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Research and Application of an Environmental-Friendly Low-Damage and High Salt-Resistance Slick Water Fracturing Fluid System

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Abstract

The Sulige gas field is a typical low-pressure low-permeability tight sandstone gas reservoir. The reservoir has poor seepage capacity, strong heterogeneity, high mineralization of formation water and extremely scarce water resources on the site. These unfavorable factors have brought great difficulties to the on-site mining process. Now, a nano-composite green environmental protection slick water fracturing fluid system CQFR can be quickly dissolved because of the larger specific surface area, and the small molecular size makes the damage to the reservoir less than 5%, and the average drag reduction effect can reach more than 73%. It can quickly and well dissolve and maintain performance under high salinity conditions and fracturing flowback fluids. It responds well to the complex reservoir conditions on the construction site and makes the flowback fluid recyable, which greatly reduces the consumption of water resources on the construction site and effectively improves the construction efficiency and economic benefits.

Keywords

Tight Sandstone, Recyclable Backwater, Green Environmentally Friendly, Low-Damage, High Salt-Resistance, Slick Water

1. Introduction

Tight sandstone gas is an unconventional natural gas resource stored in low-permeability or ultra-low-permeability tight sandstone reservoirs. It is generally found in low-permeability gas-bearing formations in various basins around the world. Current research indicates that China's tight sandstone gas reservoirs are widely distributed and have great development potential [1] [2] [3]. Therefore, accelerating the research, development and utilization of tight sandstone gas resources is of great strategic significance for China's energy security.

The main working area of the tight sandstone gas reservoir exploitation in Changqing Oilfield is in the Sulige area, which exploration area is about 2×10^4 km^2. It is located in the northwest of the Yishan slope in the Ordos Basin. It is a typical Upper Paleozoic low-permeability sandstone gas field. The main gas-producing strata are the 8^{th} section of the Lower Shihezi Formation and the 1st section of the Shanxi Formation. The low permeability is developed on the westward slope of the single slope and is the most dense area of the Ordos Basin. The porosity is 3%. ~15%, the peak value is 7%, and the permeability is mainly distributed in $(0.025\sim15.6) \times 10^{-3}$ μm^2, and the peak value is 0.63×10^{-3} μm^2. In general, the reservoirs in the Sulig area of Changqing Oilfield are low-pressure, low-permeability, low-abundance, small pore space in the reservoir, poor seepage capacity, strong heterogeneity, no priming, and need to be modified to obtain capacity. Therefore, after completion of drilling and completion operations, fracturing and other transformation operations must be carried out in order to carry out the mining. Traditional drag reducers usually require a longer dissolution time, which takes more time for on-site construction. The high salinity of the formation water in the Sulige area will make the structure of the drag reducer molecules damaged and unable to function, and the modified drag reducer molecules will agglomerate and form precipitates, blocking the seepage channel and causing reservoir damage. Therefore, the effect of fracturing construction in early mining is not ideal [4]-[9]. To solve these problems, the slick water system must have the characteristics of small reservoir damage, low biotoxicity, salt-resistant properties and it is better to be self-cleaning so that fracturing fluids and reservoir production water can be recycled for subsequent fracturing applications [10]-[14].

According to the reservoir geological characteristics and formation water characteristics of tight sandstone gas reservoirs [15] [16] [17], the slick water fracturing fluid system CQFR was developed, and its key auxiliary agent is the drag reducing agent CQFR. It is a nano-composite polymer in structure. Through this technology, a larger specific surface area can be obtained to dissolve more quickly and stably in a complex water environment; the increase in molecular weight can obtain a longer chain structure that can pass through the chain. The expansion and contraction of the structure can store and release more energy to achieve the purpose of reducing friction consumption and friction coefficient; smaller molecular size can make CQFR play a better reservoir protection effect than other drag reducers of the same type. It enhances the protection of functional groups so that it can maintain performance in high salinity formation water that is sufficient to destroy the molecular structure of ordinary drag reducers, and can also be used normally in fracturing flowback fluids. For the low viscosity of the CQFR slick water fracturing fluid system, the injection method of large liquid volume, large displacement and low sand ratio is used in the field construction to solve the problem of slightly weak sand carrying capacity.

2. Indoor Experiment

2.1. Experimental Instruments and Materials

1) Experimental equipment: JHJZ-I high temperature and high pressure dynamic drag reduction evaluation system drag reduction rate tester, electronic scale, DXY-3 biological toxicity tester, core flooding device.

2) Materials: CQFR green clean slick water, CQFR drag reducer single agent, multi-functional treatment agent, simulated formation water, fracturing backflow liquid, sodium chloride, calcium chloride, ferric chloride, oil-based drag reducer, powder drag reducer, on-site core.

Among them, the main ion composition of simulated formation water and fracturing backflow liquid is shown in Table 1 and Table 2.

The core used for indoor evaluation of core damage is the core of a gas field in Changqing, which is a brown-gray gas-bearing medium sandstone. The main parameters of the core are shown in Table 3.

2.2. Experimental Methods

2.2.1. Compatibility Experiment

The slick water fracturing fluid contains various chemical additives, such as drag reducers, drainage aids, anti-swelling agents, etc., and whether these additives can exert their respective effects without being affected by each other is the key point of the performance of the slick water fracturing fluid system [18]. Considering the water type and high salinity of the formation water in the Sulige area, it is necessary to examine the compatibility of the other additives and the drag

Table 1. Main ion composition of simulated formation water.

Ion type	Ca^{2+}	Mg^{2+}	Na^{+}	K^{+}	Cl^{-}	SO_4^{2-}	HCO^{3-}
Ion content mg/L	410	1310	10,900	390	19,700	2740	152

Table 2. Main ion composition of fracturing and returning liquid.

Ion type	Ca^{2+}	Mg^{2+}	$Na^{+} + K^{+}$	Cl^{-}	SO_4^{2-}	HCO^{3-}
Ion content mg/L	161.45	13.31	8044.01	12,435.34	27.84	792.83

Table 3. Main parameters of core.

Well number	Horizon	Coring well section m	Density g/cm^3	Apparent porosity %	Permeability 10^{-3} μm^2
S_{83}	S_{23}	2439.75 - 2439.88	2.58	7.9	0.479

reducer to avoid the degradation of the performance of the slick water fracturing fluid due to the poor compatibility of the additives.

Deionized water, tap water, simulated formation water, and fracturing fluid were used as solvents to investigate the compatibility of the slip water fracturing fluid system composed of CQFR and multifunctional treatment agent. The mixed solution of the formula: 0.1% CQFR + 0.2% multifunctional treatment agent is added to the above liquids separately, and the solution solubility is observed after being left to stir.

2.2.2. Salt Resistance Test

Based on the high salinity of the formation water in the Sulige gas field, in order to make the performance of the fluid entering the well unaffected, and the return liquid can be reused to save water resources, the CQFR slick water system must have good salt and calcium resistance. To test the salt resistance of CQFR, two brines were prepared: 1) 3% NaCl + 2% $CaCl_2$; 2) 10% NaCl + 10% $CaCl_2$. Equal amounts of the same concentration of CQFR drag reducer, oil-based emulsion drag reducer and powder drag reducer were added to the two brines to observe their dissolution. In addition: 3) 0.01% $FeCl_3$ solution; 4) 10% $CaCl_2$ + 0.01% $FeCl_3$ solution, adding the same amount of CQFR drag reducing agent to the two brines respectively, observe the dissolution.

2.2.3. Resistance Reduction Performance Test

Refer to China's oil and gas industry standard SY/T5107-2005 "Water-based fracturing fluid performance evaluation method", select the instrument JHJZ-I high temperature and high pressure dynamic drag reduction evaluation system drag reduction rate tester to test the drag reduction rate in 3% chlorination The reduction rate of 0.1% CQFR slick water fracturing fluid was tested in sodium + 2% calcium chloride solution, and the drag reduction effect of the same concentration of powder drag reducer and oil-based drag reducer was compared. The test line is 10 mm pipe diameter, the pipe length is 2 m, and the test flow rate is 30 L/min [19].

2.2.4. Biological Toxicity Test

In recent years, environmental protection issues have become increasingly prominent, and most of China's oil and gas fields are in remote areas where water resources are scarce. For the exploration and development of oil and gas resources, the amount of water used is extremely large. In the process of oil and gas development, various chemical agents will enter the reservoir with water, and there is the possibility of polluting formation water and farmland irrigation water. Therefore, it is necessary to consider whether the chemical agents in the well fluid are toxic, that is, they need to be analyzed for biological toxicity [20].

Refer to the China National Petroleum and Natural Gas Industry Standard SY/T 6788-2010 "Water-soluble oilfield chemical agent environmental protection technology evaluation method", using the DXY-3 biological toxicity tester, using the luminescent bacteria method to calculate the biological toxicity index

EC_{50} (half maximum effect concentration) evaluation slippery The biological toxicity of the water drag reducer system. It mainly tested the biological toxicity of clean water, common oil-based drag reducer, ordinary powder drag reducer, CQFR drag reducer and slick water system.

2.2.5. Permeability Damage Test

In the process of using fracturing fluid to reform the reservoir, the fracturing fluid as the working fluid will directly contact the reservoir and cannot be completely returned after the reconstruction, so when the fracturing fluid stays in the reservoir, it will caused damage of the reservoir, which by causes the migration or expansion of the clay mineral, eventually blocking the pore throat reducing the permeability of the reservoir, or making the pore radius inside the reservoir smaller, and the damage of the fracturing fluid to the reservoir will eventually lead to the production of oil and gas has fallen. Because the fracturing fluid flows through the reservoir, the core permeability will change，therefore, the degree of damage caused by the fracturing fluid to the reservoir can be understood by the intuitive change of the core permeability.

Referring to the Chinese oil and gas industry standard SY/T5336-2006 "Core Analysis Method", the core flooding device was used to test the influence of the slick water fracturing fluid on the permeability of the core matrix, and the core permeability of the slick water prepared by different water samples was tested.

2.3. Results and Analysis

2.3.1. Compatibility

The experimental results are shown in Figure 1 below:

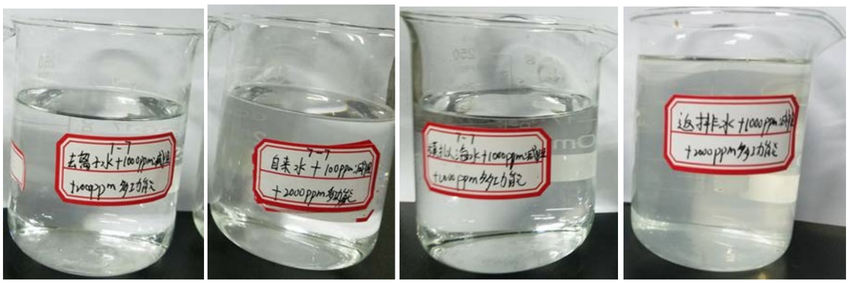

Figure 1. Deionized water, tap water, simulated formation water, and backflow fluid distribution.

It can be seen from Figure 1 that the slick water solution prepared by three kinds of water samples, such as deionized water, tap water, and simulated formation water, is colorless, clear and transparent with no obvious precipitation. The return liquid itself is slightly turbid and the slick water solution prepared by the splitting and returning liquid is in the same state as the original liquid of the returning liquid with no obvious precipitation in the solution. After the prepared slippery aqueous solution is left for 24 hours, it is found that the liquid is still in the original state, and there is no obvious precipitation in the solution.

Compatibility experiments show that CQFR (0.1%) + multi-functional treatment agent (0.2%) slick water fracturing fluid formula will not produce precipitation when using deionized water, tap water, simulated formation water, and return fluid as compatibility is good.

2.3.2. Salt Resistance

Four kinds of salt solution: ① 3% NaCl + 2% $CaCl_2$; ② 10% NaCl + 10% $CaCl_2$; ③ 0.01% $FeCl_3$ solution; ④ 10% $CaCl_2$ + 0.01% $FeCl_3$ solution. The experimental results are shown below.

As can be seen from Figure 2 and Figure 3, the brine ① is clear and transparent. The CQFR drag reducer is completely dissolved in the brine ① and the brine ②, and no floc or precipitate appears; the oil-based emulsion drag reducer is basically un-dissolved in the brine ①, and in the brine ②, they were also basically undissolved and flocculent precipitates appeared, and the solution was turbid, with more undissolved drag reducing agents attached to the beaker wall; powder drag reducing agents in brine ① And brine ② completely dissolved, no floc or precipitation, but the dissolution time is over 5 minutes. In addition, the brine ④ stock solution is a turbid opaque liquid. As can be seen from Figure 4, CQFR has no precipitation, no layering, and no floc in the solutions of ③ and ④.

Figure 2. Dissolution of blank, CQFR drag reducer, oil-based emulsion drag reducer, powder drag reducer in ①.

Figure 3. Dissolution of blank, CQFR drag reducer, powder drag reducer, oil-based emulsion drag reducer in ②.

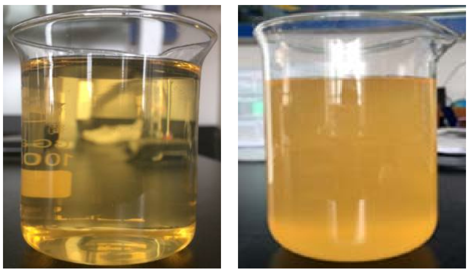

Figure 4. Dissolution of CQFR drag reducer in saline ③ and saline ④.

In summary, oil-based emulsion drag reducers have poor salt resistance in brines ① and ②; powder drag reducers have better salt resistance in brines ① and ②, but the dissolution time is longer than CQFR, which is not conducive to site construction. CQFR nanocomposite drag reducers have good salt resistance in brines ①, ②, ③, and ④. The cationic groups carried by drag reducer molecules and salt solutions repel each other, so that the drag reducing agent molecules are not affected by the salt ions, and the molecular chain is curled to form flocculation or precipitation. Because CQFR is a nanocomposite drag reducer, the specific surface area is large and can be efficiently and quickly dissolved. The dissolution time under laboratory conditions is within Both are less than two minutes, so the CQFR water skating system can be better applied to the site construction in the Sulige area where the formation water belongs to $CaCl_2$ water type and high salinity.

2.3.3. Resistance Reduction Performance

The experimental results are shown in Figure 5.

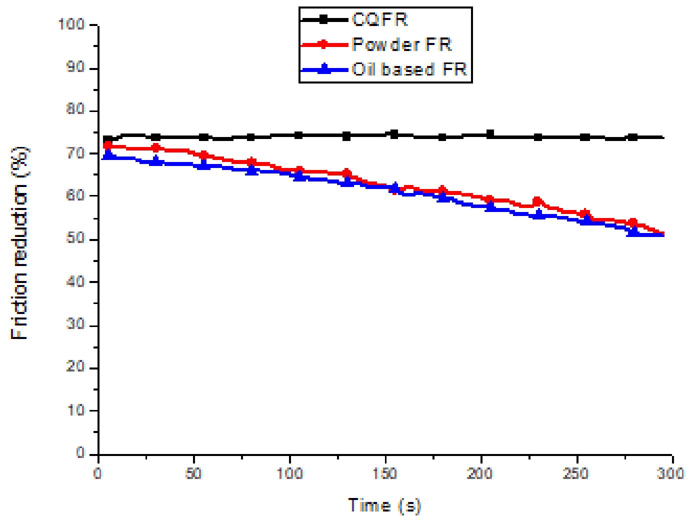

Figure 5. CQFR, powder drag reducer and oil-based drag reducer experiment.

From the experimental results, it can be seen that the CQFR nanocomposite drag reducer absorbs energy to a certain extent through the elasticity of the polymer molecular chain itself, thereby reducing friction between the fluid entering the well and the pipeline in high-speed flow, and due to the cations of the drag reducer molecule The role of the group can also maintain the polymer structure without deterioration in a high salt environment. It can also maintain good drag reduction performance in a 3% sodium chloride + 2% calcium chloride solution, and the average drag reduction rate can reach 73.4%. The maximum drag reduction rate can reach 76.4%. The results of drag reduction performance not only fully meet the standard when the drag reducer concentration is ≤0.1%, the drag reduction rate is ≥65% when using water to prepare slick water, and when the drag reduction performance is tested by backdrainage, the mineralization degree of the backdrain fluid is greater than 50,000 mg/L. The requirement of drag reduction rate ≥60% and the drag reduction effect is about 5% higher than other types of drag reducing agents. Observing the powder drag reducer and oil-based drag reducer again, it was found that the initial drag reduction effect of the powder drag and oil-based drag reducer in the 3% sodium chloride + 2% calcium chloride solution was not much different from the CQFR, but with the With the increase of time, the drag reduction rate has dropped significantly, indicating that their molecular structure has been damaged by salt ions and curled up. They cannot play the role of drag reduction normally, and cannot provide enough for several hours of fracturing construction in a high-salt calcium environment. The drag reduction effect is not conducive to the field operation of the Sulige gas field.

In addition, 0.05%, 0.08%, and 0.1% of CQFR were mixed with the return fluid, and the drag reduction rate after stabilization was recorded. The results are shown in Table 4 below and we can find that the CQFR prepared with on-site return drainage can also maintain a drag reduction rate of more than 65%, indicating that on-site return drainage can be used for subsequent construction to reduce water consumption.

Table 4. CQFR drag reduction performance experiment in reflux fluid.

Water sample	Addition of drag reducer/%	Drag reduction rate/%
backflow fluid	0.05	65.4
	0.08	67.6
	0.1	71.7

2.3.4. Biological Toxicity

The test results are shown in Table 5 below:

Table 5. Biological toxicity test.

drag reducer	EC_{50} ($mg \cdot L^{-1}$)	Toxicity level
Oil-based drag reducer	1129	Slight poison
Powder drag reducer	63.27	Moderate poison
Water	1.0×10^6	nonpoisonous
CQFR drag reducer	1.89×10^6	nonpoisonous
CQFR drag reducer system	1.86×10^6	nonpoisonous

Combined with Table 5, according to the definition in the standard that $EC_{50} > 25,000$ is non-toxic, it can be ground, and CQFR drag reducing agent substitutes are synthesized at least to meet the FDA GRAS (generally considered safe) safety standards, and have good green and non-toxic properties. The EC_{50} value of the drag reducing agent single agent is 1.89×10^6 $mg \cdot L^{-1}$, and the EC_{50} value of the slick water system formulated with the drag reducing agent as the core is 1.86×10^6 $mg \cdot L^{-1}$, which are both non-toxic and beneficial to protection of groundwater and the environment; corresponding foreign oil-based drag reducers and powder drag reducers are slightly toxic and severely toxic, respectively, which will cause environmental pollution or increase the corresponding cost of sewage purification. Therefore, in terms of environmental protection, the CQFR green clean slick water system should consider some foreign oil-based drag reducers and powder drag reducers.

2.3.5. Permeability Damage

The experimental results are shown in Table 6:

Table 6. CQFR recovery of gas phase permeability of Changqing core in different solutions.

Drag reducer	Original permeability mD	Post-injury penetration mD	Permeability recovery rate %
Water	0.479	0.473	98.7
backflow fluid	0.495	0.481	97.1

In the experiment, the displacement time using CQFR for displacement is about 20 minutes less than that with fresh water, which has a significant improvement in efficiency. As shown in Table 6, the core permeability recovery rate of CQFR drag reducer in fresh water and fracturing fluid is greater than 95%, and the core damage rate is less than 5%. This is because of the combination of the stabilization effect of clay does not easily cause the hydration and expansion of clay to block the cracks. At the same time, the drag-reducing effect of the drag reducing agent can effectively reduce the surface interfacial tension of the drag reducing agent and make it easier to return, and understand from compatibility experiments the CQFR drag reducing agent has good compatibility and will not produce floc to block the pore throat. In view of the characteristics of tight pore space, poor permeability and strong heterogeneity of reservoirs in tight sandstone gas reservoirs in the Sulige gas field, the use of the drag reducing agent in the field can effectively reduce the damage to the reservoir and protect it that is conducive to long-term exploitation.

In summary, the CQFR slick water drag reducer system has the following advantages:

No obvious damage to reservoir permeability (usually 10 - 15 mg/L residues are used in the crosslinked jelly fracturing fluid, and the content of drag reducing agent added in slick water is only 0.1%. The drag reducing efficiency is high and transparent. Residues); the materials in the raw materials meet the FDA GRAS safety standards, and only a small amount of additives is required for slick water construction, which greatly reduces the construction cost and has no pollution to the environment; recycled drainage is used for fracturing of subsequent wells. Nano-composite: fast-dissolving, efficient drag reduction, relatively simple construction organization and implementation, without prior preparation, online automatic addition, especially important for large-scale construction.

3. Site Construction

The CQFR nano-composite high salt-resistance recyclable drag reducer developed by the team has been applied to the development of tight sandstone gas reservoirs in the Sulige gas field in Changqing Oilfield. The team members also participated in field tests and provided corresponding technical services. According to relevant requirements, the field test situation will be discussed by taking Well 5 as an example.

3.1. Basic Parameters

Well 5 is located in Wushenzhao Gacha, Wushenzhao Town, Wushen County, Ordos City, Inner Mongolia Autonomous Region. The development area of the eastern area of the Sulige gas field is about 4483 km^2, which belongs to the desert-grassland transition zone. The surface is unconsolidated loose yellow sandy soil with low pressure strength, and the altitude is generally about 1250 to 1400 m. Fracturing fluid includes: active water formula (1.0% of KCl gas well clay stabilizer, 0.5% of TGF-1 gas well high efficiency drainage aid, total 60 m^3), slick water (CQFR-01 drag reducer 0.1%, total 187.6 m^3) Low viscosity (0.35% EM50 thickener, 0.5% TGF-1 gas well high efficiency drainage aid, 0.5% ZJ-2 additive, total amount 385 m^3), gel breaker (52 kg ammonium persulfate, 88 kg capsule breaker). Fracturing construction parameters are shown in Table 7 below:

Table 7. Table of water skiing construction parameters.

	stage	Liquid Types of	Fluid volume m^3	Displacement m^3/min	Sand ratio %	Proppant volume m^3	Split time min	Cumulative time min
Front fluid	1	slick water	60	3.5			17.1	17.1
	2	slick water	15	3.5	6.8	1.0	4.6	21.7
	3	slick water	55	3.5			15.7	31.4
Replacement fluid	10	slick water	9.3	3.5			2.7	109.0

3.2. Construction Effect Analysis

The construction effect of well 5 is shown in Figure 6 below:

As can be seen from the red oil pressure curve in the figure, the pressure continues to increase after starting the pumping. When the slick water is added, the pressure drop can be clearly observed and basically stable. After the injection of low viscosity liquid, the pressure of the oil pipe slightly increases and stabilizes until Construction is over.

In this field test, the total salinity of the water used for the preparation was 1521.21 mg/L.It was a calcium chloride water type and when it was used as the water for preparation, it had salt resistance to drag reducing agents. Certain requirements, that is, the ability to resist calcium and magnesium ions is strong. After pumping the CQFR drag reducer, the estimated drag reduction rate obtained on the site is about 50%. The analysis may be due to the fact that the original solution of the drag reducer was left for a long time during field construction, and the

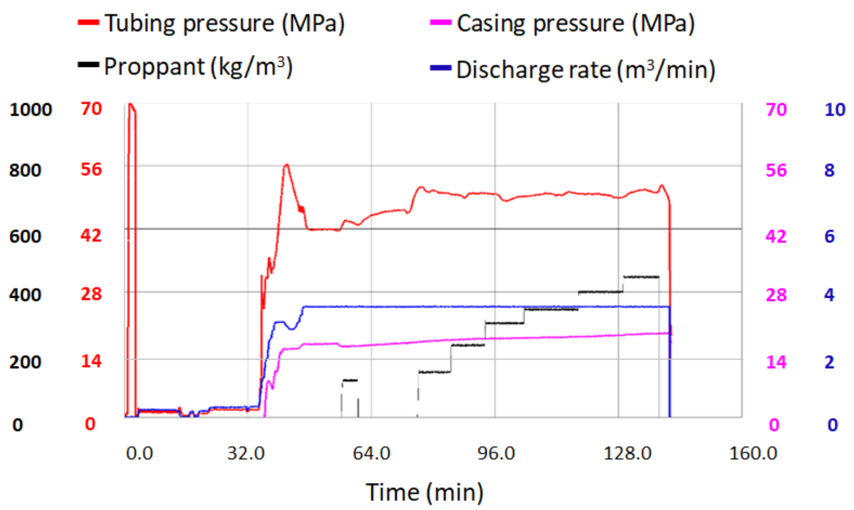

Figure 6. Fracturing construction curve of well 5.

solubility became poor, the on-site conditions are not as ideal as those in the laboratory, and it is not always possible to start a sufficient degree of dosing process. The performance is degraded, and when formulating the slick water, the liquid formulation method used cannot ensure that the drag reducer stock solution and water are mixed uniformly, so that the slick water performance is not good. After the construction standardized communication with the site staff, the construction was resumed, and the on-site drag reduction effect was significantly improved. It can be known from the results that after the CQFR drag reducer is pumped, the construction pressure and friction are significantly reduced, which strongly supports the successful progress of this fracturing construction.

4. Conclusions

1) The reservoirs in the Sulige area of Changqing Oilfield are of low pressure, low permeability, and low abundance. The reservoirs have small pore space, poor seepage capacity, and strong heterogeneity, and the long-term drought in the Sulige area of Changqing Oilfield Water and water resources are very valuable, and the formation water is $CaCl_2$ water type with high mineralization.

2) CQFR nano-composite drag reducer is green and non-toxic, and it is compatible with other additives in the system, and has less than 5% damage to the core, transparent and no residue.

3) The highly salt-resistant CQFR nano-composite drag reducer synthesized through a large number of laboratory experiments is suitable for the reconstruction and construction of tight sandstone reservoirs in the high salinity Sulige area. The drag reduction rate in salt water can reach 76.4%. The drag reduction rate in the return liquid can reach 71.7%, which can realize the recycling of the return liquid, effectively solve the problem of water shortage in the development process, and help save costs and increase efficiency.

4) The field test of CQFR nanocomposite drag reducing agent was carried out. The field test results show that CQFR can effectively reduce construction fric-

tion, improve construction efficiency, meet the construction requirements of on-site distribution and injection, and effectively reduce the field fracturing construction. Subsequent research needs to improve the stability under long-term storage of the drag reducer and further reduce its difficulty in dissolving in high-salinity water to cope with the possible inadequate stirring and excessive feed in the field construction.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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