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LABORATORY STUDIES OF POLYMER COMPOSITIONS FOR WELL-KILL UNDER INCREASED FRACTURING

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ЛАБОРАТОРНЫЕ ИССЛЕДОВАНИЯ ПОЛИМЕРНЫХ СОСТАВОВ ДЛЯ ГЛУШЕНИЯ СКВАЖИН В УСЛОВИЯХ ПОВЫШЕННОЙ ТРЕЩИНОВАТОСТИ

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Well-service under increased fracturing is often complicated as early as at the stage of well-killing operations. The presence of cracks in the reservoir formation can result in significant absorption of drilling fluid, as well as in bottomhole gas breakthrough. The laboratory studies are required to develop a blocking agent for reliable isolation of highly permeable reservoir intervals without a negative impact on the reservoir porosity and permeability and in compliance with operational safety requirements.

For the purpose of this work, the physical, chemical and rheological properties of frame-forming and gel-forming compositions, which are cross-linked systems prepared on the basis of soluble silicates, have been studied. The well-killing technology implies their consecutive injection into the well. The reviewed compounds have proven their applicability in a wide range of reservoir temperatures, which provided for the possibility of their injection into a standard well and flushing into the bottomhole formation zone due to low viscosity and slowed cross-linking. Breaker application allows us to almost completely eliminate negative impact of this technology on filtration characteristics of the bottomhole formation zone after well-killing.

The studies conducted within the scope of this work have shown application efficiency of blocking polymer compounds and opened new directions for further researches, including the necessity to undertake a sequence of laboratory and field tests of the reviewed agents, which will enable to select and validate optimal technological and economic parameters of the operation.

The results obtained can be used to improve the efficiency of oil and gas well-kills under increased fracturing during well-service operations.

Ключевые слова:

глушение скважин, подземный ремонт скважин, карбонатный коллектор, трещиноватый коллектор, осложненные условия, аномально низкое пластовое давление, высокий газовый фактор, прорыв газа, блокирующий состав, шитый полимерный состав, лабораторные исследования, высокая термостабильность, деструкция, реология, эффективная вязкость.

Подземный ремонт скважин в условиях повышенной трещиноватости зачастую сопровождается осложнениями еще на этапе проведения операций глушения скважин. Наличие трещин в породе-коллекторе может привести не только к значительным поглощениям технологической жидкости, но и к прорыву газа к забою скважины. Необходимо проведение лабораторных исследований с целью разработки блокирующего состава для надежной изоляции высокопроницаемых интервалов пластов, не оказывающего отрицательного воздействия на фильтрационно-емкостные свойства коллектора и отвечающего требованиям безопасности при проведении работ.

В рамках данной работы изучены физико-химические и реологические свойства каркасообразующего и гелеобразующего составов, представляющих собой шитые системы, приготовленные на основе растворимых силикатов. Технология проведения глушения предполагает их последовательную закачку в скважину. Рассматриваемые составы показали технологичность их применения в широком диапазоне пластовых температур, заключающуюся в возможности их закачки в типовую скважину и продавки в призабойную зону пласта за счет низкой вязкости и замедленной скорости сшивки. Применение деструктора позволяет почти полностью исключить негативное влияние данной технологии на фильтрационные характеристики призабойной зоны пласта после глушения.

Исследования, проведенные в данной работе, показали эффективность применения блокирующих полимерных составов, а также открыли новые направления для дальнейших исследований, а именно – необходимость проведения цикла лабораторных и промысловых испытаний рассмотренных составов, что позволит выбрать и обосновать оптимальные технологические и экономические параметры проводимой операции.

Полученные результаты могут быть применены для повышения эффективности глушения нефтяных и газовых скважин в условиях повышенной трещиноватости при проведении ремонтных работ.

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Introduction

Based on the estimate of reserves, carbonate reservoirs currently account for about 60 % of hydrocarbon fields and approximately the same amount of oil production in the world. Development of such reservoirs is complicated by the presence of an extensive network of natural and artificial (as a result of hydraulic fracturing) fractures, high heterogeneity, complex structure of pore space, hydrophobic properties of reservoir formations, and abnormal low reservoir pressure.

The reviewed characteristic features of carbonate reservoirs can lead to low reservoir coverage, low displacement efficiency, rapid watercuts, gas breakthrough, and, as a result, a rapid drop in oil production [1–9].

The presence of formations with similar characteristics in the field determines a particular differentiated approach to the planning of well-killing operations related to the development and selection of a blocking agent. Correct selection of blocking agents for well-killing requires comprehensive laboratory tests for specific objects of planned application.

Non-compliance with the above conditions when planning well interventions can result in the loss of significant amounts of drilling fluids, extended workover and well start-up time, which will eventually increase the cost of underground operations [10–12].

The advanced solution in the development of blocking fluids is the use of cross-linked polymer compounds with time-based gelation. Viscosity fluctuation rate can be adjusted for more efficient process operations.

The technologies using the described polymer compositions have proved to be a reliable method of isolating high-permeability formation intervals in order to prevent water and gas breakthrough into production wells [13–15].

In the context of this work, physical, chemical and rheological properties of frame-forming and gel-forming compounds as blocking polymer fluids for well-killing have been selected and studied.

These compounds are cross-linked systems prepared on the basis of soluble silicates. In this case, silicate gel is formed when an aqueous

solution with relatively high pH value, containing a sufficient amount of orthosilic acid monomer or orthosilic acid oligomers, experiences its pH value reduction or is exposed to hard cations [16, 17].

The frame-forming compound is a non-transparent heterogeneous liquid of white colour. When injected into the reservoir, the liquid phase is hardened and the dry residue is coagulated under elevated temperatures, creating a strong frame-forming screen to isolate the intervals with increased fracturing. Such compound feature allows reducing losses of drilling fluid injected into the well during well-killing.

In its turn, the gel-forming compound is a transparent homogeneous liquid that, due to its increased viscosity, has a low formation filtration rate and is able to prevent gas breakthrough under high gas-oil ratio conditions. In our case, it was prepared in two modifications with a gel-forming agent for low (20 °C) and high (90 °C) temperatures.

The technology of well-killing during their underground workover involves consecutive injection of the frame-forming and gel-forming compounds into the well.

The laboratory physical, chemical and rheological studies of compounds consisted of several consecutive stages:

- 1) density determination;
- 2) evaluation of heat resistance;
- 3) evaluation of breakability;
- 4) determination of effective viscosity and static shear stress (SSS); and
- 5) determination of gelation time of cross-linked compositions.

Physical and Chemical Studies of Compounds

The density of the frame-forming and gel-forming compounds was determined by pycnometer method as per [18] and amounted to 1.045 and 1.015 kg/m³ for low temperatures, and 1.022 kg/m³ for high temperatures.

Such density value of the studied compounds allows applying them at normal and reduced formation pressure [19–22].

To measure the heat resistance, the prepared compound was kept in thermostat at the

temperatures from 20 °C or 90 °C for three days (average duration of servicing operations). Next, the condition of the composition under study and the change in appearance were visually estimated. In case of water phase separation, breaking and sedimentation, the compound was considered unstable [23–28].

Following the heat resistance estimation of the frame-forming compound (Table 1), it was established that 15 minutes after the composition was prepared, both at 20 °C and at 90 °C, up to 20 % and 25 % of water was released, respectively. Throughout further thermostatic control, the amount of water released remained practically unchanged. At the same time, manufacturer of the frame-forming compound allows for water release from the composition volume in small amounts. Therefore, it was decided that the frame-forming compound is heat-stable at 20 °C and 90 °C and can retain its physical and chemical properties during servicing operations in the well.

Table 1

Heat-Resistance Properties of the Frame-Forming and Gel-Forming Compounds

Compound	Heat Resistance at 20 °C over 3 days	Heat Resistance at 90 °C over 3 days	Note
Frame-Forming	Yes	Yes	Release up to 20 % and 25 % of water, respectively
Gel-Forming	Yes	Yes	–

At the same time, during the thermostatic control of the gel-forming compound, no visible changes in appearance were observed over the entire period under study.

It should be noted that the requirement to such compounds at well-killing is their full or partial breaking on exposure to breaker. The compounds failing to meet the given conditions cannot be recommended for application, since it deteriorates the reservoir's permeability properties and complicates the well development and start-up after servicing.

Breakability of the frame-forming and gel-forming compounds was estimated visually at 20

°C and 90 °C over 24 hours by mixing the breaker with the reviewed blocking agents at a ratio of 1:1. 10 % aqueous sodium hydroxide solution was used as a breaker for the gel-forming compound, whereas a mixture of 12 % hydrochloric acid and 5 % hydrofluoric acid was used to break the frame-forming compound.

Following the results of breaking estimation of the frame-forming and gel-forming compounds (Table 2), it was established that these compounds can be recommended for use at well-killing.

Determination of Rheological Properties of the Compounds

The study of rheological properties of the frame-forming and gel-forming compounds consisted in measuring the following parameters:

- effective viscosity at 300 rpm;
- ‘viscosity curves’ (dependence of effective viscosity on shear rate or rotor speed);
- static shear stress after gel-formation of cross-linked compositions at controlled shear rate by plotting the dependency of shear stress τ on shear rate D , determination of shear stress value at which breaking and motion of the compound structure starts;
- gelation time of cross-linked compositions under dynamic conditions by measuring effective viscosity μ_{eff} of the compound over time at a given shear rate equal to the rate of the compound injection into the well, and under static conditions of oscillating stresses by measuring complex viscosity over time.

Plotting a viscosity curve and determining static shear stress were conducted for the frame-forming and gel-forming compounds. Static shear stress determines the initial pressure gradient, at which breaking and motion of the composition structure take place [18, 29–35].

Experiments to determine the gelation time were carried out only for cross-linked compositions that are nonlinear gels.

Rheological properties were determined as per [36] with a rotary automated viscometer Rheotest RN 4.1 (Fig. 1). Measurements were performed using a cylinder measuring system.

Table 2

Evaluation of Breaking Processes of the Frame-Forming and Gel-Forming Compounds

Blocking Compound	Temperature , °C	Breaking Result	Breaking Time, hrs	Breaker	Recommendation for use
Frame-Forming	20	Partly	1 (50 %) 24 (75 %)	12% HCl + 5% HF	Recommended
	90	Partly	1 (75 %)	12% HCl + 5% HF	Recommended
Gel-Forming	20	Fully	3	10% NaOH aqueous solution	Recommended
	90	Fully	1	10% NaOH aqueous solution	Recommended



Fig. 1. Rotary automated viscometer Rheotest RN 4.1

I. The test procedure for linear (non-cross-linked) compounds.

1. Process simulation of compound motion start (breaking of compound structure). Static shear stress is determined by plotting the dependency of shear stress on shear rate at controlled shear rate. Static shear stress is equal to the value of shear stress τ preceding the compound structure breaking, and it indicates the compound motion start. Experimental conditions:

- preliminary compound hold in the measuring cell for 15 min;
- experiment time $t = 0-500$ sec;
- shear stress $\tau = 0-2000$ Pa;
- temperature $20\text{ }^{\circ}\text{C}$ and $90\text{ }^{\circ}\text{C}$, respectively.

2. Effective viscosity was determined at controlled shear rate: viscosity curve (CR test – Controlled Rate). The essence of the investigation method was to establish the dependency of effective viscosity (shear stress) on shear rate (rotation speed) with a gradual increase of the latter parameter from 0 to 300 s^{-1} . Experimental conditions:

- time $t = 0-300$ sec;
- shear rate $\gamma = 0-300\text{ s}^{-1}$; and
- temperature $20\text{ }^{\circ}\text{C}$ and $90\text{ }^{\circ}\text{C}$, respectively.

II. Due to the fact that viscosity of the gel-forming compound changes over time, the decision was made to conduct a staged study of rheological properties according to the following scheme [37–42]:

1. Compound condition simulation immediately after preparation at wellhead to estimate filterability in annular space or in a tubing string. Compound effective viscosity at different shear rates is determined by establishing the viscosity curve. Experimental conditions:

- time $t = 0-300$ sec;
- shear rate $\gamma = 0-300\text{ s}^{-1}$; and
- temperature $20\text{ }^{\circ}\text{C}$.

2. Simulation of compound injection along the annular space into the well subject to uniform temperature change along the wellbore. Gelation time at constant shear rate is determined. Experimental conditions:

- experiment time $t = 85$ min;
- shear rate $\gamma = 21.8\text{ s}^{-1}$; and
- temperature $T = 20\text{ }^{\circ}\text{C} - 90\text{ }^{\circ}\text{C}$ (for the compound applicable at $20\text{ }^{\circ}\text{C}$, the experimental temperature $T = 20\text{ }^{\circ}\text{C}$).

3. Process simulation of compound injection into the production string space. Gelation time at constant shear rate is being determined. Experimental conditions:

- experiment time $t = 15$ min;
- shear rate $\gamma = 7.1\text{ s}^{-1}$;
- temperature $T = 90\text{ }^{\circ}\text{C}$ (for the compound applicable at $20\text{ }^{\circ}\text{C}$, the experimental temperature $T = 20\text{ }^{\circ}\text{C}$).

4. Process simulation of the compound holding for the reaction time (the end of cross-linking process) at formation temperature. Gelation time under static conditions of oscillating stresses is determined [43–45]. Experimental conditions:

- time $t = 3$ hours;
- rotor oscillation frequency $f = 1$ Hz;
- voltage module $G = 10$ Pa;
- temperature 90 °C (for the compound applicable at 20 °C, the experimental temperature $T = 20$ °C).

5. Process simulation of the compound motion start (compound structure breaking). Static shear stress is determined by plotting the dependency of shear stress on shear rate at the controlled shear rate. Experimental conditions:

- time $t = 0–500$ sec;
- shear stress $\tau = 0–2000$ Pa.
- temperature 90 °C (for the compound applicable at 20 °C, the experimental temperature $T = 20$ °C).

6. Compound condition simulation after injection into the well to estimate its effective viscosity change. Effective viscosity of the compound at different shear rates is determined by establishing the viscosity curve. Experimental conditions:

- time $t = 0–300$ sec;
- shear rate $\gamma = 0–300$ s⁻¹;
- temperature 90 °C (for the compound applicable at 20 °C, the experimental temperature $T = 20$ °C).

The summarized results of rheological studies of compounds are given in Table 3.

As a result of cross-linking time determination during the gel-forming compound injection simulation (applied for 20 °C) into a simulated well, the following has been established:

- the compound cross-linking process starts during its injection into the annular space. Gelation start induction period is approx. 40 minutes after the compound preparation;
- the compound cross-linking process ends after its injection into the well, during the well shut-in period. Gelation end induction period is approx. 2 hours 20 min after the compound preparation.

Table 3

Rheological Studies of the Frame-Forming and Gel-Forming Compounds

Compound	Temperature, °C	Effective viscosity, mPa·s, after preparation at $N = 300$ rpm	Effective viscosity, mPa·s, after cross-linking at $N = 300$ rpm	SSS, Pa
Frame-forming	90	27	–	2.66
	20	64	–	3.38
Gel-forming	90	10.7	17.4	126
	20	8	600	648

Note: ‘–’ means that the effective viscosity for the given compounds has not been measured because they are not ‘cross-linked’ and their viscosity is time-independent.

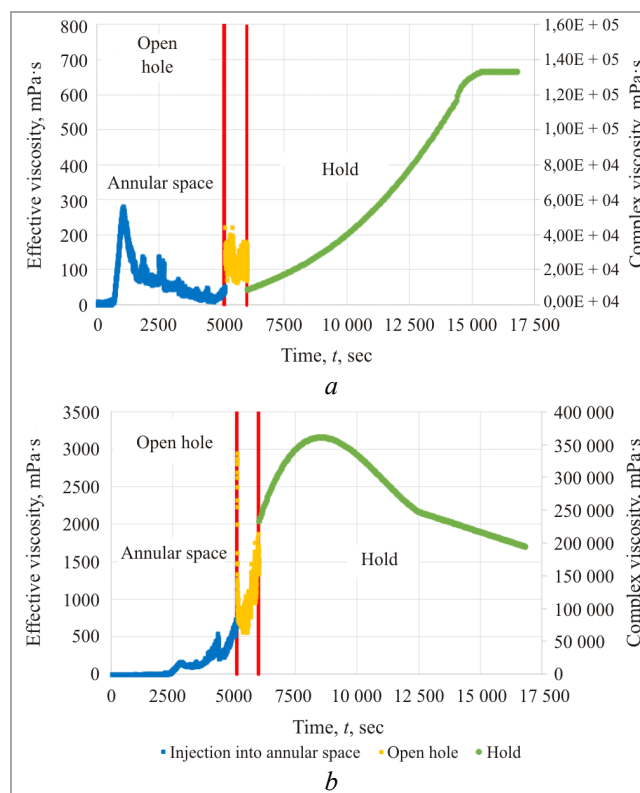


Fig. 2. Dynamics of effective and complex viscosity of the gel-forming compound: *a* – 90 °C; *b* – 20 °C

The results of gelation time determination under conditions of cross-linked compounds injection are shown in Fig. 2.

Based on the results published in [34], the higher the static shear stress, the higher is the pressure gradient the blocking agent is able to withstand without breaking. The same conclusions are true when the effective viscosity increases, since its structural strength grows.

Based on cross-linking time determination during the gel-forming compound injection simulation (applied for 90 °C) into a simulated well, we came to the following conclusions:

- the cross-linking process starts during the compound injection into the annular space. Gelation start induction period is approx. 12 minutes after the compound preparation;

- the compound cross-linking process ends during its injection into the annular space. Gelation end induction period is approx. 18 minutes after the compound preparation. After the compound injection into the well, during the well shut-in period, structuring is observed, characterized by an increase in complex viscosity, which ends 4 hours after the compound preparation. In order to increase the process efficiency (for example, to prevent mechanical breaking of the gel during its injection into the well) of well-killing using the gel-forming compound applied for 90 °C, it is recommended to adjust the component composition so that the gelation process is completed after the entire compound is injected into the well.

Conclusion

The following results were obtained based on the physical, chemical and rheological studies of frame-forming and gel-forming compounds:

1. The compounds applied at low (20 °C) and high (90 °C) temperatures are heat-resistant over the entire period of time (3 days).

2. The results of breaking estimation of the frame-forming and gel-forming compounds have shown the following:

- the gel-forming compound is broken completely by 10 % aqueous solution of NaOH over 1 and 3 hours at 90 °C and 20 °C, respectively;

- the frame-forming compound is broken by 75 % by a mixture of 12 % hydrochloric acid and 5 % hydrofluoric acid (mud acid) over 1 and 24 hours at 90 °C and 20 °C, respectively.

3. The gel-forming (8 and 10.7 mPa·s) and frame-forming (64 and 27 mPa·s) compounds feature low effective viscosity after preparation at 20 °C and 90 °C, respectively, which allows for the conclusion of their applicability for the well injection.

4. The gel-forming compounds can have high blocking ability as their SSS is 648 Pa at low temperatures (20 °C) and 126 Pa at high temperatures (90 °C).

The physical, chemical and rheological studies of the frame-forming and gel-forming compounds have shown their applicability at reservoir temperatures from 20 °C to 90 °C, involving their injection into a standard well and flushing into the bottomhole formation zone, due to their low viscosity after preparation and slow cross-linking. However, based on the studies conducted, it is recommended to adjust the contents of gel-forming compound composition (applied for 90 °C) so that the gelation process is finished after the complete injection of the compound into the well.

Besides, the breakers application allows us to reduce the negative impact of this technology on filtration characteristics of the bottomhole formation zone after well-killing due to the reduced coefficient of the relative permeability curve and pressure gradient of the oil filtration start during well ‘development’.

To address the problem of well-killing in complex geological and physical conditions of carbonate reservoir development it is necessary to conduct a sequence of laboratory and field tests of the reviewed compounds, which will enable one to select and validate optimal technological and economic parameters of the operation.

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