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Directions for Improving the Compositions of Reverse Emulsions for Well Plugging

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Направления совершенствования составов обратных эмульсий для глушения скважин

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Well killing is the most common technological process in oil production. Inverse emulsions are used as an effective fluid in the combined killing method. At present, some potential possibilities of inverse emulsions have not been analyzed both in terms of improving their compositions and technological methods of practical implementation. The paper considers a number of promising research areas for obtaining, improving the compositions of inverted emulsions and the method of their application with injection into the bottomhole formation zone before filling the wellbore with mineralized water. Natural oils of co-emulsifiers – ethoxylated oil-soluble surfactants, and filtration reducers – technical lignosulfonates, starch, latex were proposed as constituent components of hydrocarbon media. Attention was focused on the need for experimental evaluation of the rate of settling of reverse emulsions along the wellbore in formation water and oil, obtaining and studying stable reverse emulsions using high-density salts of $CaBr_2$, $ZnBr_2$, $ZnCl_2$, polyhydric alcohols.

Ключевые слова:
глушение скважин, обратная эмульсия, фильтрация, глинистые коллекторы, продуктивный пласт, углеводородная среда, водная фаза, растворы солей, осмос.

Глушение скважин является наиболее распространенным технологическим процессом в добыче нефти. В качестве эффективной жидкости при комбинированном способе глушения используются обратные эмульсии. В настоящее время не проанализированы некоторые потенциальные возможности обратных эмульсий как в плане совершенствования их составов, так и технологических приемов практической реализации. В работе рассмотрен ряд перспективных научно-исследовательских направлений получения, усовершенствования составов обратных эмульсий и способа их применения с задавкой в призабойную зону пласта перед заполнением ствола скважины минерализованной водой. В качестве составляющих компонентов углеводородных сред предложены природные масла соэмульгаторов – этоксилированные маслорастворимые ПАВ, а понизителей фильтрации – лигносульфонаты технические, крахмал, латекс. Акцентировано внимание на необходимости проведения экспериментальной оценки скорости осаждения обратных эмульсий по стволу скважин в среде пластовых вод и нефти, получения и исследования стабильных обратных эмульсий с использованием высокоплотных солей CaBr2, ZnBr2, ZnCl2, многоатомных спиртов.

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Introduction

The characteristics of inverted emulsions (IE) are as follows [1–6]:

- the possibility of using a wide range of hydrocarbon media for their production (commercial oils, gas condensate, light petroleum products, synthetic hydrocarbons, natural oils, etc.) and the hydrophilic internal phase (water, aqueous solutions of salts, acids, alkalis, polymers, polyhydric alcohols, etc..);
- controlled effective viscosity values ($\eta_э$) depending on the shear gradient (pipe transfer pressure or reservoir filtration). An increase in the $\eta_э$ value causes an increase in the volume content of the aqueous phase, the concentration of emulsifiers, the viscosity of the hydrocarbon medium, an increase in the dynamics of mixing IE and the introduction of fine fillers. The decrease in $\eta_э$ is most intensively influenced by an increase in temperature;
- controlled filtration into the reservoir space in proportion to the created pressure drop and its permeability in inverse dependence on the IE viscosity. The presence of dispersed globules and additional content of finely dispersed antifiltrants (polymers, clay, soot, aerosil, chalk, bitumen, etc.) is the most limiting factor of IE infiltration into the bottomhole formation zone (BHZ) from the well bore;
- controlled thermal stability, i.e. the ability to separate into component phases by choosing the type and concentration of emulsifiers, the component composition of the phases, additional stabilizers, special demulsifying agents (surfactants, alcohols, acids, etc.);
- reduction of swelling and disintegration of water-sensitive clay minerals due to the hydrocarbon media of the IE and controlled salinity of the aqueous phase;
- low corrosion activity related to metal equipment compared to the dispersed internal phase;
- high absorption properties with respect to H_2S, which can be enhanced by the introduction of special neutralizers;
- prevention of hydrate formation along the wellbore at a high gas factor;
- dissolving and dispersing ability in relation to asphaltene-resin-paraffin deposits (ARPD) with their retention in volume.

These and a number of other properties of IE predetermine the efficiency of their use as a flushing fluid for drilling wells [1, 3–7], perforation medium at penetration of productive formations [1, 4, 6, 7], killing fluid (KF) during underground and workover operations [1–4, 7, 8]. Less frequently, IE is used as a hydraulic fracturing fluid [1, 9], above-packer fluids during well conservation [3], delivery of salt deposition inhibitors into the bottomhole cavity [10], limiting water inflows in production wells and redistributing water flows to injection facilities [1, 11–13], prevention of gas hydrates in the wellbore [14].

Problem Analysis

To date, a number of the most important potential capabilities of IE have not been revealed both in the way of improving their compositions and technological methods of practical implementation, in particular, in relation to killing wells.

The wells are killed before perforation and workover operations using two common technologies: complete replacement of the well fluid with killing fluid and combined killing with low-filtering block packs placed against the perforation interval followed by placement of lighter fluid, usually formation water or salt solutions, up to the wellhead [1, 3–8]. When using aqueous KL and polymer-thickened block packs, their filtration into the bottomhole cavity may contribute to a number of negative processes, mainly related to the colmatisation of fluid-conducting channels and water saturation of the bottomhole cavity [1–8, 15]. The latter factor is especially critical for low-watered well stock (water saturation $w \leq 30$ %), when the increase in the share of water saturation of the reservoir space sharply reduces the relative phase permeability (RPP) for oil inflow [8]. In this respect it is more preferable the IEs that have a low infiltration into the formation, and at decomposition to a lesser extent reduce the RPP for oil.

Experimental Solutions

By bench experiments on artificial and terrigenous cores from the South Kharampur South$_1$ formation with permeability values of $k = (6–8)\cdot 10^{-3}$ mkm^2 it has been determined their permeability recovery coefficient (β) on the oil model after contact with IE under repression at the level of $\beta = 0{,}92–1{,}02$ in the presence of finely dispersed filtration reducers as part of the IE, which provide the penetration of only hydrocarbon medium into the BHZ [1]. Similar results were obtained by foreign researchers [15].

One of the main reasons for early watering of production wells is the inherently low oil saturation of the producing formations. In particular, this was characteristic of the Sutorminskoye and Mamontovskoye fields in Western Siberia from the beginning of their development [16, 17]. It is assumed that a small area of increased water saturation is concentrated in the BHZ, which controls the determining flow of formation water into the wellbore through a network of the most permeable channels in proportion to the oil viscosity growth [18].

This fact requires the exclusion of additional introduction of water phase into the bottom-hole zone from the composition of the used drilling fluids during secondary penetration, killing of wells and other stimulation treatments, and, on the contrary, hydrocarbon saturation of the bottom-hole zone can positively affect the well production rate in the post-workover period.

By thickening commercial oil, gas condensate or diesel fuel with calcium resinate rosin and organobentonite it was produced the UTZ-VIP hydrocarbon liquid with a density range of 760–1200 kg/m^3 by additional injection of finely dispersed hydrophobic chalk [4]. It is stable up to $t = 80–120$ °C and is characterized by an increase in effective viscosity values with a temperature increase of up to 80 ˚C from 1000 to ~ 3600 mPa·s at low shear gradients. “UTZ-VIP” was successfully

Table 1

Results of the experiment on thickening a tarine solution

Parameter	Value									
Volume of additionally injected aqueous phase, %	–	10	20	30	40	50	60	70	100	150
	Tarin solution to diesel fuel ratio 30:70									
$\eta_э$, mPa·s	31	54	50	51	52	58	85	108	181	384
	Tarin solution to diesel fuel ratio It50:50									
$\eta_э$, mPa·s	30	51	78	118	176	247	–	–	–	–

used at production facilities in Western Siberia and Udmurtia during perforation, killing and conservation of wells as a block pack with a partial insertion into the BHZ.

In our opinion, development of hydrocarbon fluids with density values < 1000 kg/m^3 for combined killing of wells with abnormally low formation pressure (ALFP) is not very promising, because the wellbore is filled with fresh or low-density water, which will displace such block pack from the perforation interval. The best option would be to use hydrocarbon fluids with the density $\geq$ 1000 kg/m^3 with their partial insertion into BHZ.

Thus, in recent years it is paid more and more serious attention to the development of environmentally acceptable compositions for drilling and killing of wells, including secondary products of their cleaning on the basis of natural oils [2, 5, 19, 20].

The paper [19] describes obtaining of thickened castor oil with density 997 kg/m^3 by additional introduction of 4 % of calcium rosin resinate and 3 % of organobentonite. By introducing $CaCl_2$ solution up to 30 vol. %, 1040 kg/m^3 density of IE with thermostability up to 120 °C was obtained. Using 5-6 vol. % of cube residues from production of sebacic acid which is the part of castor oil by their saponification in diesel fuel with 40 % NaOH solution, structured gels with thermostability up to 90 ^{0}C were obtained [10]. Such gels spontaneously disperse the aqueous phase in the form of solutions of multivalent metal salts with the formation of IE.

Experimental Results

Below there are presented the results of experiments on thickening of tarin solution with acid number 22.2 mg caustic potash (KON)/g (50 % solution of tall oil pitch in hydrocarbons containing 16–21 % of resin acids and 27–38 % of fatty acids) in diesel fuel by pretreatment with 20 % NaOH solution and subsequent mixing with Devonian formation water of Romashkinskoye field with density 1128 kg/m^3 [1]. The values of η_e were taken at 20 °C and a shear gradient of 145.8 s^{-1} (Table 1).

Similarly, hydrocarbon gels and IEs can be prepared using available tall oil, tall oil fatty acids, rosin, and rosin oil.

The most acceptable approach to obtaining gels based on vegetable oils (sunflower, corn, cotton, rapeseed, soya, castor, palm, tall oil), as well as oil and fat tar, fish oil, etc. is to saponify them with alkaline agents at elevated temperatures. Subsequent introduction of solutions of salts of multivalent metals ($CaCl_2$, $MgCl_2$, $AlCl_3$, $FeCl_3$ and others) will make it easy to obtain the IE of the required density [2]

Such IEs may additionally contain antifilterants: lignosulfonates technical up to 4 vol. % and up to 2 % of starch [1]. In order to reduce oil consumption it may be used their compositions with low-toxic hydrocarbons.

When killing low-temperature formations as block packs or preliminary injection into the bottomhole cavity of the rim with subsequent placement in the wellbore of formation water or aqueous solution of the required density hydrocarbon solutions of oil-soluble ethoxylated surfactants, in particular neonol AF_9-6, may be recommended. Such compositions, as well as their combination with neonol AF_9-12, are characterised by spontaneous mixing with the aqueous phase with an intensive increase in viscosity up to a temperature of 35–40 °C [12]. In [21], the formation of IE with a paste-like structure was found at a concentration of neonol AF_9-6 in hydrocarbons of 8–10 % after introduction of 6 to 10 volumes of mineralised water (130–200 g/dm^3).

One of the distinctive properties of water- and oil-soluble ethoxylated surfactants, such as neonol AF_9-6, is the stabilisation of direct emulsions at low temperatures and, as the temperature increases, of the reverse emulsions due to a complete transition to the oil phase [2, 22]. In this way, it can be obtained IEs from direct microemulsions with the content of aqueous phase up to 90 % by heating up to 30–50 °C [23].

Such formulations under the brand name SNPH-9633 have been used extensively to restrict water flow in production wells and redistribution of water flows at injection facilities [13]. Similar compositions were proposed by Hungarian scientists [24, 25].

In order to expand the temperature range of practical use of reversing emulsions, we consider it expedient to conduct studies with hydrocarbon compositions AF_9-6, additionally including oil-soluble emulsifiers IE. Obtaining of such IE does not require the use of special dispersants, water of any degree of mineralisation can be used as an aqueous phase, its transition to a pasty state occurs at introduction of $\geq$ 0.5 volume of aqueous phase, and intensive viscosity gain at introduction of only 0.1 volume of the initial volume of hydrocarbon composition.

Table 2

Water activity in saturated salt solutions at 25 °C

Salt	*C*, mass %	a_w	Salt	*C*, mass %	a_w
HCl	41.4	0.111	$MgCl_2·6H_2O$	35.7	0.330
NH_4NO_3	68.2	0.611	$MgSO_4·6H_2O$	30.7	0.870
NH_4Cl	28.3	0.771	$CaCl_2·6H_2O$	45.1	0.287
NaCl	26.5	0.753	$Ca(NO_3)_2·4H_2O$	58.0	0.491
$NaNO_3$	47.8	0.738	$CaBr_2·6H_2O$	60.2	0.146
Formate Na	47.4	0.620	$ZnCl_2·3H_2O$	80.3	0.030
NaBr	48.6	0.577	$ZnBr_2·2H_2O$	82.5	0.072
KCl	26.5	0.843	$AlCl_3·6H_2O$	31.6	0.400
K_2CO_3	53.0	0.428	$Al(NO_3)_3$	40.2	0.602
KNO_3	28.0	0.924	Glycerol	100	0.180
Formate K	76.8	0.280	–	–	–

Table 3

The concentration of salts at which a_w = 0,5 at 25 °C

Salt	*C*, mass %	Salt	*C*, mass %	Salt	*C*, mass %
`NaOH	28.2	$MgBr_2$	43.8	$ZnBr_2$	63.9
KOH	33.8	$CaCl_2$	35.6	$AlCl_3$	28.2
Formate K	58.0	$CaBr_2$	47.1	Glycerol	80.0
$MgCl_2$	30.3	$ZnCl_2$	53.2	–	–

Regulation of IE density up to values ~ 1700 kg/m^3 is carried out by using aqueous solutions NaCl, KCl, $MgCl_2$, $CaCl_2$, $Ca(NO_3)_2$, $ZnCl_2$, $CaBr_2$, $ZnBr_2$, formates of sodium, potassium and their combination at the volume content up to ≈ 70 %.

In [2] the properties of IE with density up to 1310 kg/m^3 using $Ca(NO_3)_2$ solutions alkalinised to pH = 8-9, with density up to 1367 kg/m^3 using $Ca(NO_3)_2$ solutions alkalinised to pH = 8–9, with density up to 1367 kg/m^3 using solutions of $Ca(NO_3)_2$ mixtures alkalinised to pH ≈ 11 solutions of $CaCl_2$ + $CaBr_2$ mixture and density up to 1545 kg/m^3 using alkalinised up to pH = 5.6 of $CaBr_2$ + $ZnCl_2$ mixture at volume content of water phase 60 % on the basis of diesel fuel and oil of the Sutorminskoye field. IE with $Ca(NO_3)_2$ and $CaCl_2$ + $CaBr_2$ are heat – stable up to ~ 150 °C, while with $CaCl_2$ + $ZnCl_2$ – up to 100 °C.

For thermal stabilisation of IE, increase of structural and rheological properties and decrease of hydrocarbon medium filtration, additional latex input up to 5 vol. % and combination of latex with bentonite clay (up to 10 kg/m^3) are recommended [2, 26].

For secondary penetration of productive intervals of horizontal wells in the Norwegian sector of the North Sea were used IE of 1650 kg/m^3 density with internal phase of caesium formate solution of 2200 kg/m^3 density and IE of 1350 kg/m^3 density with $CaBr_2$ solution of 1650 kg/m^3 density, stabilised with 3.5 vol. % emulsifier, 2 kg/m^3 organobentonite, 5 kg/m^3 silicate flour and 30–90 kg/m^3 chalk with thermostability at 115 °C for 7 weeks [27]. Bench experiments on high-permeability cores Berea k = 0.5–0.8 μm^2 established 100% recovery of their permeability for oil after preliminary filtration of IE with density of 1702 kg/m^3 using $CaBr_2$ + $ZnBr_2$ solution with density of 2301 kg/m^3 and 56 % recovery of permeability – in low-permeability cores 1702 kg/m^3 using $CaBr_2$ + $ZnBr_2$ solution with density 2301 kg/m^3 and 56% permeability recovery – in low-permeable cores k = 0.06–0.08 μm^2 [28]. Additional injection of 114 kg/m^3 chalk in IE at 0.1 pore volume (PV) filtration reduced core permeability from 0.08 to 0.02 μm^2 with recovery to 0.05 μm^2 after reverse filtration 0,4 PV of diesel fuel, and 0.4 PV of 15 % HCl – up to 0.08 μm^2.

At filtration of IE in the reservoir space, absorption of the water phase from clayey reservoirs even with residual water saturation occurs through adsorption-solvation layer of emulsions with the internal phase represented by solutions of electrolytes and polyatomic alcohols [2, 29–33]. The driving force of this process is the osmotic pressure (P_{osm}), directed for the water flow from clay minerals into IE globules, proportional to the decrease of water activity (a_w) in them compared to its activity in minerals. The value of P_{osm} is determined by the formula:

$$P_{osm} = -\frac{RT}{v_w}\ln a_w, \text{ MPa}$$

where R – universal gas constant, dm^3·MPa/mole·K; T – absolute temperature, K; v_w – partial molar volume of water, dm^3/mole.

Values of a_w in low-permeability shale reservoirs $k = 0.34 \cdot 10^{-3}$ μm^2 are 0.78, and at $k = 0.026·10^{-3}$ μm^2 – 0.89 [32]. For illite shale, a_w ≈ 0.7–0.75. The method for assessing the stability of samples of water-sensitive clays in IE is described in [31].

Table 1. 2 and 3 show data on the activity of water in solutions of common salts and glycerol [2].

This problem of violation of the integrity of productive clayed reservoirs and a sharp decrease in their permeability in contact with water systems is characteristic of the J0 formation of the Bazhenov reservoir, containing ~ 2 % of residual water, with natural fracturing, which requires their killing with hydrocarbon compounds with minimal repression [34, 35].

Development of the Achimov formation of a number of fields in Western Siberia is associated with intensive water inflows due to under-saturation of oil and excessive residual water saturation, propagation of fracturing cracks into water-saturated intervals and formation of autofracturing cracks from injection to production wells [36, 37].

Effective development of promising gas deposits in low-permeable, clayed and water-saturated Turonian sediments of Western Siberia, occurring at depths of 950–1100 m, is possible with the use of hydrocarbon-based or alcohol-containing compositions at all stages of well construction and operation [38, 39].

Complete levelling of the water saturation of the BHZ when using DEs can be achieved by replacing their aqueous phase with polyatomic alcohols [28]. The problem that deserves close attention is the accurate determination of IE deposition rate in downhole fluids. The empirical results from [6, 40] correlate satisfactorily with each other and the calculated formula from [1]. At the same time, at deposition of IE through the oil medium filling the wellbore, their partial mixing with density loss will occur [1]. This fact also requires experimental verification.

Conclusion

Based on this review of the key and promising problems in the use of inverted emulsions for well killing, a list of important tasks for their improvement can be identified.

1. Bench tests of IE deposition rate along the wellbore depending on the difference in density, viscosity of phases and mixing zone with oil.
2. Preparation of IE by *in situ* method using natural oils or their compositions with hydrocarbons by preliminary saponification with alkaline agents and subsequent dispersion with solutions of salts of multivalent electrolytes.
3. Analysis of the behavior of spontaneous IE from a combination of ethoxylated surfactants ans oil soluble emulsifiers in hydrocarbon medium with sunsequent introduction of mineralized solutions.
4. Preparation and study of IE with polyatomic alcohols as an internal phase with subsequent evaluation of stability of intensively hydrated clays in their environment and recovery coefficient of permeability of water-saturated clay cores for oil after their contact with such IE.
5. Preparation and study of high-density IE using $CaBr_2$, $ZnCl_2$, $ZnBr_2$ salts and their mixtures for killing wells with water-sensitive reservoirs, AHFP and elevated reservoir temperature.
6. Bench studies on cores of the efficiency of well killing technology by preliminary plugging of BHZ with hydrocarbon gels followed by placement of aqueous liquids of the required density throughout the wellbore.

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