act as stimulating compositions, improving conditions of oil flow to the producing well bottoms [23–25].

The results of numerous studies have shown that for the preparation of high-quality emulsion compositions, it is necessary to use an effective emulsifier reagent. However, the emulsifier reagents on the market have many disadvantages [10,23,26]:

- Low aggregative stability;
- Low thermal stability.

In this regard, there is a need for research aimed at the development of new emulsifier reagents, hydrophobic compositions of well killing fluids, and technologies for their use to preserve, restore and improve the filtration characteristics of the BFZ during workover operation, which are considered relevant in the oil and gas industry.

To substantiate the application of blocking compositions in specific geological and technological conditions of field development, it is necessary to carry out a set of works, including physicochemical and coreflood laboratory experiments. Conducting a full range of laboratory tests helps assess the possible impact of process fluids on well productivity, as well as to identify compositions that provide the best technological indicators of the well killing process [25–28].

Process fluids used during well killing in complicated conditions should prevent [29–32]:

- Loss of process fluid by the pay zone or its saturation with filtrate;
- Formation of insoluble precipitation in the bottomhole zone as a result of the process fluid contact with formation fluids and temperature changes;
- Swelling of clay material in the formation as a result of interaction with process fluid filtrate;
- Colmatation of formation pores by particles of the solid phase of process fluid.

Non-compliance with these requirements may result in difficulties during well development and complications during the well operation, which may cause reduced oil recovery.

2. Materials and Methods

Experimental laboratory tests were conducted using modern high-technology equipment in the Enhanced Oil Recovery Laboratory of the Department of Development and Operation of Oil and Gas Fields at St. Petersburg Mining University:

- Investigations of the rheological properties of inverse emulsion compositions were carried out on a rotational viscometer Rheotest Rn 4.1 of the company Messgerate Medingen GmbH (Germany) at various temperature conditions.
- Coreflood tests were carried out in thermobaric reservoir conditions using natural core samples on the formation damage evaluation system FDES-645 unit from company Coretest Systems Corporation (USA).
- Evaluation of the hydrophobic effect was carried out using the TGC-764 gravimetric capillary pressure measurement system from company Coretest Systems Corporation, etc.

The main objectives of the research were:

- 1. Physical and chemical studies of blocking compositions on the determination of: appearance; density; thermal stability; corrosion rate; time and degree of destruction; mixing (solubility) with produced fluids, solutions of sodium, potassium and calcium chlorides.
- 2. Rheological studies including measurements of effective viscosity of compositions at different shear rates, as well as determination of gel formation time.
- 3. Coreflood studies of blocking compositions: assessment of blocking and filtration properties.

Experimental studies were conducted in accordance with the current state and industry standards.

The initial chemical reagents used in the development of new compositions of process fluids were non-ionic synthesized surfactants provided by Sintez-TNP LLC (Russia, Republic of Bashkortostan, Ufa):

- Emulsifier YALAN-E2;
- Oil-wetting agent NG-1.

When preparing inverse emulsions, the following ratio of components was used % wt.:

- Emulsifier YALAN-E2—1–5;
- Hydrocarbon fluid (summer/winter diesel fuel)—27–45;
- Aqueous solution of hydrochloric acid (20% HCl) or aqueous solution of calcium chloride (10–40% CaCl₂)—the rest.

Studies of aggregative and thermal stability have shown that the type of diesel fuel (summer and winter) does not affect the characteristics of the prepared emulsions.

Microcalcite (MC) of MC-500, MC-1000 and MC-1500 grades—3% wt. was used as a bridging agent–colmatant (in equal parts).

When preparing a water-based hydrophobic composition, the following ratio of components was used, % wt.:

- Oil-wetting agent NG-1—1;
- Technical water—the rest.

The composition was prepared on a propeller-type mixer with vigorous stirring (5000 rpm) for 5 min.

Laboratory research aimed at the development of inverse emulsion and hydrophobic compositions based on surfactant data, namely [1]:

- Blocking composition of the IWOE stabilized by the emulsifier YALAN-E2 (to preserve the filtration characteristics of terrigenous reservoir rocks);
- Blocking composition of inverse water-oil emulsion with colmatant (IWOE-C) stabilized by YALAN-E2 (to prevent fluid loss during well killing operation with carbonate reservoir);
- Stimulation composition of inverse acid–oil emulsion (IAOE) stabilized by YALAN-E2 (to improve filtration characteristics of carbonate reservoirs);
- Hydrophobic composition in the form of water dispersion HG-1 oil-wetting agent (to restore filtration characteristics of terrigenous reservoir rocks).

The aggregate stability of emulsions was determined in a heat-resistant sealed measuring tank of 25 cm³. The measuring tank was placed in a heating chamber with a temperature of 80 °C and kept for 10 days. Every 24 h, the amount of the separated aqueous phase was visually determined. For a stable (aggregate stability = 100%) emulsion system, the sediment value of the lower layer (aqueous solution of CaCl₂ (or HCl)) should be zero.

The dynamic viscosity of the emulsions was determined on a Rheotest Rn 4.1 rotary viscometer in the temperature range of 20–80 $^{\circ}$ C at a shear rate of up to 73.2 s⁻¹.

Corrosion activity was determined according to the standard method using St-3 metal plates. Steel plates measuring $50 \times 20 \times 20$ mm were carefully cleaned. Then, the total area of its surface was determined. After that, the plates were washed with water, alcohol, and acetone, dried for 2–3 min in the air, and then weighed. The corrosion rate was determined from the difference in the masses of the plates.

The study of the rate of interaction of the acid composition with the carbonate rock was based on the gravimetric determination of the mass of the rock remaining after interaction with acid for a certain period of time. A polished marble cube with a predetermined surface area was placed in a glass beaker and weighed. After that, 25 cm³ of the studying acid composition was added to a glass with a cube. To observe the dynamics of interaction, 4 time intervals of interaction with the rock were used (0.25; 0.5; 1; 2 h). The rate of interaction of the acid composition with the carbonate rock was determined by the difference in the masses of the cubes.

The study of the effect of the water hydrophobic composition on the capillary pressure of the reservoir rock was determined using the TGC-764 system from company Coretest Systems Corporation (Figure 1).

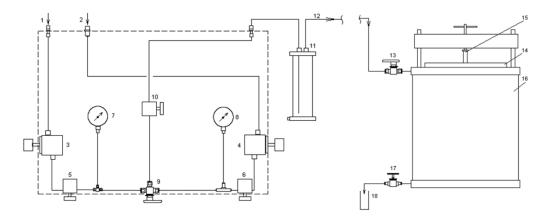


Figure 1. Schematic diagram of the system for measuring the capillary pressure of natural cores TGC-764: 1 (2)—gas inlet to the high (low) pressure line; 3 (4)—pressure regulator in the high (low) pressure line; 5, 6—valves that cut off the high (low) pressure line; 7, 8—current pressure sensors in the high (low) pressure line; 10—isolating valve; 11—gas humidifier; 13—pressure valve at the entrance to the group measuring chamber 16; 14—chamber cover; 15—clamp of the chamber cover; 17—external valve (fluid outlet from the membrane); 18—receiving container for draining.

Before starting the measurements, the samples were 100% saturated with mineralized water. Then, the core samples were placed inside the measuring chamber on a semipermeable membrane designed for 15 atm. The membrane was preliminarily saturated with the same fluid as the core samples. Then, the required pressure was created in the chamber. The pressure in the chamber ensured the displacement of fluid from the core. To plot the dependence of the capillary pressure on the residual water saturation of the core, six points of pressure values were used: 0.07; 0.2; 0.7; 1.7; 3.4; 6.5 atm. After that, an aqueous hydrophobic composition was injected through the studied core samples, and the experiment to determine the capillary pressure was repeated again.

Based on the measured and calculated data, graphs of the dependence of capillary pressure on the saturation of core samples with mineralized water were built before and after pumping an aqueous hydrophobic composition.

Coreflood tests were carried out in thermobaric reservoir conditions using natural core samples on the formation damage evaluation system FDES-645 unit from company Coretest Systems Corporation (Figure 2).

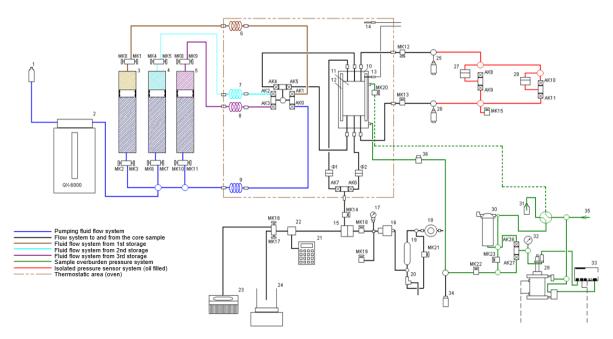


Figure 2. Schematic diagram of the equipment for evaluating the quality of formation damage FDES-645.

The effectiveness of the studied well killing fluids was evaluated based on the results of their influence on the permeability of water-saturated and oil-saturated cores before and after treatment.

The permeability coefficient was calculated based on the Darcy formula:

$$\mathbf{k} = (\boldsymbol{\mu} \times \mathbf{L} \times \mathbf{Q}) / (\mathbf{S} \times \Delta \mathbf{P}), \tag{1}$$

where k is core permeability coefficient, μm^2 ; fluid viscosity, μ -mPa·s; L is core length, m; Q is specified fluid flow rate through the core, m^3/s ; S is cross-sectional area of the core sample, m^2 ; ΔP is pressure ratio at the ends of the core sample at a given flow rate, MPa.

The research was carried out at a temperature of 80 $^{\circ}$ C, reservoir pressure of 25 MPa and rock pressure of 27 MPa. The research was carried out on the basis of the principle: constant flow rates–changing pressure ratio.

At the first stage, the initial permeability of the sample for the hydrocarbon phase (oil) was measured. At the second stage, the well killing process was modeled. Injection was carried out into the core of the studied well killing fluid in the direction opposite to the oil filtration in the amount of 1 pore volume. At the third stage, the well development process was modeled by measuring the permeability of the sample for the hydrocarbon phase (oil).

The return permeability (RP) of the core sample after exposure to the well killing fluid was determined by the following formula:

$$K_{\rm RP} = (k_2/k_1) \times 100, \tag{2}$$

where K_{RP} is return permeability, %; k_1 is core sample permeability to the well killing process, μm^2 ; k_2 is core sample permeability after the well killing process, μm^2 .

The efficiency of the intensifying composition of the IAOE was evaluated on bulk core models using a coreflood unit, the schematic diagram of which is shown in Figure 3.

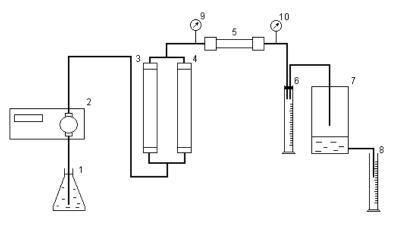


Figure 3. Schematic diagram of a coreflood unit for the study of stimulation compositions: 1—container with pumping liquid (distilled water); 2—pump; 3, 4—piston accumulators with diesel fuel and acid composition; 5—core holder; 6, 8—easuring cylinders; 7—gas meter; 9, 10—pressure sensors at the inlet and outlet of the core holder.

The prepared core sample was saturated under vacuum with oil (or formation water model). After determining the initial permeability of the core in the opposite direction, the acid composition was injected in the amount of one pore volume. During the injection process, the volume of evolved gas (CO_2) was recorded. Then, the final permeability of the core was determined.

Enhancement ratio (ER) was determined by the formula:

$$ER = (k_2/k_1) \times 100, \tag{3}$$

where ER is enhancement ratio of the acid composition, %; k_1 is core sample permeability to the enhancement process, μm^2 ; k_2 is core sample permeability after the enhancement process, μm^2 .

When measuring the main technological parameters of well killing fluids and stimulation of wells, systematic (instrumentation error) and random ("human" factor, instability of inverse emulsion compositions in time) errors were identified.

The error of the instrumentation used in research is $\pm 1-3\%$. Random errors during research are $\pm 3-5\%$. The value of the permissible error in the case of workover operations (well killing) is usually taken equal to 5-10%.

Thus, systematic and random errors are within acceptable limits.

3. Results

The primary task when developing formulations of new compositions of process fluids was to determine the optimal concentration of surfactants. The results of the conducted researches have revealed the ability of YALAN-E2 emulsifier to form aggregately stable systems not only on the basis of aqueous solutions of salts but also of acids. Thus, the use of surfactants of this type is possible as a reagent emulsifier both in technologies of preservation and improvement of filtration characteristics of the BFZ with terrigenous (application of IWOE) and carbonate (application of IWOE-C and IAOE) reservoirs. It should also be noted that compositions of inverse emulsions stabilized with reagent emulsifier YALAN-E2 have high thermal stability, i.e., 100% aggregation stability at 80 °C, which makes it possible to recommend this type of surfactant for use in conditions of increased reservoir temperatures, in particular, in the fields of Western Siberia.

The study of the influence of the concentration of reagent emulsifier on the aggregative stability of IWOE showed that its optimal content in the volume of emulsion is 3%. At the same time, IWOE (and IWOE-C) maintains 100% aggregative stability for more than 10 days (Figure 4), which corresponds to the average duration of workover operations. The optimal concentration of reagent emulsifier in IAOE was 1% vol. In this case, the emulsion composition is stable within 24 h. It is enough for operations on its injection and pressing into the reservoir. At the same time, according to the research results, it was revealed that the type of diesel fuel (summer and winter) does not affect the aggregate stability and thermal stability of emulsion compositions. In this regard, in further studies, a summer type of diesel fuel was used.

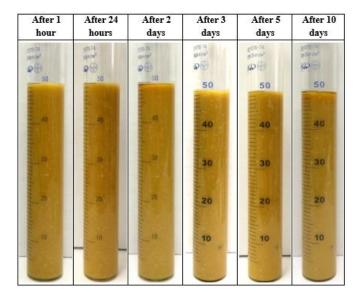


Figure 4. Aggregative stability inverse water-oil emulsion with colmatant (IWOE-C).

The increased thermal stability of inverse emulsions (with the presence of reagent emulsifier YALAN-E2) is explained by the high surface activity of this reagent, which is confirmed by the results of measurements of its inter-phase tension (Figure 5).

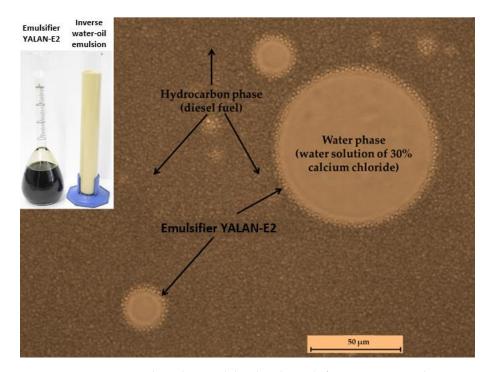


Figure 5. Inverse water-oil emulsion stabilized with emulsifier YALAN-E2: Yalan-E2—3% wt., diesel fuel (summer)—27% wt., water solution of 30% calcium chloride—70% wt.

One of the main advantages of the developed emulsion compositions over traditionally used water-based process fluids is the possibility to regulate their technological properties by changing the number and type of their dispersed (water) phase. Thus, the density of IWOE is a controlled value and can vary within a fairly wide range (0.950–1.420 g/cm³). When the content of the disperse phase in the compositions varies from 50 to 70% vol., the viscosity of emulsions varies in a wide range (200–3000 mPa·s at shear rates 14.6–73.2 s⁻¹), which allows to regulate the degree of their penetration into the reservoir depending on the purpose of treatment [33].

Research of corrosion activity of the developed compositions IWOE, IWOE-C and IAOE (Figure 6) has shown their high protective properties in relation to metals in comparison with traditionally applied process fluids on water basis. According to the analysis of the results of laboratory tests, emulsion compositions have lower corrosion rate in comparison with aqueous solutions of $CaCl_2$ and HCl (IWOE by 3 times and IAOE by 30 times). The similar effect is explained by the fact that the dispersion medium of these compositions is a hydrocarbon liquid (oil), which, in contact with a metal surface, reduces the degree of interaction of the dispersion phase of the emulsion (aqueous $CaCl_2$ or HCl solutions) with metal. The protective effect of the compositions is enhanced by the presence of surfactants based on YALAN-E2.

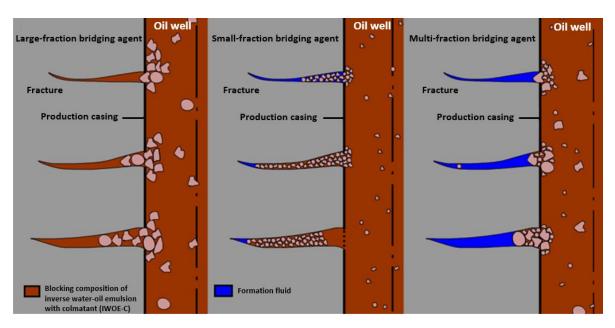


Figure 6. Corrosion studies of inverse water-oil emulsion (IWOE) and IWOE-C compositions.

To study the influence of IWOE composition stabilized by reagent emulsifier YALAN-E2 on filtration characteristics of reservoir rocks, laboratory tests with simulation of the process of "well killing and development" under thermobaric conditions with the use of natural core samples of terrigenous reservoirs were performed. The studies were carried out on an FDES-645 coreflood unit from company Coretest Systems Corporation. Experimental studies have confirmed the negative effect of the CaCl₂ aqueous solution on the filtration characteristics of the BFZ, which manifested itself in the reduction of permeability of the core sample by the hydrocarbon phase (the return permeability (RP) after treatment was 20–40%).

Meanwhile, the composition of IWOE stabilized by the reagent-emulsifier YALAN-E2 showed hydrophobic properties when entering into the porous medium of the reservoir rock, which manifested itself in the preservation of permeability of the hydrocarbon phase (RP amounted to 80–100%) and an increase in filtration resistance relative to the water phase (RP was 50% on average).

Loss of well killing fluids in the BFZ can be significantly reduced, and its reservoir properties preserved to a greater extent, by introducing acid-soluble bridging agents, which reduce the depth of penetration of aqueous filtrate and polymer molecules into the reservoir. In this connection, studies have been carried out to select the concentration and fractional composition of microcalcite for the use in IWOE stabilized reagent emulsifier YALAN-E2. The role of multifractional microcalcite is to create arched jumpers on the filtration surface of the reservoir (on fractures). As a result, rapid and effective plugging of the pore (fracture) mouth occurs and a strong layer of dispersed particles is formed, which prevents deeper



penetration into the reservoir not only of the solid phase, but also of the filtrate of the blocking composition, even under increased repressions (Figure 7).

Figure 7. Scheme of the mechanism of filtration crust formation in application of the IWOE-C well killing fluid.

According to the results of the filtration studies, the optimal concentration of microcalcite in the IWOE was revealed—3% wt. It is recommended to use a mixture of microcalcites (MC) of the MC-500, MC-1000 and MC-1500 grades—3% wt. (in equal parts). The resulting IWOE-C mixture is capable of creating a blocking screen at the entrance to a fracture with an opening of 1.5 mm (Figure 8).



Figure 8. Appearance of a fine filter cake ("bridge") above the fracture after IWOE-C filtration: 1—integral part of the filter cake; 2—the destroyed part of the filter cake during draining.

The results of laboratory coreflood tests showed that the addition of microcalcite to the blocking composition of IWOE allows to increase the recovery factor of core permeability. This is explained by the formation of a finely dispersed jumper of microcalcite at the core entrance, which prevents further filtration into the fracture of the emulsion composition. Thus, the use of multifractional microcalcite in the IWOE composition will help to preserve the natural permeability of the carbonate reservoir.

The influence of the IAOE composition stabilized by the reagent emulsifier YALAN-E2 on the porous medium of the reservoir rock was estimated by the results of coreflood tests in its interaction with the bulk model of carbonate rock. The results of the studies showed that the developed composition, as compared to the traditionally used aqueous HCl solution, slows down the interaction rates of its disperse phase (aqueous HCl solution) with hydrocarbon- and water-saturated reservoir rock samples by 2–3 times (Figure 9).

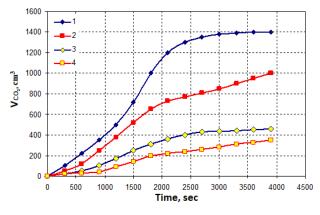


Figure 9. Dynamics of change of CO_2 excreted volume at interaction of acid composition with carbonate rock model: (1, 2) 20% aqueous solution HCl in interaction with water- and hydrocarbon-saturated porous medium; (3, 4) inverse acid-oil emulsion (IAOE) (emulsifier—1% vol., diesel fuel (summer)—49% vol., 20% aqueous solution HCl—50% vol.) in interaction with water- and hydrocarbon-saturated porous medium.

For the fields at the late stage of development with high water-cut content of the well products, it is not expedient to use hydrophobic emulsion compositions as in this case; it is necessary to reduce water-cut content and improve conditions of the oil flow to the well bottom, i.e., to restore BFZ filtration characteristics. To solve this problem, research was carried out to develop an aqueous-based hydrophobic composition (AHC), which is aqueous surfactant dispersion (HG-1 oil-wetting agent). Laboratory tests showed that the optimal concentration of surfactants in this composition is 1% vol. The mechanism of influence of the developed composition on the porous medium of the reservoir rock was studied by measuring the capillary pressure in the pores of natural core of terrigenous sediments.

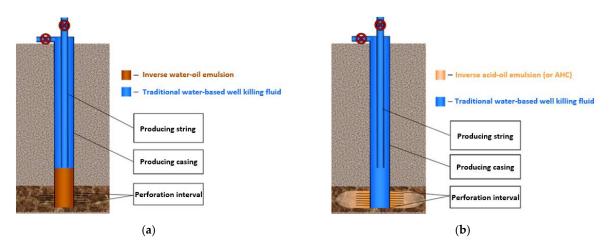
According to the results of studies carried out using the TGC-764 unit (Coretest Systems Corporation), the capillary pressure of the water-saturated core sample increased by 1.5–2.0 times after treatment with the composition, which indicates an increase in filtration resistance in relation to the water phase. The obtained effect is explained by the process of reservoir hydrophobization, in particular, by the change in wettability character of the porous core medium under the action of reagent. Application of this hydrophobic composition as an AHC before workover operation will allow reducing water-cut of produced products and increasing well flow rates for oil.

4. Discussion

Thus, to increase the efficiency of production well operation, new compositions of process fluids for directed regulation of BFZ filtration characteristics during workover operation are recommended. The developed compositions have hydrophobic properties, which distinguishes them from traditionally used water-based systems.

The technology of application of blocking compositions IWOE and IWOE-C consists in their placement opposite to the perforation interval (Figure 10a), while compositions IAOE and AHC shall be pressed in the BFZ (Figure 10b).

Taking into account the high frequency of workover operation (average 1 well workover for 1–1.5 years), a new approach to solving the problem of preservation, restoration and improvement of BFZ filtration characteristics was proposed. The essence of this approach is the combination of each well killing operation before workover with the effect of developed



hydrophobic compositions of process fluids on BFZ. Information on technologies and areas of effective application of these compositions is presented in the Table 1 below [1].

Figure 10. Well killing technology scheme: (**a**) with the blocking composition IWOE (or IWOE-C); (**b**) with stimulating composition IAOE (or AHC).

| Table 1. Technologies of application of the developed compositions of process fluids. |
|--|
|--|

| Indicators | Developed Composition | | | |
|------------------------------------|--|--|---|---|
| | Inverse water-oil emulsion with (IWOE) | Inverse water-oil emulsion with colmatant (IWOE-C) | Aqueous-based hydrophobic composition (AHC) | Inverse acid–oil emulsion (IAOE) |
| Characteristics of the composition | Inverse water–oil emulsion/blocking hydrophobic composition | Inverse water-oil emulsion with microcalcite-blocking hydrophobic composition with colmatant | Aqueous hydrophobic composition (1% aqueous dispersion of oil-wetting agent) | Inverse acid–oil emulsion–hydrophobic stimulation composition |
| Effective application area | Low permeability reservoirs with low and medium water-cut content (up to 60%) | High permeability or fractured reservoirs with low and medium water-cut content (up to 60%) | Medium and high permeability reservoirs with high water-cut content (over 60%) | Medium and high permeability reservoirs |
| Reservoir type | Terrigenous | Carbonate | Terrigenous | Carbonate |
| Application technology | Overlapping perforation interval without squeezing in the bottomhole formation zone (BFZ). The volume depends on the diameter of the production casing and the length of the perforated interval | Overlapping of perforation interval with BFZ injection at the rate by an average of 2.0–3.0 m ³ per 1 m of effective thickness of pay zone | BFZ compression at the rate by an average of 1.5–2.0 m ³ per 1 m effective thickness of the pay zone | BFZ compression at the rate by an average of 1.0–1.5 m ³ per 1 m effective thickness of the pay zone |

The efficiency of blocking BFZ technology before workover operation by IWOE composition developed jointly with OTO LLC is confirmed by the results of field tests conducted on 290 wells of West Siberian fields (Figure 11). Field test results showed the high efficiency of this technology in the form of increased oil production rate by an average of 5–10 m³/day, reduced the time required for the well to start operating up to 1–3 days, and reduce the water cut of formation fluid by 20–30% [1].



Figure 11. IWOE preparation process in the field.

The required volume of developed process fluids (PF), pushed into the bottomhole zone, depends on many factors and can be determined by the following formula:

$$V_{PF} = \pi \times (R^2 - r_w^2) \times h \times m$$
(4)

where R is planned radius of treatment, m; r_w is well radius, m; h is reservoir thickness, m; m is porosity, unit fraction.

5. Conclusions

The results obtained in the course of the work allowed drawing the following conclusions:

- Increase of well killing efficiency in complicated conditions can be achieved by directional regulation of BFZ filtration characteristics before workover operation in various geological, physical and technological conditions of field development through the use of new hydrophobic compositions of well killing fluids and well stimulation.
- 2. New compositions of well killing and stimulation fluids have been developed, which provide for prevention of fluid loss, gas breakthrough, as well as preservation, restoration and improvement of BFZ filtration characteristics in the conditions of terrigenous and carbonate reservoirs at different stages of oil field development.
- 3. As a result of experimental studies, it was found that the developed process fluids containing surfactants (YALAN-E2 and NG-1) have a hydrophobic effect on the porous medium of the reservoir rocks, which ultimately helps to preserve, restore and improve the filtration characteristics of BFZs.
- 4. The possibility to combine well killing operations before workover with the influence of developed compositions of process fluids on the BFZ allows to regulate BFZ filtration characteristics with maximum efficiency and sufficient coverage of the productive reservoir. Considering rather high frequency of well workover operations, the prospects of using such technology are obvious.
- 5. The results of field tests of BFZ technology blocking with an IWOE composition during well killing operation before workover showed improvement of the well operation efficiency in the form of increasing well flow rates by 5–10 m³/day on average, reducing the time required for the well to start operating up to 1–3 days and reducing the water-cut of the produced products by 20–30%.

Author Contributions: Experimental studies of physical, chemical and filtration properties of well killing fluids, analysis of the obtained results, D.V.M.; technological efficiency analysis of developed well killing fluids, M.K.R.; technological efficiency analysis of developed reagent emulsifier and oil-wetting agent, Y.V.Z.; analysis of normative documents and technologies of application of the developed compositions of well killing fluids, V.V.M. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The study did not report any data.

Conflicts of Interest: The authors declare no conflict of interest.

References

- Mardashov, D.V.; Rogachev, M.K. Development of blocking hydrophobic emulsion composition at well killing before well servicing. *Life Sci. J.* 2014, 11, 283–285.
- Li, J.; Li, Q.; Li, N.; Teng, X.; Ren, L.; Liu, H.; Guo, B.; Li, S.; Hisham, N.-E.-D.; Al-Mujalhem, M. Ultra-high density oil-based drilling fluids laboratory evaluation and applications in ultra-HPhT reservoir. *Soc. Pet. Eng. SPE* 2019, 1–13. [CrossRef]
- Leusheva, E.L.; Morenov, V.A.; Tabatabaee Moradi, S. Effect of Carbonate Additives on Dynamic Filtration Index of Drilling Mud. Int. J. Eng. 2020, 33, 934–939. [CrossRef]
- 4. Savari, S.; Whitfill, D.; Walker, J. Acid-soluble lost circulation material for use in large, naturally fractured formations and reservoirs. *Soc. Pet. Eng.* **2016**, 1–8. [CrossRef]
- 5. Dorman, J.; Udvary, F. Comparative evaluation of temporary blocking fluid systems for controlling fluid loss through perforations. *Soc. Pet. Eng.* **1996**, 1–11. [CrossRef]
- Tokunov, V.I.; Saushin, A.Z. Process Fluids and Mixtures for Oil and Gas Well Production Increase; Nedra: Moscow, Russia, 2004; pp. 7–10.
- Savari, S.; Whitfill, D. Managing losses in naturally fractured formations: Sometimes nano is too small. Soc. Pet. Eng. 2015, 1–10. [CrossRef]
- 8. Warren, F.P.; Robert, C.; Brookey, T.; Robinson, R.; Paradis, J. Adopting Aphron Fluid technology for completion and workover applications. *Soc. Pet. Eng.* 2008, 1–10. [CrossRef]
- 9. Binks, B.P. Modern Aspects of Emulsion Science Technology; Royal Society of Chemistry: Cambridge, UK, 1998; pp. 50–120.
- Orlov, G.A.; Kendis, M.S.; Glushchenko, V.N. Application of Invert Emulsions in Oil Production; Nedra: Moscow, Russia, 1991; pp. 3–4.
- 11. Allen, T.; Scott, K.; Baker, S.; Redburn, M.; Bechaver, M.; Dearing, H.; Ward, E.; Chamberlain, D. A novel invert emulsion system using a polyglycerol internal phase. *Offshore Mediterr. Conf.* **2015**, *OMC-2015-382*, 1–8.
- 12. Lu, S.; Foxenberg, W.E. Double-emulsion spacer design for highly efficient invert emulsion displacement. *Soc. Pet. Eng. SPE* 2012, 1–18. [CrossRef]
- 13. Ryabokon, S.A. Process Fluids for Well Completion and Workover, 2nd ed.; Prosveshcheniye-Yug: Krasnodar, Russia, 2009; pp. 6–13.
- 14. Petrov, N.A.; Soloviev, A.Y.; Sultanov, V.G.; Krotov, S.A.; Davydova, I.N. *Emulsion Solutions in Oil and Gas Processes*; Chemistry: Moscow, Russia, 2008; pp. 15–20.
- Sergeev, V.; Tanimoto, K.; Abe, M. Innovative emulsion-suspension systems based on nanoparticles for drilling and well workover operation. Soc. Pet. Eng. 2019, 1–12. [CrossRef]
- 16. Rogov, E.A. Study of the near-bottomhole zone permeability during treatment by process fluids. *J. Min. Inst.* **2020**, 242, 169–173. [CrossRef]
- 17. Palyanitsina, A.N.; Sukhikh, A.S. Peculiarities of assessing the reservoir properties of clayish reservoirs depending on the water of reservoir pressure maintenance system properties. *J. Appl. Eng. Sci.* **2020**, *18*, 10–14. [CrossRef]
- 18. Li, Q.; Zhu, J.; Li, S.; Zhang, S.; Hisham, N.-E.-D.; Ren, L.; Li, J.; Al-Mujalhem, M. Laboratory evaluation of weighting materials for ultra-high density oil-based drilling fluids in ultra-HPHT wellbore applications. *Soc. Pet. Eng.* **2019**, 1–10. [CrossRef]
- 19. Caenn, R.; Darley, H.C.H.; Gray, G.R. *Composition and Properties of Drilling and Completion Fluids*; Gulf Professional Publishing: Houston, TX, USA, 2011; pp. 187–215.
- 20. Volkov, V.; Turapin, A.; Ermilov, A.; Vasyutkin, S.; Fomin, D.; Sorokina, A. Experience of gas wells development in complex carbonate reservoirs in different stages of development. *Soc. Pet. Eng.* **2019**, SPE-196915-MS. [CrossRef]
- 21. Ezzat, A.M.; Blattel, S.R. Solids-free brine-in-oil emulsions for well completion. Soc. Pet. Eng. 1989, 1–7. [CrossRef]
- 22. Morenov, V.A.; Leusheva, E.L. Influence of the solid phase fractional composition on the filtration characteristics of the drilling mud. *Int. J. Eng.* **2019**, *32*, 794–798. [CrossRef]
- 23. Ramasamy, J.; Amanullah, M.; Alsaihati, M. Emulsifier developed from waste vegetable oil for application in invert-emulsion oil based mud. *Soc. Pet. Eng.* 2018, 1–6. [CrossRef]
- 24. Tagirov, K.M. Foaming well-killing composition based on organic foam and CaCl2 for oil and gas wells. *Fuel Energy Abstr.* 2002, 43, 241. [CrossRef]
- 25. Silin, E.M.; Magadova, L.G.; Cherygova, M.A.; Malkin, D.N.; Gayevoy, E.G.; Magadov, V.R.; Eliseev, D.Y. Application of killing fluids on the polysaccharide base in wells with low pressure and after fracturing. *Oil Ind. J.* **2010**, *4*, 104–107.
- 26. Demakhin, S.G.; Merkulov, G.; Kasyanov, D.N.; Maalyko, S.V.; Anfinogentov, D.A.; Chumakov, E.M. Well killing with block-packs is an effective means of saving the properties of a productive formation. *Oil. Gas. Innov.* **2015**, *1*, 66–69.
- 27. Prischepa, O.M.; Nefedov, Y.V.; Kochneva, O.E. Raw material base of hard-to-extract oil reserves of Russia. *Period. Tche Quim.* **2020**, *17*, 915–924.
- 28. Sabukevich, V.S.; Podoprigora, D.G.; Shagiakhmetov, A.M. Rationale for selection of an oil field optimal development system in the eastern part of the Pechora sea and its calculation. *Period. Tche Quim.* **2020**, *17*, 634–655.

- 29. Khaibullina, K.S.; Korobov, G.Y.; Lekomtsev, A.V. Development of an asphalt-resin-paraffin deposits inhibitor and substantiation of the technological parameters of its injection into the bottom-hole formation zone. *Period. Tche Quim.* **2020**, *17*, 769–781.
- Khaibullina, K.S.; Sagirova, L.R.; Sandyga, M.S. Substantiation and selection of an inhibitor for preventing the formation of asphalt-resin-paraffin deposits. *Period. Tche Quim.* 2020, 17, 541–551.
- 31. Musabirov, M.K.; Kuryashov, D.A.; Garifov, K.M.; Dmitriyeva, A.Y.; Abusalimov, E.M. Developing structure-forming colloidal systems for matrix acidizing of porous-fractured carbonate reservoirs. *Oil Ind. J.* **2019**, *6*, 71–73.
- 32. Foxenberg, W.E.; Ali, S.A.; Ke, M. Effects of completion fluid loss on well productivity. Soc. Pet. Eng. 1996, 1–16. [CrossRef]
- 33. Shagiakhmetov, A.M.; Podoprigora, D.G.; Terleyev, A.V. The study of the dependence of the rheological properties of gelforming compositions on the crack opening when modeling their flow on a rotational viscometer. *Period. Tche Quim.* **2020**, *17*, 933–939.