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**Hydrophilization of the Reservoir Surface in the Processes of Impact on the Bottomhole Formation Zone****Viktor N. Glushchenko<sup>1</sup>, Grigoriy P. Khizhnyak<sup>2</sup>, Mikhail S. Turbakov<sup>2</sup>, Dmitriy V. Kobayakov<sup>2</sup>**<sup>1</sup>Independent author (36A Narodny Boul., Belgorod, 308001, Russian Federation)<sup>2</sup>Perm National Research Polytechnic University (29 Komsomolskiy av., Perm, 614990, Russian Federation)**Гидрофилизация коллекторской поверхности в процессах воздействия на призабойную зону пласта****В.Н. Глущенко<sup>1</sup>, Г.П. Хижняк<sup>2</sup>, М.С. Турбаков<sup>2</sup>, Д.В. Кобяков<sup>2</sup>**<sup>1</sup>Независимый автор (Российская Федерация, 308001, г. Белгород, Народный бульвар, 36А, кв. 11)<sup>2</sup>Пермский национальный исследовательский политехнический университет (Российская Федерация, 614990, г. Пермь, Комсомольский пр., 29)

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The evidence base of supporters of the need for hydrophobization of the reservoir surface in the bottomhole formation zone when exposed to aqueous process fluids is based on erroneous ideas about the improvement of oil filtration in this case compared to water. A critical analysis of literary sources on the topic of hydrophobization of the bottomhole formation zone indicates an incorrect premise of many domestic researchers in the interpretation of the main provisions of the mechanism of its action in the real reservoir space on the flow of formation fluids under the influence of hydraulic pressure. The efficiency achieved in field conditions from the presence of, in particular, cationic surfactants is explained not by its conversion to a hydrophobic state, but, at best, by partial hydrophilization and a number of other associated effects: hydrocarbon saturation of the bottomhole formation zone, complex action of acidic compositions, etc. A more acceptable and explainable is the need to maintain a hydrophilic state of the reservoir surface in the bottomhole formation zone, which is ensured by non-ionic surfactants and/or polar non-electrolytes. This is confirmed by oil field practice and analytical calculations of the real role of capillary forces. The impossibility of achieving complete hydrophobization of a heterogeneously wetted reservoir space along the length of penetration of filtrate with cationic surfactants deep into the formation from the wellbore was confirmed by both laboratory experiments and calculations of their adsorption on polymictic rock. Based on the materials presented in the three parts of this article, it is necessary to more consciously approach the selection of surfactants for process fluids in the methods of influencing the bottomhole formation zone based on the fundamental principles of formation fluids filtration, the role of reservoir surface wettability, colmatation processes, their prevention and elimination.

**Ключевые слова:**

призабойная зона пласта, коллекторы нефти и газа, технологические жидкости, фильтрация, смачиваемость, поверхностно-активные вещества, полярные неэлектролиты, адсорбция, адгезия, гидрофобизация, гидрофилизация, капиллярное давление, водонефтяные эмульсии.

Доказательная база сторонников необходимости гидрофобизации коллекторской поверхности в призабойной зоне пласта при воздействии на нее водных технологических жидкостей базируется на ошибочных представлениях об улучшении при этом фильтрации нефти по сравнению с водой. Критический анализ литературных источников информации по теме гидрофобизации призабойной зоны пласта свидетельствует о неверной предпосылке многих отечественных исследователей в трактовке основных положений механизма ее действия в реальном коллекторском пространстве на течение пластовых флюидов под влиянием гидравлического давления. Достижимая в промышленных условиях эффективность от присутствия, в частности, катионных поверхностно-активных веществ объясняется не переводом ее в гидрофобное состояние, а в лучшем случае частичной гидрофилизацией и рядом других сопряженных эффектов: углеводородонасыщением призабойной зоны пласта, комплексным действием кислотных составов и др. Более приемлемой и объяснимой является необходимость поддержания в призабойной зоне пласта гидрофильного состояния коллекторской поверхности, что обеспечивается неионными поверхностно-активными веществами и/или полярными неэлектролитами. Это подтверждается нефтепромышленной практикой и аналитическими расчетами реальной роли капиллярных сил. Недостижимость полной гидрофобизации разномоченного коллекторского пространства по длине проникновения фильтрата с катионными поверхностно-активными веществами вглубь пласта от ствола скважины подтверждается как лабораторными экспериментами, так и расчетами по их адсорбции на полимиктовой породе. На основании изложенных в трех частях данной статьи материалов следует более осознанно подходить к выбору поверхностно-активных веществ для технологических жидкостей в методах воздействия на призабойную зону пласта с опорой на фундаментальные основы фильтрации пластовых флюидов, роли смачиваемости коллекторской поверхности, коагуляционных процессов, их предупреждения и устранения.

© **Viktor N. Glushchenko** – PhD in Engineering (tel.: +007 (910) 220 86 63, e-mail: vng.51@mail.ru).© **Grigoriy P. Khizhnyak** (Author ID in Scopus: 36711848000; ORCID: 0000-0003-2138-7083) – Professor, Doctor in Engineering, Professor at the Department of Oil and Gas Technologies (tel.: +007 (905) 863 76 55, e-mail: xgp@mail.ru). The contact person for correspondence.© **Mikhail S. Turbakov** (Author ID in Scopus: 36443127500, ORCID: 0000-0002-9336-5847) – PhD in Engineering, Associate Professor, Associate Professor at the Department of Oil and Gas Technologies (e-mail: turbakov@mail.ru)© **Dmitriy V. Kobayakov** – Researcher at the Laboratory of Natural Gas Hydrates (e-mail: kvd@gmail.com)© **Глущенко Виктор Николаевич** – кандидат технических наук (тел.: +007 (910) 220 86 63, e-mail: vng.51@mail.ru).© **Хижняк Григорий Петрович** – доктор технических наук, доцент, профессор кафедры нефтегазовых технологий (тел.: +007 (905) 863 76 55, e-mail: xgp@mail.ru). Контактное лицо для переписки.© **Турбаков Михаил Сергеевич** – кандидат технических наук, доцент, доцент кафедры нефтегазовых технологий (e-mail: turbakov@mail.ru)© **Кобяков Дмитрий Вадимович** – научный сотрудник лаборатории природных газовых гидратов (e-mail: kvd@gmail.com)

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Introduction

The method of reservoir surface hydrophobization considered in various variants [1, 2] according to the authors of these works is aimed at improving the state of the bottomhole formation zone (BFZ) for the advanced inflow of oil into the wellbore compared to the aqueous phase. However, its evidence base was built on the isolated and exaggerated role of capillary pressure, as well as the possibility of solving it with cationic surfactants (CSA) alone.

"We must be ready to reconsider any of our ideas. This principle requires "courage of mind." We must change the idea when there are compelling reasons, evidence forcing us to change it. This principle requires "honesty of mind." We must not change ideas arbitrarily, without sufficient grounds. This principle requires "wise restraint" [3].

Numerous publications on the topic of the possible limited or negative use of cationic surfactants and other hydrophobic agents in technological processes of treating the bottomhole zone (BHZ) of productive formations cited in works [2, 3] remained unnoticed. In the article [4] and monograph [5] V.N. Glushchenko considered the issues of the preference of BHZ hydrophilization with non-ionic surfactants (NSA) and/or polar non-electrolytes as an alternative to cationic surfactants.

In accordance with the task set in [2] of considering wettability taking into account the general state of the BFZ, where many technogenic processes accumulate throughout the entire period of well operation, we study their course in more detail.

During the development of the oil and gas production industry, technologies of "chemical" impact on the BFZ were solved mainly in three directions:

- preventing the occurrence of negative processes in the BFZ by carefully selecting the composition of process fluids (PF);
- eliminating the consequences of the deteriorated state of the BFZ from previous impacts and those occurring during well operation;
- use of complex technologies of the BHZ carried out in one cycle.

Immediately after the experimental clarifying the negative effect of the BFZ hydrophobization with surfactants in 1992 [3], A.T. Gorbunov and his colleagues, without diminishing the role of their work, turned to the using complex technologies of influence.

In this regard, following the first direction the choice of the PF composition and the method of its use should be based on the maximum considering the factors of the probable negative impact on the state of the BFZ, their minimization, as well as the simultaneous possible elimination of the colmatating factors already present in the BFZ to increase the well productivity coefficient  $K_{pr}$  for oil with a decrease in the water cut of the product in accordance with the Dupuis equation.

To preserve the filtration properties of the productive formation combined well killing is effective [6–36]. To modify the properties of PF including well-killing fluid (WKF) and water-based acid compositions (AC) various surface-active compounds are most widely used, which we grade into two conventional groups: by hydrophilizing action, including ethoxylated nonionic surfactants (NSA) and polar non-electrolytes (alcohols, ethers, ketones), and hydrophobizing – in relation to the surface of sand and polymictic reservoirs: CSA.

Table 1

Values of the wetting angle  $\Theta$  of hydrophilic and hydrophobic surfaces at different concentrations of DDAB in NSA solutions

Parameter	Value						
DDAB, g/dm <sup>3</sup>	0	3·10 <sup>-5</sup>	3·10 <sup>-3</sup>	0,3	0,6	1,5	3,0
Environment:	$\Theta$ , degrees in the environment:						
Hydro philic	0	47	85	91	82	57	0
Hydrophobic	106	105	96	72	60	0	0

Table 2

The influence of the concentration of nonionic surfactants AF<sub>9</sub>-10 on the wetting angle  $\Theta$  of the quartz surface

Parameter	Value				
AF <sub>9</sub> -10, %	0	0,1	0,2	1,0	2,0
Environment:	$\Theta$ , degrees in the environment:				
Oil	106	94	90	85	66
Diesel	86	-	56	40	16

Bench and Field Tests

In order to determine the conditions for the maximum manifestation of the inherent positive properties of each group of chemical compounds in the PF composition, the latter should be divided into WKF, AC, hydraulic fracturing fluids (HFF), compositions for the delivery of scale inhibitors (SI) to the BFZ or individual injection into the BFZ for the purpose of its decolmatation.

It should be noted right away that American specialists [37] do not allow the use of surfactants in aqueous HFF fluids on terrigenous reservoirs, where, due to the hydrophobization of the crack surface, water migrates to the smallest pores which reduces the relative phase permeability (RPP) for oil. The use of proppants with a hydrophobic surface is also not recommended.

In gas shale formations for aqueous HFF fluids, the best solution is the introduction of nonionic surfactants in order to reduce the surface tension and hydrophilize the reservoir surface to increase the RPP for gas and prevent the formation of a water-oil emulsion (WOE) [38]. An alternative to cationic surfactants are ethoxylated nonionic surfactants which are widely and successfully used in various fluids at the stages of reservoir drilling, development and operation of wells [5, 39–42], which requires a comparative assessment of their effectiveness in the compositions of aqueous fluids. Thus, the wettability of a quartz and real terrigenous reservoir surface is not so unambiguous and can have an inverting nature depending on its initial state and the concentration of the surfactant. In particular, for solutions of dodecylammonium bromide (DDAB) cationic surfactant, the following values of  $\Theta$  are recorded for a glassy hydrophilic and hydrophobized octadecane (C<sub>8</sub>H<sub>18</sub>) surface in the concentration range [39] (Table 1).

Accordingly, for aqueous solutions of nonionic surfactants AF<sub>9</sub>-10, a stable hydrophilizing effect is observed on the quartz surface both in a more viscous oil environment and in diesel fuel (Table 2). Although on a purely hydrophilic surface they can also exhibit a weak inversion effect without bringing it to a hydrophobic state ( $\Theta < 90^\circ$ ). While treating a quartz surface with aqueous solutions of nonionic surfactants, the applied oil droplets practically do not wet it and slide along such a substrate which increases the RPP for oil compared to water in such channels.

Table 3

Change in core wettability during filtration of 0.1 % aqueous solutions of AF<sub>9</sub>-12

Parameter	Value			
Number of cerns	$k_{sp}, \mu\text{m}^2$	$m, \%$	$M_0$	$M_1$
Hydro philic – 4	0,164	23,5	0,93	0,50
Hydrophobic – 5	0,172	31,2	0,09	0,23

Table 4

Influencing the type and concentration of surfactants in aqueous WKF ( $p = 1170 \text{ kg/m}^3$ ) on the values of the core permeability recovery coefficient for oil (according to data from [48])

Core characteristics				SA in WKF model		Oil coefficient of permeability $k_{sp}, \mu\text{m}^2$		$\beta_0, \%$
$L_m, \text{cm}$	$d_m, \text{cm}$	$m, \%$	$k_c, \mu\text{m}^2$	Type	%	Before	After	
Bed sandstone of D <sub>1</sub> Romashkinskoye field								
16.5	2.8	21.04	0.167	–	–	0.145	0.071	49
17.1	2.8	21.65	0.173	ML-81B	1.0	0.143	0.119	83
16.7	2.8	20.74	0.158	Neftenol VVD	1.0	0.134	0.122	91
16.3	2.8	20.33	0.164	Neftenol VKS	1.0	0.138	0.128	93
16.5	2.8	22.05	0.155	Neonol AF <sub>9</sub> -12	–	0.116	0.091	79
Polymictic bed sandstone BS <sub>10</sub> Ust-Balyk field								
16.4	2.8	22.05	0.129	–	–	0.056	0.015	27.1
16.7	2.8	20.91	0.140	Neftenol VVD	0.5	0.042	0.016	38.5
16.4	2.8	20.99	0.148	Neftenol VVD	2.0	0.037	0.005	11.9
16.8	2.9	19.83	0.135	Neftenol K	2.0	0.036	0.026	71.5
16.8	2.9	19.69	0.131	IVV-1	2.0	0.038	0.014	35.9

Table 5

Permeability recovery coefficients  $\beta$  for the BS<sub>10</sub> formation of the Ust-Balyk field for various killing fluids

Parameter	WKF	KCl	KCl + 1 % IVV-1	CaCl <sub>2</sub>	CaCl <sub>2</sub> + 1 % IVV-1
Value	$\beta, \%$	0.57	0.53	0.21	0.46

In reservoir conditions a number of coupled processes will occur due to heterogeneous wettability of the filtration channels surface, from a linear increase in the degree of its hydrophobicity in hydrophilic areas, a hysteresis transition to their hydrophilization at a high concentration of cationic surfactants and hydrophilization of hydrophobic surfaces. This again gives the porous medium the same mosaic wettability but in an artificial form.

In terms of reducing the interfacial tension at the boundary with oil, aqueous and isoconcentrated solutions of nonionic surfactants and cationic surfactants have a comparable effect. In work [43] a 30–40 % higher rate of spontaneous impregnating 0.35 % aqueous solutions of ethoxylated alcohols and alkyl sulfates in intermediately wetted and predominantly hydrophobic cores containing 30–40 % oil and 60–70 % water from the pore volume at  $k = 0.014\text{--}0.263 \mu\text{m}^2$  and  $m = 15\text{--}22 \%$  for 30 days was established compared to water.

During this time, the filtration surface of the cores changed its wettability from hydrophobic to slightly hydrophobic by ~0.7 points from  $-0.8 \div -1.0$  to  $0 \div -0.3$  on the U.S.B.M. scale ( $-1$  – purely hydrophobic, and  $+1$  – purely hydrophilic).

In the presence of oil the wetting ability of water can decrease [44], and that of the oil itself can increase [45–47]. Later, partial hydrophilizing the surface of hydrophobic cores and hydrophobization of hydrophilic ones were established by B.I. Tulbovich [47].

While filtering 10 PD 0.1 % aqueous solutions of AF<sub>9</sub>-12 with different initial wettability M<sub>0</sub> (according to the author's method or the Amott scale) through them until M<sub>1</sub> values were obtained at the end of the experiment, the data are given in Table 3.

As a result of such an inverting effect, AF<sub>9</sub>-12 samples acquired an intermediate wettability character which

according to a number of foreign specialists has the highest oil recovery coefficient.

With good compatibility with highly mineralized solutions of WKF, nonionic surfactants have low thermal stability ( $<100 \text{ }^\circ\text{C}$ ) which increases with rise in the degree of their ethoxylation, acidification of the medium and with the additional introduction of alcohols. Triethanolamine salt of ethoxylated alkyl sulfate AF<sub>9,10</sub>-12 (Neftenol VVD) or ethoxylated cationic surfactants (Neftenol K) have increased thermal stability and interfacial activity [39].

The results of bench tests on cationic surfactants in the composition of mineralized WKF for the values of the permeability recovery coefficient of cores for oil are also not at all impressive which is presented in Table 4 [48].

The experiments used extracted cores with the creation of residual water saturation in them, then the oil model was filtered with the determination of phase permeability, an aqueous solution of WKF without surfactants and its concentration-species composition was pumped in the opposite direction to 100% water cut at the outlet of the core and then the oil model was filtered in the forward direction until the pressure was stabilized with the fixation of phase permeability and the calculation of the permeability recovery coefficient  $\beta$ . Apparently, the advertised facts of successful industrial results of well killing with aqueous solutions of surfactants are a consequence of the hydrophilizing ability of surfactants in relation to hydrophobic surfaces of the reservoir space, a decrease in  $\sigma_{12}$  values, a demulsifying effect on the WOE and other factors that are not fully manifested in the cores.

Using a similar technique, the effect of KCl ( $p = 1180 \text{ kg/m}^3$ ) and CaCl<sub>2</sub> ( $p = 1260 \text{ kg/m}^3$ ) solutions with the addition of 1 % IVV-1 cationic surfactant on the

value of  $\beta$  for oil was studied in [49] on polymictic cores of the BS10 formation of the Ust-Balyk field (Table 5).

While simulating well killing with water in addition to 0.1 % Neonol BS-1 CSA on a core sample of silty sandstone with an initial gas permeability of  $k_g = 28.8 \cdot 10^{-3} \mu\text{m}^2$  and water saturation  $S_w = 0.542$  R. Sh. Salikhov and Yu. V. Pakharukov [50] established the phase permeability of the sample for oil

$k_0 = 8.03 \cdot 10^{-3} \mu\text{m}^2$ . After displacing oil with water in the opposite direction, the permeability for it was  $k_w = 0.66 \cdot 10^{-3} \mu\text{m}^2$ .

In the same direction WKF was injected and oil was further displaced with water establishing the values of  $k_w = 0.69 \cdot 10^{-3} \mu\text{m}^2$  and  $S_w = 0.824$ . Then, water with WKF was displaced with oil in the forward direction, and  $k_0 = 10.99 \cdot 10^{-3} \mu\text{m}^2$  was obtained. Thus, the phase permeability for oil increased by approximately 37 %, and for water – by approximately 4 %.

It is difficult to select thermally stable surfactants in concentrated solutions of  $\text{CaCl}_2$ ,  $\text{Ca}(\text{NO}_3)_2$ ,  $\text{CaBr}_2$  or their mixtures when the  $\beta$  value for oil in the initial state decreases proportionally to the increase in the density of WKF and the decrease in the reservoir permeability [51, 52]. The value of dynamic viscosity in such compositions reaches  $\sim 100$  mPa·s [52].

In particular, for the layers of the Abalak suite AC10–12 of the Priobskoye field, V.N. Gusakov et al. [51] obtained the following values of  $\beta$  (Table 6).

A similar negative pattern is characteristic while assessing the influence of aqueous WKF on low-permeability Jurassic formations and Achimov deposits.

A strange situation arises for the scientific community during considering in literary articles the effectiveness of compositions or reagents under conventional numbers, trade marks, or even simply “hydrophobizer” [49, 52–54]. Nevertheless, we will present the effect of the best of the five selected brands of surfactants – water repellents (WE) without specifying the concentration on the  $\beta$  values for the oil model on polymictic cores of the [BS]<sub>7</sub> formation of the Sorovskoye field in Western Siberia [54]. The cores were saturated with the formation water model to a residual  $S_w = 40$  % with the determination of their permeability for oil  $k_1$  in the forward direction at 87 °C. Then three PO WKF with WR were filtered in the reverse direction with the establishment of the phase permeability for PF and the value of the phase permeability for oil  $k_2$  in the forward direction was again recorded with the establishment of the value of  $\beta = k_2/k_1$ . The obtained results are presented in Table 7.

Even for the NaCl solution with the best WR, the result is not impressive.

Based on the results of killing more than 100 flooded wells at the Samotlor field with NaCl and KCl solutions with the addition of 1.5 % of the water repellent “Aquatek-510B” and 1.5 % of the scale inhibitor “Aquatek-510A”, representatives of NPO “Aquatek” in 2013 came to the following conclusion: “Despite the fact that the water repellent can be successfully used to prevent water blockage, the reagent is not particularly effective as a means of combating it” [53]. The author recognized the best solution as the injection of mutual solvents individually before killing the wells, for example, “Aquatek 400E”. The same conclusion was experimentally reached in the work [51]. In this regard, let us briefly consider the main properties of polar non-electrolytes. Figure 1 shows the concentration dependence of the wetting the paraffin surface with

Table 6

Effecting density of killing fluid on the coefficient of recovering permeability of Abalak suite AC<sub>10-12</sub> formation in Priobyie field

Parameter	WKF, kg/m <sup>3</sup>	1160	1350	1420	1517	1605
Value	$\beta$ , %	27	18	16	9	8

Table 7

Permeability recovery coefficients of polymictic cores of the formation in Sorovskoye field BS<sub>7</sub>

WKF	$k \cdot 10^3, \mu\text{m}^2$		$\beta$ , %
Solution NaCl $p = 1140 \text{ kg/m}^3$	Oil – 20.1	WKF – 2.1	Oil – 10.03
The same + WR № 1	Oil – 13.6	WKF – 0.74	Oil – 9.85

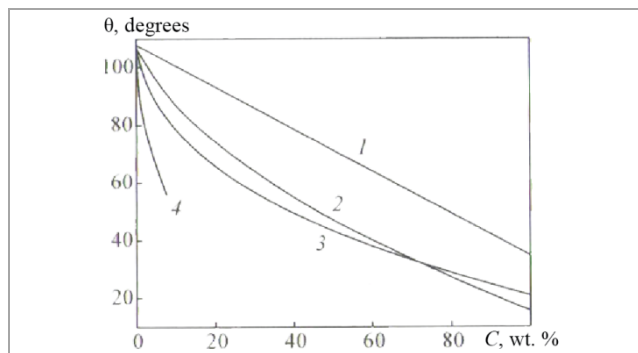


Fig. 1. Change in wettability of paraffin surface with aqueous solutions of alcohols at 25 °C: 1 – methanol; 2 – ethanol; 3 – propanol; 4 – butanol

Table 8

The influence of the concentration of ethanol in an aqueous solution on the contact angle  $\theta$  of a hydrophobic surface

Parameter	Value					
$C, \text{r/dm}^3$	0	35	87	299	537	754
$\theta$ , degrees	108	101	95	77	66	44

Table 9

$C_w$  and  $C_{inv}$  values of aliphatic alcohols

Alcohol	$C_w$ , mass. %	$C_{inv}$ , gr/dm <sup>3</sup>
Methanol	Unlimited	159
Ethanol	Same	127
Isopropanol	Same	50
Propanol	Same	20
Isobutanol	9.0	14
Butanol	7.9	8
Isopentanol	2.8	5
Hexanol	0.6	–

aqueous solutions of alcohols [39]. The values of the contact angle of wetting a hydrophobic surface by drops of an aqueous ethanol solution are presented in Table 8.

Starting with butanol, aliphatic alcohols have limited solubility in water  $C_w$  which is shown below and also reduce their concentration in water required to convert a hydrophobic surface into a hydrophilic one ( $C_{inv}$ ) [41] (Table 9). Alcohols are practically indifferent to changes in the wettability of hydrophilic surfaces. The term universal solvents (US) or mutual solvents (MS) is used for alcohols C1–C3, as well as carboxylic acids C1–C3, acetone, dioxane, lower ethers of alcohols and glycols (cellosolves) which are capable of dissolving in water and hydrocarbons. However, when a certain amount of water is introduced into a mixture of hydrocarbons and alcohols, stratification occurs with enrichment of the hydrocarbon phase with alcohols to a greater extent, the higher their molecular weight.

Table 10

Values of interfacial tension at the boundary of an aqueous solution of isopropanol with toluene and a mixture of a wide fraction of hydrocarbons with isopropanol at the boundary with the model of Cenomanian water

IP, mas. %	$\sigma_{12}$ , mN/m	WHF + IP, vol. %	$\sigma_{12}$ , mN/m
7.8	19.2	100	0
14.4	12.1	80	20
26.3	5.8	60	40
53.9	1.9	50	50
71.6	0.9		
98.4	0.5		

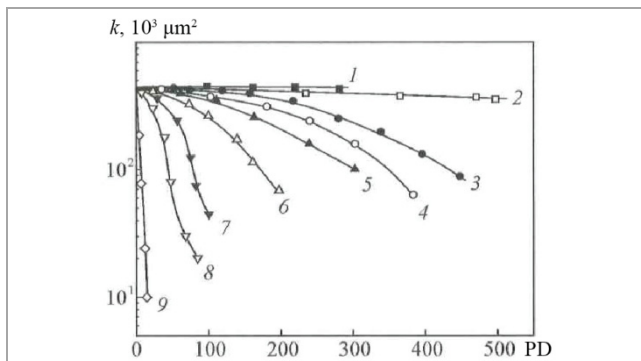


Fig. 2. Change in the permeability of Cypress sandstone ( $k_g = 1 \mu\text{m}^2$ ,  $k_w = 0.45 \mu\text{m}^2$ ) with the volume of water filtered through it depending on the content of fine particles in it: 1, 2, 3, 4, 5, 6, 7, 8, 9 – 2; 2.5; 14; 26; 50; 48; 94; 110; 485 g/t, respectively

If the process of alcohol transition into oil is not accompanied by the formation of new AAS in a separate phase and their precipitation under reservoir conditions, then this is a positive moment of imparting hydrophilizing and demulsifying properties to oil. Below are the values of interfacial tension  $\sigma_{12}$  at the boundary of an aqueous solution of isopropanol (IP) with toluene and a mixture of a wide fraction of hydrocarbons (WHF) with IP at the boundary with the Cenomanian water model (Table 10).

Considering the low solubility of higher alcohol homologues in water with higher interfacial activity and hydrophilizing function, they are composed with lower alcohols. From patent information, IP compositions with octanol in a volume ratio of 5:1 are known which add 30–70 vol.% to 15 % HCl.

According to another patent, this composition in a ratio of 2:1 is combined with nonionic surfactants [41]. Abroad and, less frequently, in Russia, in order to prevent and destroy the WOE, and to clear the BFZ from mechanical impurities, ethylene glycol monobutyl ether (EGMBE) is introduced into the AC which dissolves in water and hydrocarbons with an effective reduction in interfacial tension. In addition, it does not initiate the precipitation of asphaltenes from the oil.

Solid-phase colmatation is the most serious factor in reducing the reservoir properties of productive reservoirs, as shown in Fig. 2 according to R.N. Tuttle, J.H. Barkman [5]. Its occurrence is due to the introduction of fine particles from the composition of the solid-phase fluid into the BFZ at the stages of primary and secondary opening of productive formations, well killing, acidic and especially clay-acid BHZ. The second source is the suffusion of particles from the composition of the reservoirs due to their destruction by the water flow from injection facilities with migration along the formation. With regard to well killing, all methods of preliminary cleaning of the liquid gas can be recommended, as well as the introduction of small quantities of polymers for flocculation of fine particles already in the wellbore with their deposition on the bottomhole [5]. As a rule, such

particles are concentrated in the most open channels, fracture space, acid dissolution channels, where, upon contact with oil, they become hydrophobic and form adhesive-active aggregates. The work of adhering particles on a solid surface is determined by the Dupre-Young relationship [55]:

$$W_a \approx \sigma_{12}(1 - \cos\Theta), \text{ H/m.} \quad (1)$$

The issues of easy removing oil-wetted fine impurities from the BFZ are considered from the position of the free energy of their specific contact interaction per  $\text{m}^2$  according to B.V. Deryagin [56]:

$$\sigma_\tau \approx \sigma_c \Delta S (1,5Z\phi/\pi r^2)^{3/2}, \text{ N/m,} \quad (2)$$

which in calculations for hydrophobized particles can be replaced by  $\sigma_{12}$ ; where  $\sigma_c$  is the interfacial tension at the particle-medium boundary, N/m;  $\Delta S$  is the area of individual particles contact,  $\text{m}^2$ ; Z is the coordination number of the particle packing, reaching six at a volume fraction of particles in the liquid of  $\phi = 0.52$ ; r is the radius of the particles, m.

In an aqueous medium with a dissolved surfactant, the values of  $\sigma_\tau$  obey the relationship

$$\sigma_\tau = 2\sigma_c(1 - \cos\Theta), \text{ N/m.} \quad (3)$$

At  $\Theta = 20^\circ$ ,  $\cos\Theta = 0.94$  for hydrophilic particles in an aqueous medium with surfactants with values of  $\sigma_c = 30 \text{ mN/m}$  we obtain  $\sigma_\tau \approx 0.0036 \text{ N/m}$ , and for the hydrophobic state of particles  $\Theta = 100^\circ$ ,  $\cos\Theta = -0.17$  in a hydrocarbon medium  $\sigma_c = 30 \text{ mN/m}$  we have  $\sigma_\tau \approx 0.070$ , or  $\sim 20$  times greater.

For example, the force of individual contact between clay particles of fr.  $7.5 \pm 1 \mu\text{m}$  in an aqueous medium is additionally reduced by dissolving aliphatic alcohols in it by approximately two times for their following concentrations ( $\text{g/dm}^3$ ): methanol  $\sim 16$ , ethanol  $\sim 10$ , propanol  $\sim 5$  and butanol  $\sim 4$  [5]. Hydrophobized glass beads of 1 mm in size and  $\Theta = 100^\circ$  have a value of  $\sigma_\tau \leq 40 \cdot 10^{-3} \text{ N/m}$  in air and  $\sigma_\tau \approx 80 \cdot 10^{-3} \text{ N/m}$  in water but they coagulate intensively. In alcohol solutions, the values of  $\sigma_\tau$  can be reduced by four times or more – in proportion to their hydrophilizing capacity and concentration (see Fig. 1).

The force of individual contact in hydrophilic quartz particles of 5–10  $\mu\text{m}$  in an aqueous medium is  $\sim 1.2 \cdot 10^{-6} \text{ N/contact}$ , and in a medium of  $0.05 \text{ g/dm}^3$  of cetylpyridinium bromide surfactant solution it increases to  $\sim 5.4 \cdot 10^{-6} \text{ N/contact}$ , i.e. 4.5 times.

However, the best solution for removing mechanical impurities hydrophobized by asphalten deposits of oil from the reservoir space is the use of alcohol or EGMBE solutions in light hydrocarbons, which provide both the washout of the hydrophobic film and the hydrophilization of the surface. According to the ratio (1), this also helps to reduce the adhesion of particles on the reservoir surface.

It can also be concluded that the presence of surfactants in the PF with the assumption of their parallel effect on the removal of solid-phase colmatants from the BFZ will have a negative effect. At the same time, the fact of the hydrophobizing effect of surfactants on clay and other particles of mechanical impurities in the WKF composition will be positive for their flocculation and sedimentation on the bottomhole while the WKF is in the wellbore.

The presence of alcohols in the GCM minimizes the formation of aluminosilicate gels in the process of MAT and stabilization of clay minerals against their disintegration [57]. This is confirmed by the results of filtration studies on the impact of alcohol-containing hydrochloric and clay-acid compositions on terrigenous reservoirs and by field data on the bottomhole treatment zone with such compositions at the fields of Western Siberia [58].

Water saturation of the bottomhole zone from the PF composition is perhaps the second most important factor in their negative impact on the well kpr. Its influence extends to a decrease in the relative permeability of oil, emulsion formation with oil, scaling when mixed with formation waters and a number of other negative processes.

As noted in [3] the role of capillary pressure with variation in  $\sigma_{12}$  of aqueous PF extends to the network of micro-open channels and dead-end oil-containing pores, as well as the movement of oil and AAS-stabilized water globules which is discussed below. The prevailing flow of PF and water filtrate penetrates into macro-open oil-containing channels, which are the main "arteries" for the inflow of oil. In this case, the function of the surfactants introduced into the WKF consists more in their hydrophilizing ability with the aim of facilitating the removal of mechanical impurities and "activating" the movement of oil flow through such channels. In the works [51, 52], an increase in the  $\beta_0$  values for oil after simulating their "killing" with highly mineralized aqueous WKF and an increase in the oil filtration rate was established on low-permeability cores of a number of West Siberian fields. This indicates a certain reserve in increasing the relative permeability of oil at the stage of well development by regulating depression.

Particularly "destructive" is the penetration of the aqueous phase into hydrophobic reservoirs with an initially low water content. These include productive formations of the Bazhenov suite and Domanik deposits. Here, one killing with aqueous compositions is enough for the oil permeability to decrease sharply. Thus, below are the corresponding experimental results of O.B. Bennion et al. [59] on the filtration of purified formation water through oil-saturated sand cores with a low initial water content  $S_w^0$  with an estimate of the current water content  $S_w$ , oil permeability at the beginning  $k_0$ , at the end of the experiment  $k$  and its decrease (Table 11). In the work [60], bench experiments on Berea sandstone cores were used to study the efficiency of the technology for unblocking the BFZ from water blockage after simulating killing or hydraulic fracturing with aqueous compositions. By successive filtration of a 2 % KCl solution and an oil model into a core with a gas permeability of  $k_g \approx 0.05 \mu\text{m}^2$  at 60 °C, oil saturation conditions with residual water were created. Then, a 2 % KCl solution with the addition of 1 % nonionic surfactants or 1 % cationic surfactants was filtered in the reverse direction, and then oil was filtered in the forward direction to estimate  $\beta$ , which turned out to be ~1.7 times higher when using nonionic surfactants. Almost complete restoration of the phase permeability for oil was achieved by injecting a 1 % nonionic surfactant solution in methanol in the reverse direction. A number of foreign solutions also provide for the implementation of multi-volume US injection into the BFZ at the stage of inflow stimulation after killing or inflow stimulation, more often in hydrocarbon compositions which is covered in detail in [40, 41].

In the work [51], in order to reduce the negative impact of heavy liquid hydrocarbons, it was proposed to pre-place buffer rims of methanol, MS or hydrocarbon solvent "Nefras" in the BFZ. The  $\beta_0$  values for oil increased for methanol from 20 to 52 %, MS – from 20 to 36 % and "Nefras" – from 20 to 26 %. Note, without any "hydrophobizers". Similar solutions for preliminary and final injection of polyglycol or aromatic solvent rims into the BFZ during the treatment with alcohol-containing solutions of 22 % HCl were proposed by A.G. Telin et al. [61]. Apparently, such integrated technologies for well killing and BFZ AT will be most effective at the current water saturation of the BFZ up to the intersection point of the RPP curves without "dagger" water breakthroughs into the wellbore of the producing wells.

Table 11

Experimental results of O.B. Bennion et al. [59] on the filtration of purified formation water through oil-saturated sand cores

$S_w^0, \%$	$k_0 \cdot 10^3, \mu\text{m}^2$	$S_w, \%$	$k', \mu\text{m}^2$	$\frac{k_0 - k'}{k_0} \cdot 100, \%$
4.0	156.6	22.6	5.83	96.3
2.6	51.8	20.6	3.42	93.4
4.5	132.3	34.1	5.83	95.6

Table 12

Efficiency of acid compositions in the BHZ in the Jurassic formations of the Lovinskoye field

Parameter	Value			
AV	HCl	HCl + IVV-1	HCl + HF + IVV-1	HCl + HF + Sinol-kam
Efficiency, %	33	30	79	44

I.B. Dubkov and Yu. V. Zemtsov [62] in the analysis of 171 bottomhole treatment zones with HCl and HCl + HF solutions of the Jurassic formations of the Lovinskoye field established their minimum efficiency when 0.1–2 % of the IVV-1 cationic surfactant was added to the acid solutions and the maximum efficiency was achieved with a mixture of nonionic surfactants and cationic surfactants (Sinol-Kam), which is presented in Table 12.

The formation of stable VNE in the bottomhole zone, as well as the globular movement of phases along filtration channels of varying openness, is also a serious complicating factor for oil inflow into the wellbore.

The presence of WOE in the bottomhole zone was recorded in field conditions by their extraction during well development, AT and bringing wells into operation using special studies on cores and formation models [5, 63].

The conditions for their easy formation are:

- the hydrophobic state of the walls of filtration channels with an increase in their openness and the presence of cracks;
- gradual saturation of the oil phase with water;
- increase in oil viscosity with increasing amount of AAS;
- presence of finely dispersed solid phase, especially oxides, iron sulfides, aluminosilicate gels, gas phase and asphaltenes, which is typical for the process of BFZ AT;
- barocyclic loads on the BFZ during tripping processes in the wellbore, perforation, inflow stimulation, etc.

A predictive assessment of the occurrence of WOE in the reservoir space can be carried out based on the values of the capillary number  $N_k > 10^{-4}$ :

$$N_c = \frac{\eta_o V_o}{\sigma_{12}} \approx \frac{\eta_o \gamma_o r_k m}{\sigma_{12}}, \quad (4)$$

where  $\eta_o$  – dynamic viscosity of oil, Pa·s;  $V_o$  – true oil filtration rate, m/s;  $m$  – porosity, fractions of a unit;  $\sigma_{12}$  – interfacial tension between oil and water phase, N/m;  $\gamma_o$  – oil shear gradient,  $\text{c}^{-1}$ ;  $r_k$  – radius of filtration channels, m.

Taking into consideration  $\eta_o = 10 \text{ mPa}\cdot\text{s}$ ,  $V_o \approx 10^{-5} \text{ m/s}$  (300 m/year),  $\sigma_{12} = 10 \text{ mN/m}$ , It is possible to note the difficulty of the appearing WOE during conventional flooding of formations, and the oil will be displaced in piston or globular modes in the depth of the formation.

In the BFZ, with a well flow rate of  $Q = 50 \text{ m}^3/\text{day}$ ,  $h = 10 \text{ m}$ ,  $m = 0.2$ ,  $R = 0.2 \text{ m}$  behind the radius of the casing, the values of  $V_o \approx 2.3 \cdot 10^{-4} \text{ m/s}$  which already contributes to the formation of WOE. Their occurrence is even more likely when OF injection into the BFZ, for example, at a

rate of 3 m<sup>3</sup>/h in other identical conditions, when the value of Vo will be 3.3·10<sup>-4</sup> m/s.

A promoting factor is the presence of perforations in the casing. At their number of 200 and diameter of 1.27 cm the value of Vo reaches ~3.3·10<sup>-3</sup> m/s and in the matrix after their completion it is ~1.7·10<sup>-2</sup> m/s. The main obstacle to the passage of oil or WOE globules is the narrowing of the filtration channels under conditions where their radius rr exceeds the radius of the channels rk. Such conditions are characterized by a hysteresis of the contact angles of wetting the walls globules of the filtration channels in the head part Θ<sub>A</sub> and at the contact with the displacing liquid Θ<sub>R</sub>, as well as the values of σ<sub>12</sub> with the resulting gradient of capillary pressure P<sub>k</sub> along the length of the channels L:

$$\frac{P_k}{L} = \frac{4\sigma_{12}}{d_3} \left( \frac{1}{d_k} - \frac{1}{d_r} \right) (\cos\Theta_R - \cos\Theta_A), \text{ Pa/m}, \quad (5)$$

where d<sub>3</sub>, d<sub>k</sub>, d<sub>r</sub> – characteristic diameters of rock grains, channels and globules, m.

Assuming for normal conditions of oil displacement by water σ<sub>12</sub> = 10 mN/m d<sub>3</sub> = 10<sup>-4</sup> m, d<sub>k</sub> = 10 μm, d<sub>r</sub> = 15 μm, cosΘ<sub>R</sub> = 1 and cosΘ<sub>A</sub> = -1, we obtain ΔP<sub>k</sub> ≈ 26 MPa/m, which is an insurmountable obstacle to the passage of globules through such a reservoir space.

One of the options for reducing ΔP<sub>k</sub> is to hydrophilize the reservoir surface to eliminate hysteresis and sharply reduce the σ<sub>12</sub> values. For example, under conditions of σ<sub>12</sub> = 0.01 mN/m, we obtain ΔP<sub>k</sub> ≈ 1.3 MPa/m, which will facilitate the movement of globules at a distance of ~3 m from the wellbore with a depression in it of 5 MPa. An even more complex situation may arise with the simultaneous movement of a hydrophobized suspension and aggregates of globules of highly viscous WOE.

From these calculations close to reservoir conditions, it can be concluded that only the σ<sub>12</sub> values and the hydrophilization of the reservoir surface can be regulated, but it is difficult to implement them using surfactants alone due to their adsorption on the rock (see below). Here, there are two options that are used in oilfield practice: the introduction of practically non-adsorbable polar non-electrolytes into the RF in order to prevent the formation of stable WOE or with a minimum globule size and their effective destruction after the completion of repair work by pumping polar non-electrolyte solutions into the BFZ or individually, possibly in a composition of various non-electrolytes, non-electrolytes and surfactants with a high demulsifying effect which do not include CSA as described above. In the work [3] while considering the hydrophobic effect of cationic surfactants, attention is drawn to the fact that the data of studies in laboratory conditions on the wetting effect on quartz plates, impregnation of models of porous media with cationic surfactant solutions or water after their treatment with such solutions with the establishment of effective concentrations of cationic surfactants for this are far from their true behavior in the reservoir space of productive formations.

Thus, already in the process of preparing WKF containing mechanical impurities, adsorption of any surfactants occurs on them, and when pumped along the wellbore into the perforation interval, additionally on the surface of underground equipment, including finely dispersed corrosion products, asphalten deposits, salts, oil film which is noted in [49]. As surfactant solutions filter in the reservoir space, in addition to adsorption on the surface of differently wetted rock, they diffuse into the contacting oil phase, interact with AAS and, finally, are diluted with formation waters. This can sharply reduce

their concentration, increase the values of interfacial tension, which will affect the wetting function of surfactants and complicate the removal of aqueous filtrate from the BFZ.

Thus, the adsorption of surfactants is their Achilles heel, reducing the manifestation of target functions. Let us recall that the rejection of the widely advertised flooding with low-concentration OP-10 solutions (0.05 %) in order to increase oil recovery was mainly due to high adsorption losses under reservoir conditions.

According to the data summarized in [39], the adsorption of various surfactants on sand and sandstone of crushed cores varies within 0.5–13 g/kg of rock, and on clay minerals it reaches 75 g/kg. Limestones and dolomites absorb them up to 4 g/kg. Finely dispersed iron oxides (d = 0.5–0.8 μm) in the amount of 0.3–1 kg/kg at pH = 5 and 20 °C have the maximum adsorption capacity of cationic surfactants from their solutions (0.34 g/dm<sup>3</sup>). The degree of adsorption losses of surfactants increases with a decrease in the permeability of porous media (an increase in the specific filtration surface), an increase in the content of clay minerals in them, the mineralization of aqueous solutions, temperature and concentration of surfactants in aqueous solutions. For nonionic surfactants, lower adsorption losses are noted with predominantly physical contact which facilitates their partial washing by the subsequent water flow and the preservation of activity in such a solution. Thus, the adsorption of OP-10 in the terrigenous core k = 0.044 μm<sup>2</sup> from a 0.05 % solution in distilled water was ~0.5 g/kg at 20 °C, and from a 5 % CaCl<sub>2</sub> solution in the core k = 0.077 μm<sup>2</sup> ~2 g/kg.

On bulk models of porous media saturated with formation water (ρ = 1100 kg/m<sup>3</sup>) L = 1 and 3 m, d = 1 cm made of quartz sand and disintegrated sand core with a volumetric flow rate of 6 cm<sup>3</sup>/h at 23–25 °C, OP-10 and AF9-12 solutions were filtered in this water until solutions with the initial concentration of surfactant appeared at the outlet of the models. Then the injection of formation water was continued until the surfactants disappeared in it in order to determine the degree of their desorption [42]. The results obtained are presented in Table 13.

Thus, the irreversible losses of AF9-12 vary within the range of 16–21 %.

For formation conditions, a correlation formula for assessing the adsorption losses of surfactants has been proposed [39, 64]:

$$M = A_{\infty} \pi (R^2 - r_c^2) h (1 - m) \rho_p, \text{ kg}, \quad (6)$$

where A<sub>∞</sub> – maximum adsorption of surfactant, kg/kg; R is the radius of penetration of the filtrate with surfactant into the BFZ from the wellbore of radius r<sub>c</sub>, m; h is the effective perforated thickness of the formation, m; m is the formation porosity, fractions of a unit; ρ<sub>p</sub> is the density of the reservoir rock, kg/m<sup>3</sup>.

In accordance with (6), we will approximately estimate the adsorption losses of the cationic surfactant DON-52 based on the experimental data of A.T. Gorbunov et al. [65], obtained on disintegrated cores of the AV1 and BV10 formations of the Samotlor field by filtering DON-52 solutions through their models. For the cores of the AV1 formation, the values of A<sub>∞</sub> were 7.50–11.25 g/kg, and for the BV10 formation – 2.50 g/kg.

Taking A<sub>∞</sub> = 0.0025 kg/kg, h = 1 m, m = 0.2, r<sub>p</sub> = 2500 kg/m<sup>3</sup>, we have the following series of values of M for the radius of penetration of the WKF filtrate into the BFZ and, accordingly, its volume at this distance with a surfactant content at an initial concentration in the wellbore of 10 kg/m<sup>3</sup> (1 %) (Table 14).

It follows from these data that at a distance of 0.5 m from the wellbore, the adsorption will be 5.5 kg with a filtrate volume of 0.22 m<sup>3</sup> and a surfactant content of 2.2 kg. However, this amount is completely adsorbed at a distance of  $R \leq 0.3$  m, and then pure filtrate without surfactants will enter. Naturally, its removal from the BZP will be difficult due to the approach of the  $\sigma_{12}$  values to a solution without surfactants. In case of  $A_{\infty} = 11.25$  g/kg, the  $M$  value should be multiplied by 4.5 with identical filtrate volume and surfactant concentration in it. This indicates its complete adsorption already in the first 10 cm from the wellbore at a depth of  $R = 0.5$  m.

Consequently, from this point of view, non-ionic surfactants, and especially polar non-electrolytes, also have a clear advantage over cationic surfactants.

Summarizing the materials presented in the three parts of this article on the problem under consideration, it should be noted that, according to literary sources, it is reduced practically to the facilitated removal of the aqueous phase from the bottomhole zone with a decrease in the intensity of repeated water saturation using one "hydrophobizing agent", often without disclosing its chemical structure. The driving force behind these processes is considered to be hypertrophied capillary forces by the authors, an increase in the relative permeability of oil in the hydrophobic filtration space and a decrease in the relative permeability of the water phase without the use of hydrodynamic pressure, which contradicts modern ideas about the essence of such phenomena and hinders the development of truly scientific directions for solving this problem.

It is necessary to consider the BFZ as a dynamic system complicated by the occurrence of many negative processes of a colmatation nature.

And such a set of problems should be solved by complex technologies at all stages of reservoir opening, development and operation of wells. The very development of the hydrophobization topic pushed its supporters from the initially "narrow" approach, in particular, the use of one surfactants composition, to the transition from production facilities to injection facilities, integration with other compositions.

In a demonstrative form we set out the main scientifically substantiated prerequisites and ways of implementing the complex task of bringing the reservoir properties of the reservoir space in the BFZ in accordance with the remote part of the formation or even improving them.

**Conclusion**

A critical analyzing literary sources of information on the topic of bottomhole formation zone hydrophobization indicates an incorrect assumption of many domestic researchers in the interpretation of the main provisions of the mechanism of its action in the real reservoir space on the flow of reservoir fluids under the influence of hydraulic pressure.

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Table 13

Adsorption and desorption of OP-10 and AF<sub>9</sub>-12 solutions

SA	C, %	Absorption, Desorption,		Absorption, Desorption,	
		gr/kg	gr/kg	gr/kg	gr/kg
		L = 1 m (sand)		L = 3 m (sand)	
OP-10	0.05	0.51	0.38	0.23	0.13
		L = 1 m (core)		L = 3 m (core)	
AF <sub>9</sub> -12	0.1	1.19	1.0	1.02	0.78

Table 14

Adsorption losses of cationic surfactant DON-52 on disintegrated cores of formations AB1 and BV10 of the Samotlor field

Parameter	Value								
R, m	0.1	0.2	0.3	0.4	0.5	1.0	1.5	2.0	
M, kg	0.47	1.26	2.36	3.77	5.5	18.8	35.3	62.8	
V, m <sup>3</sup>	0.019	0.05	0.09	0.15	0.22	0.75	1.41	2.51	
C, kg	0.19	0.50	0.94	1.51	2.2	7.5	14.1	25.1	

Bench experiments on cores and reservoir models have proven the negative effect of reservoir space surface hydrophobization on the relative permeability of oil including cationic surfactants.

Foreign experts do not recommend the use of cationic surfactants in compositions for bottomhole formation zone treatment of producing wells.

Individual impact on the bottomhole formation zone with hydrocarbon compositions of hydrophobizers including cationic surfactants, AAS, finely dispersed hydrophobic silicon oxide, organosilicon compounds, indicates the main achieved effect of reducing the relative permeability of water due to hydrocarbon saturation of the reservoir space and/or plugging of highly conductive channels. Complex treatment of the BFZ with hydrocarbon surfactant compositions including AT with preliminary and subsequent injection of oil does not allow isolating the specific efficiency of the surfactant.

Theoretical calculations and literature data from bench experiments have established the real role of capillary effects in the reservoir space of the BFZ with applied hydraulic pressure and the presence of various surfactants in the composition of aqueous PFs with the preferring the hydrophilic state of the collector surface.

The observed efficiency of using surfactants in the composition of aqueous WKF and AC can be attributed to the reduction of interfacial tension, corrosion rate of steel equipment, cleaning from mechanical impurities in the wellbore, stabilization of clay minerals and moderate hydrophobization of the collector surface to an intermediate-wetted state.

An alternative method for preserving, restoring and increasing well productivity is the use of hydrophilizing non-ionic surfactants and/or polar non-electrolytes both in the composition of PFs and as technological rims at the stages of well killing, inflow stimulation and conducting BFZ AT.



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