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**Results of Studying the Influence of Destroying Liquids on Polymineral Filter Cake****Artyom A. Petrov, Nikolay I. Nikolaev**

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**Результаты исследования влияния жидкостей-разрушителей на полимерминеральную фильтрационную корку****А.А. Петров, Н.И. Николаев**

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In the course of well construction, deterioration of the natural reservoir properties of the formation is inevitable. A dense low-permeability filter cake formed on the surface of the bottomhole formation zone at the stage of completion contributes to a decrease in the reservoir properties of the rock, and hence the productivity of the well. In addition, the cake can contribute to the plugging of structural elements such as the well screen or inflow control devices, thereby also exerting a negative impact on the flow rate. In most cases, the impossibility of achieving the required cleaning of the bottomhole zone during well completion in the future leads to the need to use expensive repeated operations and overhaul, chemical and mechanical methods of cleaning the bottomhole formation zone. The development of technologies for the complete removal of the formed filter cake from the borehole walls is a necessary task.

The paper considers the water-based biopolymer solution of the primary opening of the productive formation and presents its component composition and parameters. A method is described for studying the effect of breaker systems on a polymer-mineral filter cake under high-pressure conditions using filter press HT-HP, and a method for determining the dissolving capacity of individual components of the system (chelate and enzyme). Based on the results of this study, the optimal composition of the breaker of the enzyme-chelate base was selected, after the action of which the most complete destruction of the crust constituents was observed. The results were evaluated by comparing the dissolving power of calcium carbonate when exposed to different chelates. The enzyme alpha-amylase was used as a starch breaker. The efficiency of the composition was confirmed by the change in the filtration-capacitive properties of ceramic discs before and after processing in comparison with foreign analogues of breaker systems.

**Ключевые слова:**

обработка призабойной зоны пласта, фильтрационная корка, брейкер, брейкерная системы, хелаты, комплексобразователи, энзимы, проницаемость, фильтрационно-емкостные свойства, первичное вскрытие пласта, биополимерные растворы, освоение скважин, скважинные фильтры.

В процессе строительства скважины ухудшение естественных коллекторских свойств пласта неизбежно. Образующаяся на поверхности призабойной зоны пласта плотная малопроницаемая фильтрационная корка (ФК) на стадии освоения способствует снижению фильтрационно-емкостных свойств породы, а значит и продуктивности скважины. Помимо этого корка может способствовать закупориванию таких элементов конструкции, как скважинный фильтр или устройства контроля притока, тем самым также оказывая негативное влияние на дебит. В большинстве случаев невозможность достижения требуемой очистки призабойной зоны при освоении скважины в дальнейшем ведет к необходимости применения дорогостоящих повторных операций и капитальному ремонту, химических и механических способов очистки ПЗП. Разработка технологий полного удаления сформировавшейся фильтрационной корки со стенок ствола скважины является необходимой задачей.

В работе рассматривается биополимерный раствор первичного вскрытия продуктивного пласта на водной основе, приведен компонентный состав и его параметры. Описана методика исследования действия брейкерных систем на полимер-минеральную фильтрационную корку в условиях высокого давления на фильтр-прессе HT-HP, а также приведена методика определения растворяющей способности отдельных компонентов системы (хелата и энзима). По результатам данного исследования авторами был подобран оптимальный состав брейкера энзим-хелатной основы, после действия которого наблюдается наиболее полное разрушение составляющих фильтрационной корки. Результаты оценивались на основании сравнения растворяющей способности карбоната кальция при воздействии различных хелатов. В качестве разрушителя крахмала был протестирован энзим альфа-амилаза. Эффективность работы состава подтверждена на основании изменения фильтрационно-емкостных свойств керамических дисков до и после обработки в сравнении с зарубежными аналогами брейкерных систем.

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Introduction

Table 1

Component composition of the primary opening solution

Reagent	Concentration, %	Reagent purpose
KCl	4	Density regulator
Biopolymer	0.4	Gelling and structure-forming agent
Starch	1.5	Water loss regulator
CaCO <sub>3</sub>	8	Bridging agent
"Sludge"	2*	Drilling cuttings simulation

Note: \* 2 % of solid phase (as simulated cuttings) is the maximum permissible solid phase content for primary opening solutions, which has no impact on the increase in repression and strength properties of the filter cake.

In the process of opening a productive formation, the deterioration of natural permeability of the reservoir is inevitable due to the drilling mud impact on the bottomhole zone [1-3]. However, formation of a dense filter cake on the reservoir surface minimizes the impact of the drilling mud on the reservoir [4, 5]. But at the stage of development, especially in long horizontal wellbores [6–8], the filter cake acts as a barrier resulting in a decrease in the rock permeability [9, 10], well filters plugging [11, 12] and, as a consequence, in decreased well productivity [13–15]. In this regard, a crucial task is to develop such process fluids composition and such technology of their application, which would ensure removal of the filter cake when used in an open wellbore.

Recently, both foreign [19–21] and Russian [22–24] service companies have actively developed a trend of filter cake washing out or destruction by special compositions in order to maximize the hydrodynamic well-reservoir connection [16–18]. This is due to the need to carry out work in the bottomhole zone of horizontal well sections with installed filters, inflow control devices, etc., which are prone to plugging. It is also necessary to consider the filter materials that are chemically attacked by the compositions. That is, process fluids should not have a significant impact on the filter and at the same time should be effective in destroying all the components that form the filter cake, while not harming the productive formation [25, 26].

It is worth noting that the use of process fluids to break the filter cake helps to facilitate inflow stimulation, which is especially important for wells with drawdown constraints [11, 27, 28].

The problem of productive formation damage control by breaking the filter cake is relevant for wells of the Vostochno-Messoyakhskoye field [29].

Research procedure

Laboratory research was carried out in the laboratory of the Mining University Well Drilling Department in two stages. As a result, the most effective composition was selected for destruction of the filter cake formed when a primary opening solution was exposed to the bottomhole zone.

At the first stage, the performance of three foreign destroying liquids was studied and evaluated. The purpose of this stage was to study the impact of destroying liquids different in their nature on the corresponding critical components of the filter cake [30] and selection of the most optimal and effective components of the developed destroying liquid.

The tasks of the first stage included:

1. Preparation of the solution for the productive formation opening. The optimal drilling fluid for productive formation opening should contain only the components that are easily dissolved and dispersed during well development [25, 31, 32]. Clay-free biopolymer systems most fully meet the well flushing requirements, including ones with horizontal wellbores, and are actively used in drilling practice due to minimal negative impact on the bottomhole formation zone [33–35].

A standard formulation of the primary opening solution used in the Vostochno-Messoyakhskoye field was selected for testing. The composition and assignment of each reagent are shown in Table 1.

The solution was prepared by introducing reagents in a certain sequence: KCl, starch, biopolymer, carbonate, and simulated cuttings, with thorough mixing. Crushed

core of the productive formation of the Vostochno-Messoyakhskoye field was used as sludge. Then the solution was left for at least 16 hours, during which structural opening of the biopolymer took place. The characteristics of the biopolymer solution are shown in Table 2.

2. Filter cake formation by means of an HT–HP filter press (Fann) on paper discs (Fig. 1). After 16 hours, the solution was stirred for 5 minutes and placed in the cell of the HT–HP filter press for 30 minutes (at room temperature and pressure of 2 MPa) to form a filter cake.

3. Holding the formed filter cakes in the breaker system. The formed filter cake was placed in a container with the preprepared breaker (according to the procedure of the manufacturer) and left for 24 hours. At least three filter cake destruction tests were carried out for each composition of the destroying liquid.

4. Evaluation of the obtained result. After 24 hours, the cake was removed, and then a quick test was carried out for the presence of starch and calcium carbonate by interaction with 5 % iodine solution and 10 % hydrochloric acid solution, respectively. A change in the color of the iodine solution to violet (blue) indicated the presence of starch in the filter cake. A violent reaction with hydrochloric acid indicated the presence of calcium carbonate.

The purpose of the second stage of laboratory tests is to study the permeability of ceramic discs that simulate the formation, after treatment with a destroying liquid composition to record changes in their filtration and capacity properties.

After selecting the component composition of the breaker, the efficiency of the studied compositions under high pressure conditions was assessed. The research was carried out according to the following procedure:

1) rheological parameters and density of the drilling mud were measured;

2) a filter cake was formed on an HT–HP device (ceramic disc 10 μm, temperature 22 °C, time 30 min);

3) then the filtration cell was disassembled and the filter cake was photographed;

4) the filtration cell was reassembled and a ceramic disc with the previously formed filter cake was placed in it;

5) the destroying liquid composition was poured into the cell, and then heat aging occurred at 23 °C and pressure of 110 psi for 24 hours;

6) the HT–HP cell was disassembled after 24 hours and the disc with the filter cake residues (if any) was photographed;

7) the presence of starch and calcium carbonate on the disc was determined;

8) the cell was reassembled (turning the disc with the filter cake down) and the flow time of 300 ml of engine oil was measured at a pressure of 300 psi (or the filtrate volume was measured during 30 minutes of filtration);

9) the filter cell was disassembled, the filter disc was removed, and the presence of starch and calcium carbonate on the disc was determined by means of quick tests.

Table 2

Biopolymer solution parameters

Parameter	Density, kg/m <sup>3</sup>	F 30 min, ml	Gel 10 sec	Gel 10 min	R600	R300	R6	R3	<sup>o</sup> solut, MPa*s	<sup>o</sup> spec, lb/100 ft <sup>2</sup>
Value	1080	4	11	14	43	32	9	7	11	21

**Laboratory Research (Main Part)**

At the first stage of laboratory research, three foreign filter cake breakers were tested. After keeping for 24 hours in the destroying liquid, quick tests were carried out for the presence of main contaminating components in the filter cake, i.e. starch and calcium carbonate. The most effective breaker for the filter cake destruction was the composition based on chelate and enzyme compounds, which is also confirmed by the analysis of available literature [36, 37].

When using this breaker, complete destruction of the cake is achieved over a specified period (Fig. 2, a). Other two foreign-made breakers based on organic and inorganic acids fulfill their function partially: the filter cake is not completely destroyed, and there are "residues" of its components (Fig. 2, b).

The next step of the research was chelate and enzyme selection for the developed liquid.

**Selection of Chelate Compound**

The chelating agent promotes the formation of metal ion complexes present in filter cakes formed by drill-in fluids and in completion fluids [38], especially calcium, iron, and magnesium ions. It reacts exclusively with calcium carbonate and does not cause corrosion, as opposed to acids or oxidizing agents [39].

In order to select the most effective chelating compound, a study on determination of calcium carbonate solving power was carried out. This procedure was proposed by the specialists of Burintekh, Ltd. As part of the study, three complexing agents (chelates) were tested: amine-containing (chelate No. 1), phosphorus-containing (chelate No. 2), and acetic acid salts (chelate No. 3).

The studies were carried out according to the following procedure: a 10 g sample of chalk was placed in a vessel with a chelate compound and kept at different pH values of the medium to study the effect of this parameter on the reaction behavior. Then the vessel was placed in an oven, where the liquid was evaporated. Then, the residual mass of calcium carbonate was estimated by weighing after interaction with the chelate (*m*<sub>2</sub>), and the mass fraction of substances soluble in the complexing agent (*K*) was calculated by the formula (1):

$$K = \left( \frac{m_1 - m_2}{m_1} \right) \cdot 100 \%, \tag{1}$$

where *m*<sub>1</sub> – initial weight of the chalk sample, g; *m*<sub>2</sub> – weight of chalk after reaction with chelate and drying, g.

According to the results of the completed tests, a high dissolving ability of the used complexing agents is noted at various pH values. The highest rate is observed in the range of pH = 4–7, since the acidic medium promotes intensive dissolution of carbonates. The amount of dissolved chalk increases by 9–17 % in the acidic medium. This can be explained by the fact that in an alkaline medium, a large amount of OH<sup>-</sup> ions contributes to occurrence of side (undesirable) reactions, interacting with the original substances and reducing the selective character of the reaction [40]. In terms of the amount

of dissolved chalk, chelates No. 1 and No. 3 surpass the foreign reagent.

Based on the completed studies, the most effective chelate in terms of calcium carbonate dissolving power is a chelate being the acetate (Fig. 3). The mass fraction of dissolved substances (calcium carbonate) upon interaction with this chelate was 92 % at pH = 4–7 (84 % at pH = 7–10).

**Selection of Enzyme (Starch Destroying Liquid)**

An enzyme is a complex three-dimensional protein molecule produced by living cells. When interacting with starch molecules, the enzyme breaks them down into ordinary carbohydrates and sugars (it is a catalyst of the starch hydrolysis), while disrupting the integrity of the filter cake and increasing the efficiency of well development [41].

Poor availability, as well as rather high price of enzymes on the reagent market, became the determining factors when making a selection of a starch destroying enzyme. Analysis of literature sources [42–44] showed that the starch destruction is most efficiently carried out by a special enzyme – alpha-amylase. Alpha-amylase is a complex organic compound of a protein nature, a biological catalyst. Interaction of starch with alpha-amylase is accompanied by hydrolysis, which results in the formation of monosaccharide chains [41].

In laboratory conditions, the effect of alpha-amylase on the starch contained in the filter cake was tested. The formed filter cake was placed in a container with the enzyme and kept at various temperatures and pH values. After removing the filter cake, a starch test was carried out: the assessment was made based on the color change of the iodine solution when interacting with filter cake components. The presence of starch gives a blue coloration of iodine, and the absence of it – brown.

In the course of laboratory tests of enzymes, it was revealed that the destruction of starch occurs most effectively at a reaction temperature of 50–75 °C – complete removal of starch from the disc surface is observed. At lower temperatures the enzyme is only locally destroyed, and at temperatures above 90 °C it begins to coagulate, which means it loses its ability to act on starch. In this case, the enzyme is not only ineffective, but also becomes a source of reservoir contamination [45]. Also, enzymes do not function at pH values above 10.

The main components of the filter cake breaker were selected based on the results of the first stage studies. They were salts of acetic acid as a solvent for calcium carbonate, and alpha-amylase as a starch breaker.

The second stage of the research involved pressure testing of three foreign breakers and a breaker developed in this work. All tests were carried out on the HT-HP filter press according to the foregoing procedure.

In addition to testing breakers at a pressure of 110 psi (for 24 hours), tests were carried out when the breaker was left for 24 hours at a cell pressure of 500 psi – the closest one to the bottomhole pressure in laboratory conditions as per API guidelines. The research results are shown in Table 3.

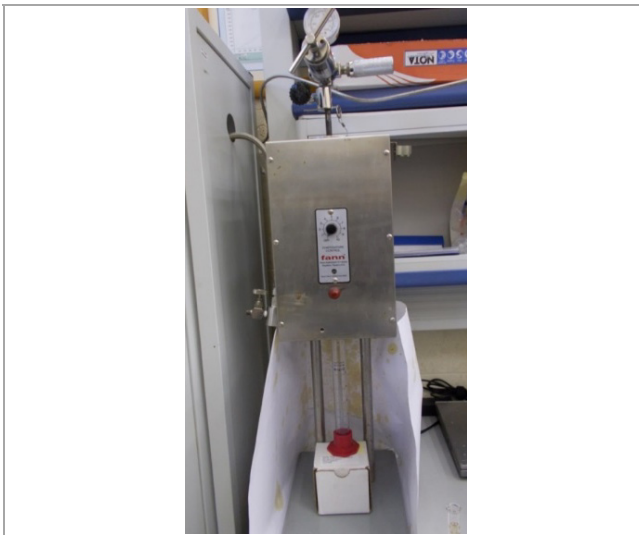


Fig. 1. HT-HP filter press

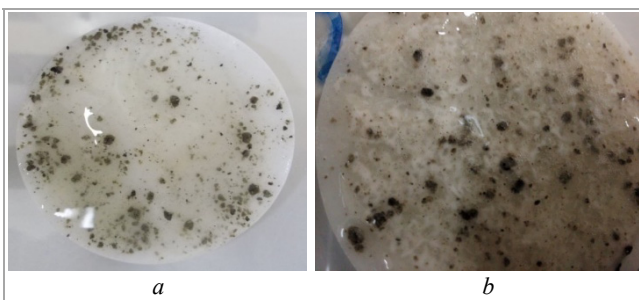


Fig. 2. Laboratory studies using a breaker: *a* – filter cake after using a breaker based on chelates and enzymes; *b* – paper filter with filter cake residues after organic-acid-based breaker application

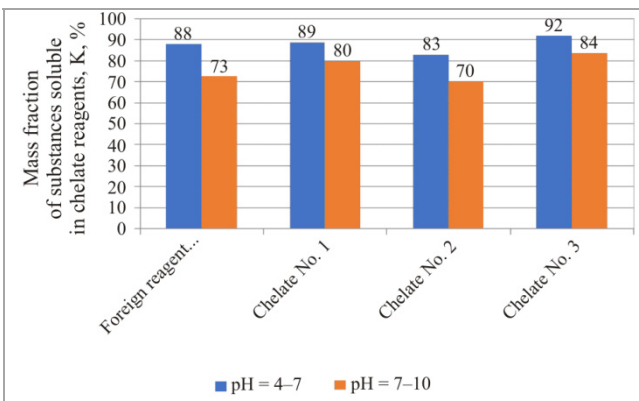


Fig. 3. Mass fraction of substances soluble in chelating reagents at room temperature and different pH levels



Fig. 4. Discoloring of the iodine solution, indicating the presence of starch in the filter cake

The results obtained for engine oil filtering through a ceramic disc (after holding the filter cake in a breaker liquid) are compared with the filtration of the same engine oil through a disc without filter cake and a disc with its filter cake not broken. Comparison of these values allows us to make a conclusion about the breaker fluid effectiveness in the disc filtration properties recovery.

During testing, it was noted that at 500 psi in the filter press cell the starch and calcium carbonate test was positive, and therefore the filter cake breaker system did not work. A pressure increase in the working medium prevents the release of carbon dioxide which is formed as a result of the interaction between chelate and calcium carbonate, and finally complicates the chemical reaction, since its rate decreases extremely [46, 47].

Evaluation of the breaker system effectiveness in filter cake destruction was carried out on the basis of comparing the values of engine oil consumption through ceramic filters:

- 1) a ceramic disc without cake formed: it is possible to evaluate the formation permeability not disturbed by primary opening solution by the flow rate through this filter disc;
- 2) a filter disc with the filter cake untreated with the breaker – it is possible to evaluate permeability deterioration due to the use of the primary opening solution;
- 3) a ceramic filter with the broken filter cake by means of a breaker system.

In the course of the tests, the time of 300 ml of engine oil flowing through a ceramic disc without filter cake (at a pressure of 300 psi in the HT-HP filter press cell) was measured – 3 min 20 s, while the flow rate was 1.5 ml/s. The flow rate through a ceramic disc with the formed filter cake was determined in the same way (without the filter cake breaker systems). Filtration volume of 300 ml of engine oil per 30 min was 0.4 ml – the flow rate was 0.000222 ml/s.

Comparison of the values of the engine oil flow rate through ceramic discs will make it possible to evaluate the reservoir filtration properties and quality of the formation-well communication channel (Table 4).

Based on the values of engine oil flow through ceramic discs, an increase in engine oil flow rate due to the breaker systems application indicates their effectiveness. The flow rate changes 8–14-fold when using foreign filter cake breaker systems and 4.5-fold when using the developed composition (see Table 4).

**Conclusion**

Based on the results of the laboratory studies analysis, the effectiveness of the existing polymer-mineral filter cake breaker systems is worth noting.

The components of the developed breaker system selected in the process of research, namely chelate and enzyme, have demonstrated their effectiveness. The best result in terms of the ability to dissolve calcium carbonate was shown by chelate constituting acetic acid salt. In turn, alpha-amylase was chosen as the starch breaker, which demonstrated a good destructuring effect on the polymer.

The developed composition of the enzyme-chelate base destroys the polymer-mineral filtration cake – the filtration properties of the ceramic disc are improved 4.5-fold. The composition needs to be improved to increase the performance indicators at least up to the level of foreign manufacturers or higher.

Table 3

Results of the second stage of the research

Breaker	After holding in the cell (22 °C, 500 psi)		Engine oil filtrate volume after 30 min, ml	After holding in the cell (22 °C, 110 psi)		Engine oil filtrate volume after 30 min, ml	After holding in indoor conditions (22 °C)		Engine oil filtrate volume after 30 min through FC (untreated with breaker), ml
	starch test	CaCO <sub>3</sub> test		starch test	CaCO <sub>3</sub> test		starch test	CaCO <sub>3</sub> test	
No. 1	+	+	1.2	+	-	5.6	+	-	0.4
No. 2	+	+	0.75	+	-	3.2	+	-	
No. 3	+	+	0.8	+	-	5.4	+	-	
Developed composition	+	+	0.55	+	-	1.8	+	-	

Note: FC – filter cake.

Table 4

The flow rate of engine oil when using various media

Breaker	Engine oil flow rate (Q) through the ceramic disc, ml/s		
	after using the breaker system	with filter cake untreated by the breaker system	without filter cake on the filter
Foreign 1	0.0031	0.000222	1.5
Foreign 2	0.00178		
Foreign 3	0.003		
Developed composition	0.001		

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