Emulsion Acid Diversion Agents for Oil Wells Containing Bottom Water with High Temperature and High Salinity

Jijiang Ge,* Xiangyu Sun, Ranran Liu, Zongsheng Wang, and Lei Wang

ABSTRACT: Conventional acid diversion agents cannot tolerate the high temperature and salinity of acidizing water-producing oil wells that contain bottom water and heterogeneous layers. Therefore, a water/oil (w/o) emulsion was proposed as an acid diversion agent to promote acidification. The selected emulsifier, oleic acid imidazoline, is a switchable emulsifier. Because this emulsifier reacts with acids to transform amines into ammonium, the emulsion rapidly demulsifies, and the emulsion acid diversion agent can use the spent acid flowback to remove plugging. Evaluation of the emulsion properties indicated that a 10 wt % emulsifier generated a stable emulsion at oil/water ratios from 1:9 to 4:6 at 90 °C. Viscosity was higher at lower oil/water ratios, and the emulsion with an oil/water ratio of 4:6, which had a low viscosity, was injected into the formation. During injection, the emulsion continued to emulsify in high-permeability channels, which increased the viscosity until the water layer was blocked. During experiments, single-tube and dual-tube models were designed to evaluate the injectivity and plugging selectivity of this emulsion. The results showed that the resistance factors exceeded 14 in the high-permeability cores when the emulsion was injected. The higher permeability ratio in parallel cores allowed a larger emulsion volume to enter the high-permeability cores. In experiments using parallel cores, the block rate of the high-permeability cores exceeded 92%, and that of the low-permeability cores was less than 12%. Finally, this emulsion was injected into two groups of parallel carbonate cores for acidification diversion tests. The results indicated that the permeability of the acidified low-permeability core was basically the same as that of the high-permeability core plugged by the emulsion. The findings of this study improve the understanding of the feasibility and advantages of using emulsions as acid diversion agents for high-temperature and high-salinity oil wells containing bottom water.

1. INTRODUCTION

Acidizing is an important technique for enhancing oil well production and improving water injection. During this process, because of formation heterogeneity, acid fluid preferentially enters high-permeability layers,1–4 which decreases production and increases water injection. Some oil wells even experience a sharp increase in water cut after acidification, as shown by the red arrow in Figure 1. Adopting effective injection acid diverting techniques is key to a successful acidization to ensure the injection of acids into the target formations.3–8 Mechanical and chemical acid diversion techniques have been developed to improve the nonuniform distribution of acidification to perfect production processes. The acid diversion method involves transferring a portion of acid to a low-permeability region, where less of it is absorbed.9 Mechanical methods typically use layered isolation tools such as packers, ball sealers, and coiled tubing.10 For example, gas-filled packers or conventional straddle packers are used to accurately control the acid location; however, mechanical methods are very cumbersome and expensive.11,12 To improve the acid diversion effect, chemical diverting agents have been developed, which change the distribution of acids and cause low damage to the formation.

The first diverting agents were wax beads and halite, where the former is made of paraffin wax, high-pressure polyethylene, resin, barite, and other ingredients, and the latter is a mixture of sodium chloride and calcium carbonate particles. Although their filter cakes divert acid in formations, after acid injection, the filter cakes do not completely dissolve in the formation fluid, which damages the formation. At present, reagents such as benzoic acid, OSRs (oil-soluble resins), foams, viscoelastic surfactants, relative permeability modifiers (RPMs), and degradable fibers are used to divert acid. Benzoic acid and OSRs first form a filter cake in a formation, which plugs highly permeable layers or cracks, forcing acid into low-permeability layers. When a production well is opened, such filter cakes can be slowly dissolved by water or oil, causing them to lose their plugging effect. The disadvantage of benzoic acid and OSRs is that these...
particles are too rigid to form dense filter cakes, and they display only a limited plugging effect. For this reason, a lot of research studies have been performed to improve their performance.13

Since the 1990s, foams have been widely used as diverting agents in gravel-packed wells in formations with permeabilities lower than 1.5 μm².14,15 In field applications, nitrogen compressors must be used, and the foaming property is easily affected by acidizing additives.16,17 Most chemical methods use surfactants to generate a gel as the blocking agent underground to complete acid diversion,18,19 but gel-blocking agents are difficult to remove after solidification, causing permanent blockage of channels at high temperatures.20–22

Few studies have investigated emulsions as acid diversion agents under high-temperature and high-salinity formation conditions; however, emulsions have been extensively studied and applied as agents for other functions in the petroleum industry.23 Pei et al. studied emulsions to improve heavy oil recovery under high temperature and high salt conditions, indicating that emulsions have good plugging performance and can be further emulsified with formation water or crude oil under high-temperature formation conditions.24,25 Hajiabadi et al. and Shirazi et al. studied the influence of emulsion drilling fluid and acid emulsion on formations, respectively, and found that emulsions containing only surfactants had low formation damage; however, the emulsion-containing acid damaged the formation, suggesting that an emulsifier should not form an emulsion with acids.26,27 Because of their easy implementation at well sites, low formation damage, and their powerful temporary blocking function, emulsions have been used to shut off the water layer before injecting acid.

The difference between an emulsion as an acid diversion agent and other functional agents is that, first, an emulsion must be able to divert acids while not being further emulsified with acid; second, an emulsion must be temporary, that is, it must be broken after acidification to remove the plugging function. Demulsification in formations is generally divided into two types. One is that emulsion droplets coalesce and spontaneously demulsify, which is in contradiction with the need to inhibit the agglomeration of emulsion droplets to maintain the plugging performance before the spent acid flowback. This demulsification method is difficult to control. The second is to make the emulsion contact other special function injections to greatly increase the oil/water interfacial tension and break the emulsion. This can be done by the spent acid flowback, which only requires the injection of spacer fluid between an injected emulsion and acid liquor. The advantage of this method is that there is no need to control the demulsification time, and the spent acid flowback can spontaneously initiate demulsification, as shown by the

Figure 1. Acidification flow chart and acidification effect using the emulsion acid diversion agent.
green arrow in Figure 1. Therefore, it is necessary to select an emulsifier that can react with acid and greatly reduce the emulsification performance after the reaction.

The disadvantage of this method is that the subsequent spontaneous emulsification after the emulsion is injected into the formation is time-consuming. Therefore, it is necessary to increase the injection speed accordingly to shorten the subsequent emulsification time. In addition, high concentrations and large amounts of emulsifier are required, and so, it is important to choose an economical emulsifier. The limitation of this method is that when the formation temperature exceeds 120 °C, the emulsion can only remain stable for a very short time, making it necessary to incorporate additives into the emulsion acid diversion agent to enhance the high-temperature stability. The subsequent process of using the waste acid for demulsification during the spent acid flowback may also have to be adjusted accordingly.

Oleic acid imidazoline, a switchable surfactant, which can reversibly transform from surface-active forms to surface-inactive forms in response to the corresponding stimuli, was selected as an emulsifier in this study, and the rheological properties, injectivity, and selective plugging ability of this emulsion were evaluated. First, the experimental materials and procedure were presented. Then, the results of each experiment were summarized and analyzed. The conclusions such as the basis for why this emulsifier was selected, the characteristics of the injected emulsion, and the acid diversion effect of the emulsion were obtained.
2. EXPERIMENTAL SECTION

2.1. Instruments and Reagents. A Brookfield viscometer was used to measure the viscosity of emulsions. A PHS-DB200U biological microscope (Phoenix Optical Group Co., Ltd.) was used to observe and measure the size of emulsions. A YXZB—25/300 mm synthetic core device (Hai An Oil Scientific Instruments Co., Ltd.) and a physical simulation device (including a constant-flux pump, injection pump, core holder, pressure containers, oven, liquid, and pressure acquisition device) were used to evaluate the injectivity and selective plugging ability of the emulsions in this study.

Hydrochloric acid (analytical grade), NaCl (analytical grade), oleic acid imidazoline (industrial grade), and 0° diesel oil were used as reagents. Oleic acid imidazoline is a brown-red liquid, with a density of 0.892 g/cm³ and a boiling point of 92.8 °C at 760 mmHg. The density of diesel oil at 20 °C is 0.85 g/cm³. The water used in the experiment was compounded, according to Table 1.

2.2. Experimental Method. 2.2.1. Evaluation of Emulsions. Active diesel with 10% emulsifier (by mass) was prepared, and then, saline water was mixed into it according to oil/water ratios of 1:9, 2:8, 3:7, and 4:6. The mixtures were oscillated at 1000 rpm/min for 5 min to form stable emulsions, whose viscosities were measured by a Brookfield viscometer under different shearing rates at 90 °C. The droplet size distribution diagram was obtained using an optical microscope with the software of ImageJ.

2.2.2. Evaluation of Injectivity and Selective Plugging Ability of the Emulsion. The artificial cores used for coreflood tests were 2.5 cm in diameter and 10 cm in length. The core was hydrophilic, with a porosity of about 16–22%. We used artificial cores with gradually increasing the permeability (0.084, 0.100, and 0.266 μm²) to evaluate the selective injectivity and the plugging performance of the emulsion. After the cores were saturated with high-salinity water, the permeability of each core was measured. Formation water with a constant-flux pump and w/o type emulsion with an injection pump were sequentially injected into the cores at rates of 0.3 and 0.2 mL/min. The pressure changes and outflow rate of the emulsion were recorded to examine the plugging ability of this w/o type emulsion.

2.2.3. Evaluation of Selective Injectivity and Diversion Ability of the Emulsion. Four groups of parallel artificial cores with different permeability ratios (3.7, 7.6, 30.2, and 42.1) were used to evaluate the selective injectivity and diversion ability: (1) The cores were first saturated with formation water, then the permeability of each core was measured, and the latter two cores were assembled in parallel. (2) The emulsion was injected into each group of parallel cores at a rate of 0.2 mL/min at 90 °C, and the injection pressure of each group of two cores and the volume of liquid discharged from the outlet of each core were observed to examine its selective injectivity and plugging ability.

2.2.4. Physical Simulation Experiment of Temporary Plugging and Acidizing. Parallel natural cores were used to evaluate the plugging abilities of the emulsion and its acidizing effect. (1) The cores were first saturated with formation water, and then, the permeability of each core was measured. The latter two cores were assembled in parallel, and an emulsion with a slug of about 0.3 PV was injected at a rate of 0.2 mL/min. (2) The emulsion was pushed by formation water, (3) Acid was injected after formation water, and (4) after acidizing, the permeability of each core was measured again. The volume of the emulsion injected was controlled during the experiment, and the injection was usually stopped immediately when the fluid began to flow out of the low-permeability core, that is, the emulsion began to enter the low-permeability core. The emulsion was made up of active diesel (diesel contains 10 wt % emulsifier) and saline solution in a 4:6 ratio. The flooding rates of the emulsion and hydrochloric acid were both 0.2 mL/min, and all experiments were done at 90 °C.

The experimental flow chart for each experiment is shown in Figure 2.

3. RESULTS AND DISCUSSION

3.1. Screening and Evaluation of Emulsifiers. Emulsifiers must have high-temperature resistance, form stable w/o emulsions within a wide salinity range at high temperatures, and must not emulsify acid.

Researchers have previously compared a series of emulsifiers such as dodecylbenzene sulfonic acid (long-chain alkyl benzene sulfonic acid), oleic acid, and oleic acid imidazoline and found that oleate imidazoline forms a stable emulsion over a wide range of oil/water ratios. The emulsion formed is a w/o type with a high viscosity that can reach a maximum of 3800 mPa s at 90 °C. The formed emulsion is stable for at least 7 days (stable for 30
days at most) at a 90 °C formation temperature, and so, oleic acid imidazoline has an ideal emulsification effect on diesel oil. This surfactant has a long lipophilic alkyl chain and a cyclic hydrophilic group, in which amine is the main group. The evaluation showed that oleic acid imidazoline improved the emulsion stability of saline solution and diesel, and the emulsion viscosity increased with increasing water salinity and decreasing oil/water ratios, as shown in Figure 3. The emulsion with an oil/water ratio of 4:6 was used as the injectant. The viscosity of this emulsion was not high, and it was easy to inject. In addition, there is a high concentration of emulsifier in the active diesel between w/o emulsion droplets, making it easily emulsified. During injection, further emulsification can occur when the emulsion encounters water in the wellbore and formation, which increases the viscosity and produces a good plugging effect.

At the same time, oleic acid imidazoline, which is a switchable surfactant, reacts with the acid, transforming amines into ammonium after acidization. This increases the hydrophilicity of

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Table 2. Parameters of Parallel Cores and Injection Volume of the Plugging Agent

| no. | permeability, $\times 10^{-3} \mu m^2$ | pore volume, mL | injection volume of the plugging agent, PV |
|-----|---------------------------------|-----------------|-------------------------------------------|
|     | high-permeability core | low-permeability core | permeability ratio | high-permeability core | low-permeability core | |
| 1   | 100.5                   | 27.4            | 3.7                  | 9.1                     | 8.6                    | 0.066                        | 0.023                       |
| 2   | 227.8                   | 30.0            | 7.6                  | 12.0                    | 11.8                   | 0.071                        | 0.038                       |
| 3   | 814.5                   | 27.0            | 30.2                 | 11.4                    | 11.8                   | 0.21                         | 0.015                       |
| 4   | 400.8                   | 9.5             | 42.1                 | 10.2                    | 11.9                   | 0.55                         | 0.034                       |
the surfactant and changes its hydrophilic–lipophilic balance. Therefore, the emulsion can be quickly destroyed in the presence of an acid, which prevents blockage of the spent acid flowback, as shown in Figure 4.

Given the characteristics of oleic acid imidazoline, water or diesel fuel slug should be injected as a spacer fluid after injecting an emulsion at on-site construction. This prevents the failure of the temporary plugging for high-permeability layers because of demulsification when the emulsion encounters acid.

3.2. Evaluation of Injectivity and Plugging Ability for the w/o Type Emulsion. As shown in Figure 5, the injection pressures of formation water and emulsion show that the resistance factors of the emulsion in the cores with permeabilities of 0.084, 0.100, and 0.266 μm² were 14, 16, and 17, respectively. It demonstrates that this w/o emulsion has a good plugging effect on these cores. The injection pressure curve trends of the three groups of core experiments were basically the same, and so, the core with the highest permeability was selected to evaluate the emulsion’s ability to plug a high-permeability formation. The pressure after emulsion injection was significantly higher than that after water injection. A dilute emulsion was produced 30 min later, and after 50 min, the average emulsion production rate approached 0.2 mL/min (the injection rate), and a thick emulsion began to appear (Figure 5c). This indicates that the emulsion continued to emulsify with the formation water in the core, which greatly enhanced the plugging effect.

Microscope analysis indicated that the droplet size distribution of the prepared emulsion was less than 12 μm, but it had a primary diameter of 2–6 μm, as shown in Figure 6. This again verifies that the emulsion continued to emulsify with the formation water in the core, and so, the droplet size of the prepared emulsion flowing from the core was larger than that of the initial emulsion, which is one of the reasons for its enhanced plugging ability. For a core with a permeability of 0.266 μm², its average pore throat radius was less than 5 μm, and so, it seems that part of the emulsion deformed through the core. This phenomenon increased the Jamin effect, which further enhanced its plugging ability.

3.3. Evaluation of Selective Injectivity and Diversion Ability of the Emulsion in Parallel Cores. Table 2 shows that upon increasing the permeability ratio in parallel cores, the volume of the emulsion that entered high-permeability cores gradually increased, while the quantity that entered low-permeability cores decreased (<0.04 PV). This illustrates that this emulsion has a good injection selectivity.

Changes in the permeability and fractional flow rates of parallel cores before and after plugging are shown in Table 3. Because of the selective plugging by the emulsion, the permeability of high-permeability cores decreased by more than 92%, and for the low-permeability cores, it decreased by less than 15%. After plugging, the flowrate of formation water through the high-permeability core was reduced to about 1/6 of its original flow, and the flow through the low-permeability core increased by about 5.4 times the original flow. The low-permeability cores served as the main fluid flow zones.

3.4. Physical Simulation of Temporary Plugging and Acidization. Two sets of natural carbonate parallel cores were built to evaluate the acidizing effect after emulsion injection. The measured basic parameters of the cores are shown in Table 4.

The pressure gradually increased when the emulsion was injected, indicating that oil and water continuously emulsified in the core, which hindered subsequent injection, as shown in
The acid was diverted into the core, after the emulsion was injected and plugging was completed,

- Table 5. Parameters before and after Acidizing

| no. | cores        | parameters before acidizing | permeability, $\times 10^{-3} \mu$m$^2$ | parameters after acidizing | permeability, $\times 10^{-3} \mu$m$^2$ | permeability change |
|-----|--------------|----------------------------|----------------------------------------|----------------------------|----------------------------------------|-------------------|
| 1   | high permeability | 0.024 | 15.7 | 0.095 | 4.0 | drop 74.5% |
|     | low permeability   | 0.135 | 3.4 | 0.105 | 4.2 | increase 23.5% |
| 2   | high permeability | 0.025 | 14.6 | 0.07 | 4.8 | drop 67.1% |
|     | low permeability   | 0.11 | 5.5 | 0.03 | 46.8$^6$ | increase 50% |

Because of excessive acidization, the cores’ exit end became loose and some even collapsed when the cores were taken out.

Figure 7. Pressure curves of injecting emulsion and hydrochloric acid in the inverse direction of cores.
emulsion achieved good plugging performance in high-permeability cores. The emulsion was easily demulsified in the presence of an acid, and so, it did not affect the flowback of the injected acid.

because of The parallel core injection and acidizing experiments demonstrated that hydrochloric acid solution was diverted into low-permeability cores after injecting the w/o emulsion. This occurred the emulsion’s selective plugging effect for high-permeability cores, which greatly improved the permeability of low-permeability cores.

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Notes
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