

Optimization and Evaluation of Stabilizers for Tight Water-Sensitive Conglomerate Reservoirs

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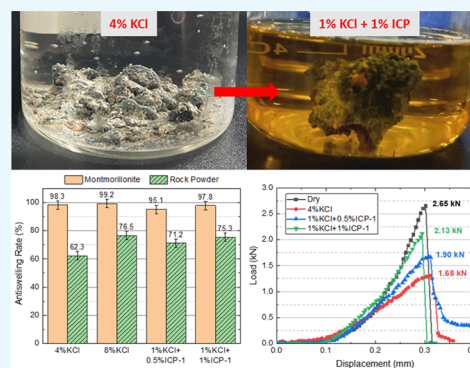
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ABSTRACT: The upper Wuerhe formation in the Mahu-1 play is a tight conglomerate reservoir that has characteristics of low porosity and low permeability. During the early stage of field development, it has been noticed that horizontal wells typically have a high flowback ratio and an extremely low oil production rate during the early production, and this is likely attributed to the water–rock interaction that causes the closure of generated hydraulic fractures. In this study, a stabilizer and its dosage in a fracturing fluid are optimized, and its effect on clay antiswelling and rock stabilization is evaluated. Experimental results indicate that a mixture of a salt and an inorganic cationic polymer can effectively inhibit the water–rock reaction by minimizing the clay swelling and compressing the electric double layer on the rock surface. The antiswelling rate of montmorillonite can reach 93.56%, and that of the reservoir rock powder can reach 75.32%. Meanwhile, Brazilian splitting tests are conducted to evaluate the mechanical property change of reservoir rocks before and after being submerged in fracturing fluids with different stabilizers. Compared to 4% KCl, which is currently used in the field, the new formula can enhance the breakdown pressure by more than 10% without increasing the cost. The findings of this work provide a solution for fracturing water-sensitive reservoirs and also establish a set of laboratory methods for optimizing stabilizers as fracturing fluid additives.



1. INTRODUCTION

Mahu tight oil reservoir is the largest conglomerate reservoir in the world, which has an estimated oil reserve of over 1.2 billion tons.¹ Along with the exploration and development since 2017, the current focus is shifted to the Wuerhe formation, especially in the Mahu-1 play, which is rich in water-sensitive minerals such as montmorillonite and laumontite.² To economically produce oil from such tight oil reservoirs, water is typically pumped to create the fracture network while proppants are pumped together to keep fractures open.^{3,4} However, a large amount of water injected during hydraulic fracturing can interact with the reservoir rock and result in formation damage that reduces the well productivity.^{5–7} During the early stage of Mahu-1 development, it has been noticed that horizontal wells typically have a large flowback ratio and an extremely low oil production rate, and 2% KCl in the fracturing fluid is not capable of preventing the interaction between the injected water and the water-sensitive minerals in the Wuerhe formation.

The formation damage mechanism of clay minerals due to water sensitivity has been studied for decades. Montmorillonite or an illite/montmorillonite mixture is the main cause of water sensitivity; since the mineral crystal has a laminated structure, water molecules can enter the interlaminal space and thus cause the swelling of the mineral crystal.^{8–10} Since the wavelength of X-rays is in a similar range to the spacings of

the lattice planes of crystals, X-ray diffraction (XRD) can be used to measure the volume expansion of water-sensitive minerals after interacting with water.^{11–13} From both XRD and laboratory swelling tests, it has been found that montmorillonite can expand 5–10 times after soaking in water. To prevent the swelling of montmorillonite, KCl or NH₄Cl is typically used. This is because potassium ions and ammonium ions have similar sizes to vacancy sizes in the interlaminal spaces of the montmorillonite crystal; once these cationic ions enter the interlaminal spaces, the electrostatic force can maintain the crystal structure and prevent water from invading.^{14–16}

Since each salt molecule has only one cationic ion, various organic antiswelling agents have been developed in past decades, which can dissociate multiple cationic ions from one molecule and thus reduce the dosage of agents in the drilling or fracturing fluid. They mainly include quaternary ammonium salts,¹⁷ polyamines,^{18,19} polyether amines,²⁰ and other long-

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Figure 1. Reservoir rock samples from the Wuerhe formation ((a) 10-cm-diameter cores, (b) 2.54-cm-diameter cores).

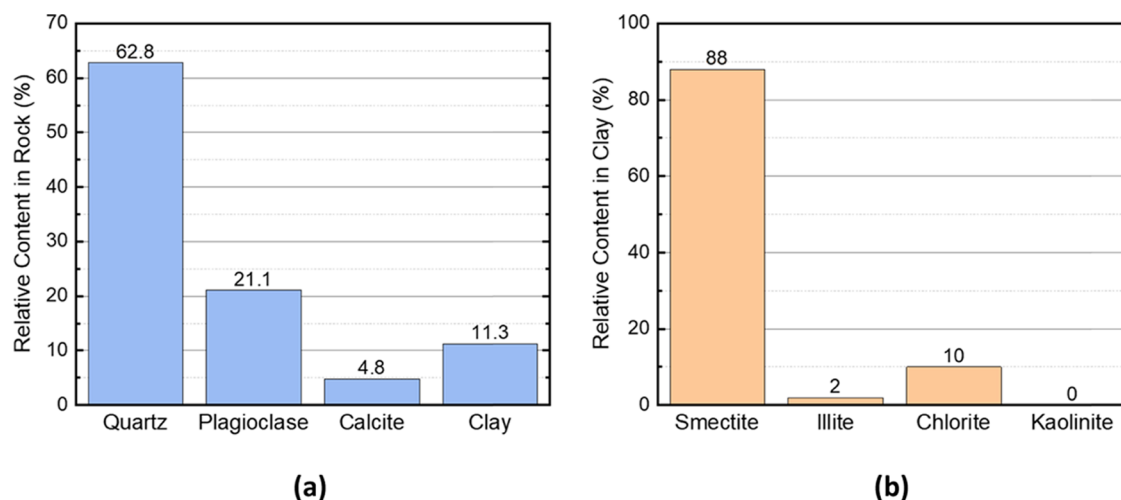


Figure 2. XRD results of reservoir rock samples ((a) mineral composition of the reservoir rock, (b) mineral composition of clay).

chain polymers.^{20,21} However, these polymers typically have linear structures and large molecular weights, and they can adhere to the surface of the created fractures that potentially inhibits the hydrocarbon flow from the rock matrix to the fractures. Studies have also shown that short-chain cationic polymers may also be effective in preventing clay swelling with a lower dosage in the fracturing fluid than conventional salts, and they can cause less formation damage than long-chain polymers.^{22–24} Laumontite is another type of water-sensitive mineral. Although it is rarely seen in typical shales, tight sandstones, or tight carbonates, it is relatively rich in the Wuerhe formation in the Mahu-1 play. Each unit cell of the laumontite crystal has 8 sites that can bond 18 water molecules during hydration, and XRD results indicate that the cell volume can expand 2–6%.^{12,25,26} Compared with montmorillonite, laumontite does not significantly swell after reacting with water; although it can affect drilling and fracture propagation due to different mechanical properties from the reservoir rock, no stabilizer has been developed for preventing the swelling of laumontite during hydraulic fracturing.^{2,27}

When water-sensitive minerals swell after reacting with water, fine particles can detach from the rock due to the electrostatic repulsion; migrant of these fine particles can further plug small pores and pore throats, as well as the created hydraulic fractures. Meanwhile, water–rock interaction can also soften the matrix and even damage the rock skeleton; this can further result in the embedment of proppants and the closure of created fractures.^{5,28,29} Therefore, it is crucial to optimize the fracturing fluid additive to prevent the water–

rock interaction and maintain the conductivity of the fracture network at the reservoir closure pressure.

In this study, a stabilizer is optimized as the fracturing fluid additive for the Mahu-1 reservoir, which contains water-sensitive minerals like montmorillonite and laumontite. Combining the antishwelling rate measurement, soaking test with XRD analysis, and Brazilian splitting test, the effectiveness of the new stabilizer is evaluated and compared with KCl, which is already used in the field. The findings of this work establish a systematic evaluation method for optimizing the stabilizer as the fracturing fluid additive and meanwhile provide a solution for fracturing water-sensitive reservoirs.

2. MATERIALS AND METHODS

2.1. Reservoir Rock Samples. Mahu reservoir is currently the largest tight conglomerate oil reservoir in the world. After the successful development of the Baikouquan formation (T_3b), a relatively shallow section without water-sensitive minerals, the current focus has been shifted to the Wuerhe formation (P_3w), which has a depth of around 3500 m and a relative concentration of montmorillonite of more than 10%. After 10-cm-diameter cores are drilled from exploration wells from the Wuerhe formation (Figure 1a), 2.54-cm-diameter core samples are drilled from the side (Figure 1b) for laboratory evaluations; meanwhile, cuttings are also taken for the XRD analysis, soaking test, and ζ potential and antishwelling rate measurements, as introduced in the next section. Although conglomerates are heterogeneous in the core scale, it is

relatively homogeneous across the whole section of the formation, as can be observed in Figure 1a,b.

Cuttings are first ground into powders of 150 mesh for the XRD analysis. As shown in Figure 2, the reservoir rock is composed of 62.8% quartz, 21.1% plagioclase, 11.3% clay, and 4.8% calcite; within the clay, water-sensitive montmorillonite is the major component and accounted for 88% of the total concentration of clay minerals in the rock.

2.2. Stabilizers. As introduced already, salt can produce cations in water through ionization, which then enters the interlaminar space of montmorillonite and prevents water from invading through the electrostatic force. To match with sizes of vacancies in the interlaminar spaces of the montmorillonite crystal, KCl is chosen as the salt in this study to prevent the swelling of montmorillonite.

The XRD analysis shows that main compositions of the reservoir rock are quartz, plagioclase, and clay; therefore, the rock surface is negatively charged in the reservoir condition. Once fine particles detach from the rock surface due to the water–rock interaction, electric double layers formed on the surface of fine particles can make them repel from each other, which then can further weaken the rock structure. An inorganic cationic polymer (ICP) can likely neutralize the electric charges of fine particles, compress the electric double layers, and bind the fine particles, thus strengthening the rock structure under the closure stress. In this study, ICP-1 is chosen for evaluating the effectiveness of this type of chemical. ICP-1 is a branched short-chain polymer with a polymerization degree of 20–40 and a molecular weight of 2000–4000; each monomer of ICP-1 has a metal ion, which has one positive charge after the polymerization. First, 1% KCl is mixed with 0.5% ICP-1 or 1% ICP-1 and then compared with 4% KCl or 8% KCl through a series of laboratory evaluations as introduced below; this is aimed to evaluate and understand the synergy effect of the salt and ICP on antishwelling clay minerals and stabilizing the rock structure.

2.3. Evaluation Methods. **2.3.1. Measurement of the Antishwelling Rate.** The antishwelling rate shows the reduction of volume change of clay minerals after being submerged in solutions with different stabilizers, and thus it can quantify the degree of water–rock interaction. Before conducting the antishwelling rate measurement, the reservoir rock or the rock cuttings are ground into powders and dried at 105 °C in an oven for 6 h; meanwhile, pure montmorillonite powders are also dried in the same manner for the baseline measurement. To conduct measurement of the antishwelling rate, 0.5 g of rock powders or montmorillonite powders are loaded in a 10 mL centrifuge tube, which is about 0.55 mL in the tube. After filling 10 mL of a solution with a stabilizer (4% KCl, 8% KCl, 1% KCl + 0.5% ICP-1 or 1% KCl + 1% ICP-1), the centrifuge tube is sealed, agitated, and placed for attaining equilibrium at room temperature for 2 h. After the tube is centrifuged at 1500 rpm for 15 min, the volume of powders in the tube is recorded; this is repeated three times, and the average of measured volumes of powders is recorded as the expanded volume after being submerged in the solution with the stabilizer (V_1). Then, the same powders are loaded in two other 10 mL centrifuge tubes, one of which is filled with 10 mL of distilled water, while the other is filled with 10 mL of kerosene. Both tubes are sealed, agitated, and centrifuged similarly to the tube with the stabilizer, and the expanded volumes of powders are recorded as V_0 (in kerosene) and V_2 (in distilled water). As V_1 , both V_0 and V_2 are also measured three times and averaged for

calculating the antishwelling rate. The antishwelling rate η is defined in eq 1 shown below.³⁰

$$\eta = \frac{V_2 - V_1}{V_2 - V_0} \times 100\% \quad (1)$$

where V_1 is the volume of rock powders after being submerged in the solution with the tested stabilizer, V_2 is the volume of rock powders after being submerged in distilled water, and V_0 is the volume of rock powders after being submerged in kerosene.

2.3.2. Soaking Test. Once the stabilizer passes the measurement of the antishwelling rate, the rock sample is soaked in the solution with a stabilizer for at least 48 h, during which the effect of this stabilizer on maintaining the rock structure in water can be evaluated. After this soaking test is finished, the beaker with the rock sample and solution is gently shaken, and the supernatant is taken to conduct the ζ potential test. Using a ζ potential analyzer, the ζ potential of fine particles floating in the supernatant can be measured to determine the strength of the electric double layer on the rock surface. To further evaluate the antishwelling mechanism of the stabilizer on the water-sensitive mineral, XRD analysis is conducted for rock powders before and after being soaked in solutions with or without the stabilizer.

2.3.3. Brazilian Splitting Test. Water–rock interaction can weaken the rock structure, thus causing the embedment of proppants and the closure of propped fractures. However, this cannot be properly evaluated through the measurement of the antishwelling rate and the soaking test, since both are conducted under ambient pressure. Therefore, the Brazilian splitting test is conducted to evaluate the mechanical property change of the reservoir rock before and after being soaked in different mimicked fracturing fluids with or without the chosen stabilizers. To conduct the Brazilian splitting tests, core samples with diameters of 25 mm and 38 mm are used to compare the change of breakdown pressure after being interacted with different stabilizers. During the test, the core sample is soaked in the solution with a chosen stabilizer for 12 h and then dried at 60 °C for 2 h; after being wiped clean, the core sample is loaded in a triaxial testing system, in which the load is applied until the core sample is broken. The change of load on the core sample is recorded with the displacement of the press for analysis.

3. RESULTS

3.1. Measurement of the Antishwelling Rate. Typically, 2% KCl is one of the routinely used stabilizers; however, since the target reservoir has a high clay content, 4% KCl was applied in the fracturing fluid in the early field tests. Therefore, 4% KCl was used to establish the baseline for later comparisons in this study, even though no obvious enhancement was observed in those field tests. Figure 3a shows the volume changes of montmorillonite powders when measuring the antishwelling rate of 4% KCl, where solutions in centrifuge tubes from left to right are distilled water, 4% KCl, and kerosene; Figure 3b shows the volume changes of montmorillonite powders when measuring the antishwelling rate of the new stabilizer, 1% KCl + 0.5% ICP-1. By direct observation, it can be noticed that volumes of montmorillonite powders in distilled water or kerosene are almost identical in two sets of measurements, which shows the reliability of this measure-

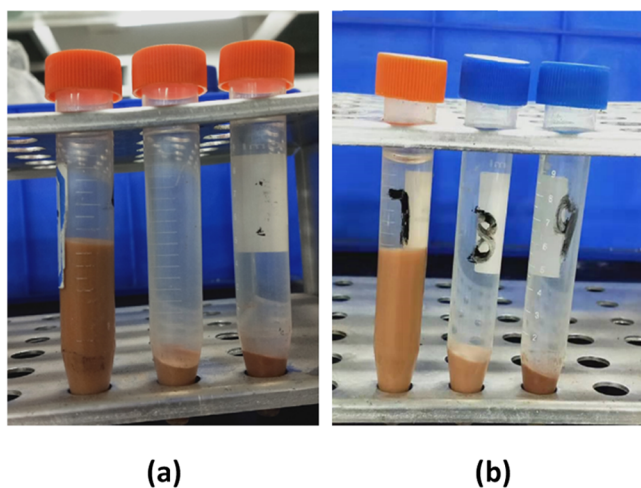


Figure 3. Volume change of montmorillonite soaked in different solutions ((a) left tube: distilled water; center tube: 4% KCl; right tube: kerosene; (b) left tube: distilled water; center tube: 1% KCl + 0.5% ICP-1; right tube: kerosene).

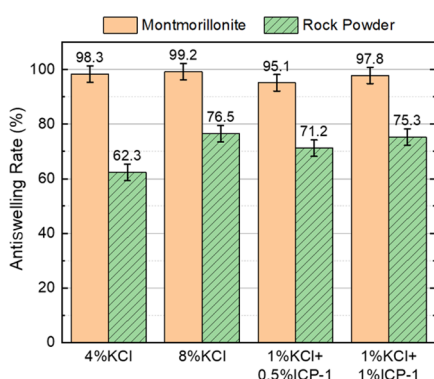


Figure 4. Calculated antishwelling rates of different stabilizers on montmorillonite or reservoir rock powders.

ment; however, the volume of montmorillonite powders is slightly larger in 1% KCl + 0.5% ICP-1 than in 4% KCl.

Figure 4 further shows the calculated antishwelling rates of different stabilizers according to eq 1, which shows that 8% KCl has the largest antishwelling rate of 98.3% on montmorillonite powders, followed by 4% KCl, 1% KCl + 0.5% ICP-1, and 1% KCl + 1% ICP-1. When the reservoir powders are used, all antishwelling rates decrease to around 60% to 70%; this is because water-sensitive minerals only take up about 10% of the reservoir rock, which reduces the difference between V_2

and V_0 and thus the antishwelling rate. When montmorillonite is used, antishwelling rates of 4% KCl and 8% KCl are both larger than that of the mixture of KCl and ICP-1; however, when the reservoir rock powder is used, antishwelling rates of the mixture of KCl and ICP-1 are slightly larger. Since the reservoir rock has a complex composition compared to the pure montmorillonite, the mixed stabilizer performs better on other potential water-sensitive minerals like laumontite; meanwhile, its synergy effect can also compress the electric double layer on the rock surface and enhance the antishwelling rate of the stabilizer.

3.2. Soaking Test. Figure 5 shows the reservoir rock samples before and after being placed in 4% KCl. Although 4% KCl can reach an antishwelling rate of over 98% on montmorillonite, it can be observed that fine particles start to detach from the rock sample once the sample is placed in 4% KCl. Small fractures form on the rock sample due to the water–rock interaction on the first day, and the rock sample completely falls into pieces on the second day, as shown in Figure 6. Although water-sensitive minerals account for only about 1% of the cement in the reservoir rock (as shown in Figure 2), they can significantly trigger the water–rock interaction that breaks the integrity of the rock. Results also indicate that the antishwelling rate by itself is not enough to be used to screen the stabilizer for water-sensitive formations like Mahu-1 play.

Meanwhile, rock cuttings from the same core plug are placed in solutions with 1% KCl + 0.5% ICP-1 and 1% KCl + 1% ICP-1 for comparison, as shown in Figure 7. After being soaked for 48 h, both samples maintain the structural integrity; small fractures can be observed in the sample submerged in 1% KCl + 0.5% ICP-1, while the other sample almost remains the same, as shown in Figure 8. When comparing rock samples soaked in 4% KCl and mixtures of KCl with ICP-1, one can find that a high antishwelling rate on water-sensitive minerals cannot guarantee good performance on preventing the softening of the reservoir rock. Unlike screening stabilizers for drilling in water-sensitive formations, more screening methods are needed besides the antishwelling rate test.

After beakers containing rock samples and different stabilizer solutions are gently shaken and placed for attaining equilibrium, the supernatant is drawn from each beaker for measuring the ζ potential of fine rock particles therein. As shown in Figure 9, rock particles are originally negatively charged with a ζ potential of -17.5 mV, which is expected from the XRD analysis as shown in Figure 2; when KCl or ICP is applied, the ζ potential is changed to positive because of the absorbed cationic ions or short-chain polymers on rock

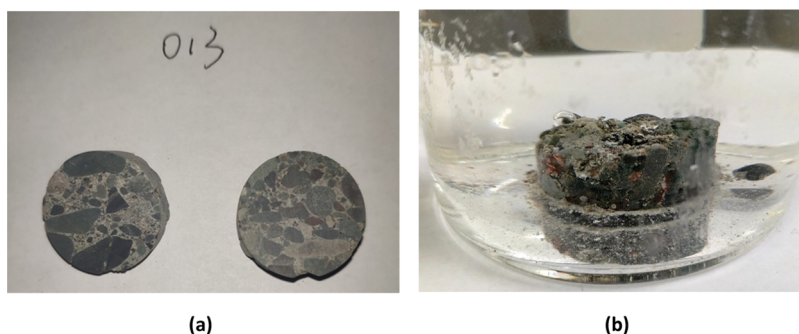


Figure 5. (a) Reservoir rock samples before conducting the soaking test; (b) rock sample just submerged in 4% KCl.

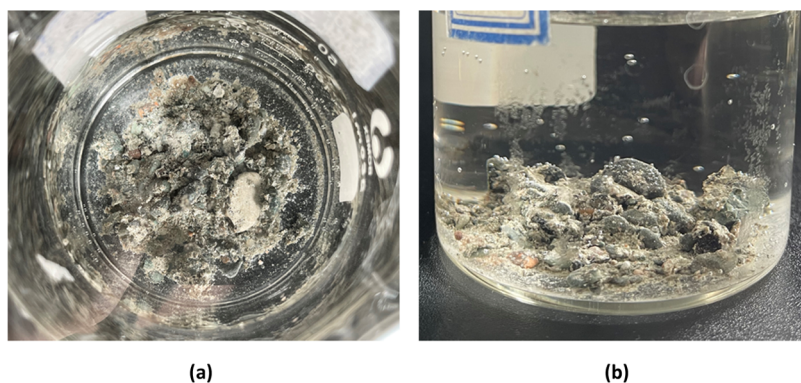


Figure 6. Top view (a) and side view (b) of the rock sample submerged in 4% KCl for 48 h.

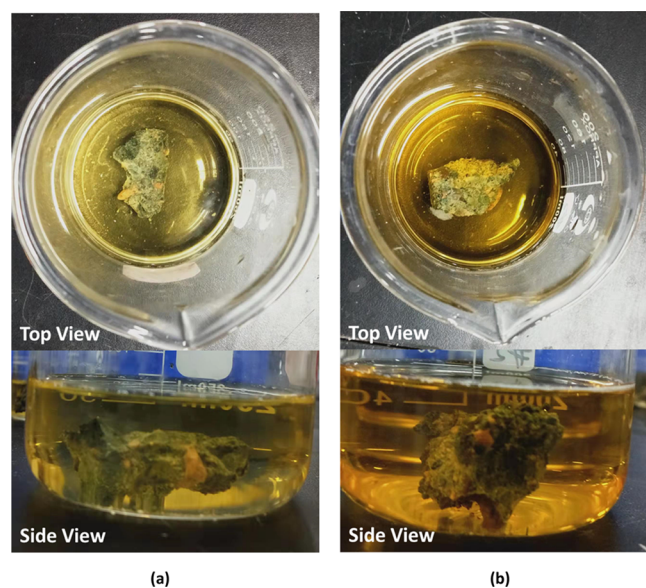


Figure 7. Rock samples just submerged in (a) 1% KCl + 0.5% ICP-1 and (b) 1% KCl + 1% ICP-1.

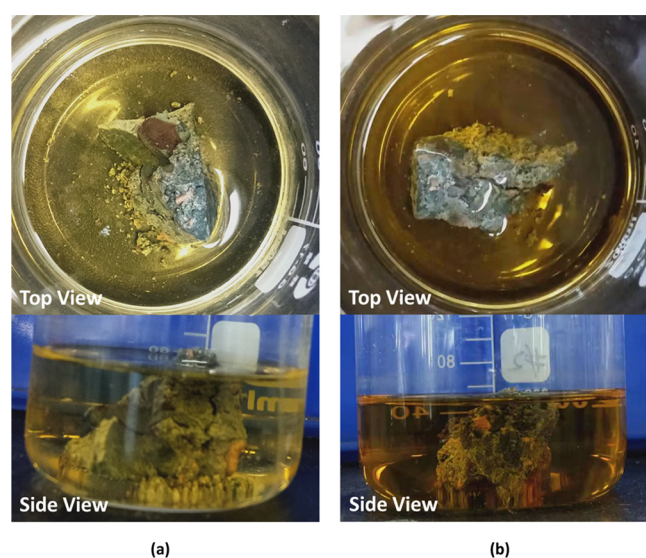


Figure 8. Rock samples submerged in (a) 1% KCl + 0.5% ICP-1 and (b) 1% KCl + 1% ICP-1 for 48 h.

powders. Compared to KCl solutions, the absolute value of the ζ potential can be reduced to 7.5 mV by 1% KCl + 0.5% ICP-1

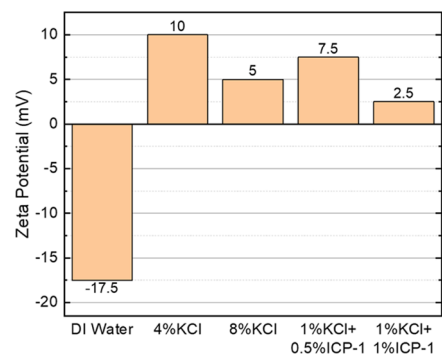


Figure 9. ζ Potentials of fine powders submerged in solutions with different stabilizers for 48 h.

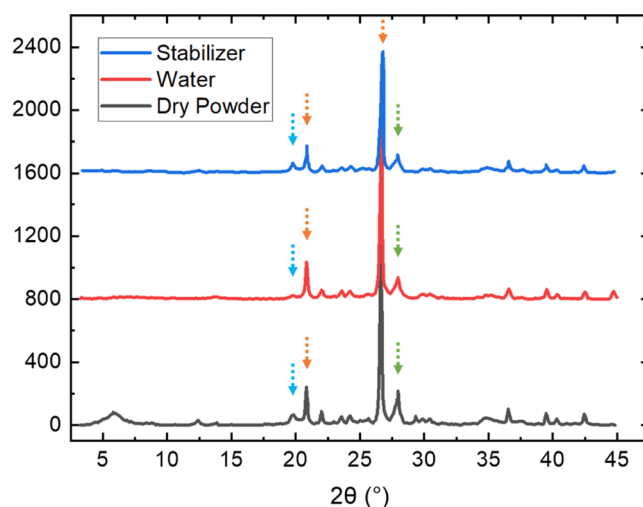


Figure 10. XRD results of reservoir rock powders after being submerged in the stabilizer solution or water for 48 h.

and further to 2.5 mV by 1% KCl + 1% ICP-1. A smaller ζ potential value indicates a thinner electric double layer and thus a smaller repulsion among fine rock particles, which explains why the new stabilizer recipes can prevent the destruction of rock structure in the soaking tests as shown in Figures 7 and 8.

To further study the antishwelling mechanism of the stabilizer on water-sensitive minerals, XRD analysis is conducted on reservoir rock powders that are submerged in distilled water or the stabilizer solution with 1% KCl + 1% ICP-1 for 48 h. Figure 10 compares the XRD curves among dry rock powders

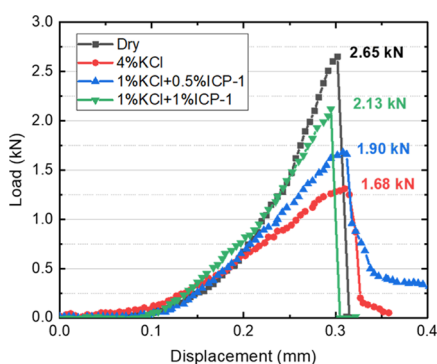


Figure 11. Load histories of 25-mm-diameter cores during Brazilian splitting tests.

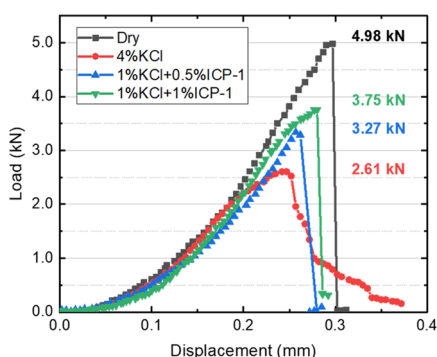


Figure 12. Load histories of 38-mm-diameter cores during Brazilian splitting tests.

(black curve at the bottom), rock powders submerged in distilled water (red curve in the middle), and rock powders submerged in the chosen stabilizer (blue curve on the top). In each curve, the two highest peaks at around 26.7 and 20.8° are characteristic peaks of quartz as pointed by red arrows; the small peak between 17.48 and 21.53° is the second characteristic peak of montmorillonite as pointed by blue arrows, while the small peak at around 28.0° is the characteristic peak of plagioclase as pointed by green arrows. After being soaked in distilled water or the stabilizer solution, quartz and plagioclase peaks remain the same since they do not interact with water. However, for the water-sensitive montmorillonite, the characteristic peak almost disappears after being soaked in distilled water; this is because water molecules can enter the interlaminar spaces of montmorillonite cells, whose expansion inhibits the diffraction of X-rays from the lattice planes generating the characteristic peaks. This

finding agrees with observations from antishwelling tests and soaking tests as shown in Figures 4–8.

3.3. Brazilian Splitting Test. Softening of the reservoir rock due to the water–rock interaction can cause the embedment of proppants and the closure of hydraulic fractures, but this cannot be characterized through the soaking test that is conducted under ambient pressure. Therefore, the Brazilian splitting test is conducted to quantify the mechanical property change due to the water–rock interaction. Figure 11 shows the load change on 25-mm-diameter rock samples when solutions with different stabilizers are applied. Although three stabilizers have similar antishwelling rates as shown in Figure 4, they perform differently on maintaining the rock structure. The breakdown load decreases from 2.65 to 1.68 kN when 4% KCl is used, which is equivalent to a 36.5% reduction compared to the dry rock; however, the breakdown load decreases only to 2.13 kN when 1% KCl + 1% ICP-1 is used, which is equivalent to a 19.9% reduction. Figure 12 shows the load change on 38-mm-diameter rock samples when different stabilizers are applied, and similar trends can be observed. This agreement shows the repeatability and reliability of this measurement in quantifying the effectiveness of clay stabilizers in preventing matrix softening. Table 1 lists the dimensions of all rock samples used in this measurement and the solution applied to these samples, as well as the measured breaking load when rock is fractured. From the straight line of each failure curve, Young's modulus of each rock sample can be calculated as also shown in Table 1. Evaluation results indicate that the new formula can enhance the Young's modulus of the reservoir rock after reacting with water by more than 50% without increasing the cost, and it is promising to be used as a fracturing fluid additive for water-sensitive reservoirs.

4. CONCLUSIONS

Mahu reservoir is the largest tight conglomerate oil reservoir in the world, and the current development focus is the Wuerhe formation, which is rich in water-sensitive minerals. Although KCl has a high antishwelling rate on clay minerals and is typically considered as an effective clay stabilizer during drilling and fracturing, early field experiments using 4% KCl in the fracturing fluid still show a high flowback ratio and a low oil production rate, as neighboring wells without using any clay stabilizer. It is believed that 4% KCl is not enough to inhibit the water–rock interaction and prevent the closure of created fractures due to matrix softening.

In this study, a systematic laboratory evaluation method is established to screen a stabilizer for the target reservoir. A mixture of a salt and a short-chain inorganic cationic polymer is chosen and compared with 4% KCl and 8% KCl. Although

Table 1. Information of Core Samples in Brazilian Splitting Tests

rock sample diameter (mm)	rock sample thickness (mm)	treatment of rock sample	breaking load (N)	reduction rate of breaking load (%)	Young's modulus (GPa)	reduction rate of Young's modulus (%)
25	12.91	dry	2649		14.57	
25	12.63	4% KCl	1680	36.5	3.76	74.2
25	12.66	1% KCl + 0.5% ICP-1	1899	28.3	5.88	59.7
25	13.05	1% KCl + 1% ICP-1	2123	19.9	9.64	34.0
38	19.44	dry	4980		15.31	
38	19.40	4% KCl	2605	47.6	7.85	48.7
38	19.42	1% KCl + 0.5% ICP-1	3266	34.4	11.87	22.5
38	19.45	1% KCl + 1% ICP-1	3752	24.7	14.55	5.0

the measurement of the antiswelling rate does not show obvious advantages of the mixture, soaking tests with ζ potential measurements indicate that the synergy effect between the two chemicals can effectively compress the electric double layers on the rock surface and prevent water from entering interlaminar spaces of the water-sensitive mineral, thus inhibiting the weakening of the rock structure due to the water–rock interaction. This is further confirmed by the Brazilian splitting test that quantifies the mechanical change of the reservoir rock after interacting with different stabilizer solutions. Experimental results successfully explain why adding 4% KCl in the fracturing fluid cannot prevent the high water flowback and fast production decline in the Mahu-1 play; meanwhile, this work also provides a solution to fracturing water-sensitive reservoirs and further establishes a set of laboratory methods for optimizing stabilizers as fracturing fluid additives.

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Notes

The authors declare no competing financial interest.

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