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Development and performance evaluation of multi-functional slickwater fracturing fluid system for shale reservoirs

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The rapid decline in energy and production following the primary fracturing development of shale oil reservoirs, and the low production of single wells are the problems facing the development of Cangdong shale oil in the Bohai Bay Basin. A multi-functional fracturing fluid system with emulsification, viscosity reduction, oil washing, and wettability improvement was developed by introducing a mixed-charge surfactant (PSG) to environmentally friendly and low-cost slickwater fracturing fluid (SWFF). The evaluation focused on several key properties including temperature and shear resistance, interfacial activity, viscosity reduction, wettability, and oil washing, all of which were assessed through laboratory experiments. The results showed that the 0.2% PSG and SWFF have high compatibility, forming a functional fracturing fluid system that exhibits exceptional temperature and shear resistance, as well as high interfacial activity. The rate of emulsification and viscosity reduction between the system and GY734H shale oil exceeds 93.45%. Moreover, the system is completely demulsified after being left to stand at 70 °C for 2 h. Furthermore, the static oil washing efficiency of the functional fracturing fluid system at 80 °C is 20.99%, while the SWFF is only 11.65%, which confirms the indoor effectiveness of the system. With the further optimization and application of the multifunctional slickwater fracturing fluid system (SWFF + PSG), it is expected to play an important role in the efficient development of shale reservoirs in the Bohai Bay Basin.

Keywords Shale oil, Fracturing fluid, Viscosity reduction, Oil washing, Cangdong Sag

In recent years, the exploration and development of unconventional oil and gas resources have received extensive attention with the continuous growth of global energy demand and the gradual depletion of conventional oil and gas resources[1]. Shale oil is an important unconventional and strategic oil and gas resource, and its development is of great significance to ensure energy security[2]. The exploration and development of shale oil has become one of the hot spots in the petroleum field. However, unlike conventional reservoirs, shale reservoirs are characterized by strong reservoir heterogeneity, ultra-low porosity, and ultra-low permeability[3–5], which makes them difficult to exploit, necessitating economical and effective development via large-scale hydraulic fracturing[6].

The amount of laminated shale oil resources in the Huanghua Depression of Bohai Bay Basin is 3.26 billion tons, which is 1.2 times that of conventional oil resources, and the exploration and development potential is huge[7]. Among them, two oil-rich depressions, i.e. Cangdong Sag and Qikou Sag, are developed in the Dagang exploration area[8]. The second member of Paleogcene Kongdian Formation (Ek_2), located in Cangdong Sag, has a burial depth of 3000–5000 m, a thickness of 400 m, and covers a favorable area of 260 km[29,10]. The Ek_2 is the main area of shale oil exploration and development, with preliminary estimated resources of 680 million tons[11]. At present, the industrial development of continental shale oil has been realized in the Ek_2, which provides a valuable reference for China's shale oil revolution[12]. However, following the primary fracturing development of shale oil reservoirs in China, the early single-well shale oil production is high, while the later production and energy rapidly decline, and the final recovery rate is still at a low level. Therefore, it is urgent to explore new technologies to enhance shale oil recovery.

For low permeability reservoirs, hydraulic fracturing technology is generally used to inject fracturing fluid into the target reservoir through injection Wells to reform the reservoir and artificially create fractures, thereby improving the flow capacity of crude oil[13]. As an environmentally friendly and highly efficient fracturing fluid

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system[14,15], slickwater fracturing fluid (SWFF) has significant advantages in improving fracture conductivity and reducing construction costs[16]. However, its ability to wash oil in complex shale reservoir conditions is limited[17]. In contrast, the viscoelastic surfactant fracturing liquid system can self-assemble into viscoelastic worm-like micelles under the electrostatic shielding of salt ions, which significantly improves the viscosity of the solution[18,19]. Moreover, the fracturing liquid system exhibits high interfacial activity, enabling full exploitation of oil-water imbibition displacement, thereby realizing the efficient development of ultra-low permeability reservoirs[20]. However, the substantial quantity and high cost of the viscoelastic surfactant fracturing fluid present significant barriers to its widespread adoption and application in oilfields[19,20]. Fan et al. found that adding surfactants to the fracturing fluid improved the imbibition efficiency, as evidenced in a study examining the development of a tight oil reservoir in the Changqing oilfield and the field test has obtained an obvious production improvement effect[21]. Zhao et al. developed a Gemini surfactant-based SWFF system for ultra-low permeability reservoirs that the final recovery rate of the system was 15.5%, which was 1.6 times that of slickwater[22]. To improve the primary fracturing production of low-permeability and high-viscosity reservoirs, Chen et al. constructed a multi-functional composite unconventional viscoelastic fracturing fluid system by adding a viscosity-reducing surfactant to fracturing fluid, and field tests found that the oil production of Well A can reach 30 t/d[23]. Nur Wijaya et al. analyzed in detail the benefits of surfactant added to fracturing fluid through mechanism studies[24]. Surfactants in fracturing fluid can decrease the interfacial tension (IFT) between shale oil and water by adsorbing at the oil-water interface[24,25]. On the other hand, they can improve the wettability of rock through adsorbing in shale micro-fractures, reducing the adhesion work between shale oil and rock, and thus improving the efficiency of imbibition displacement[24,26,27]. Simultaneously, fracturing fluid can effectively replenish the formation energy, thereby improving the production and recovery of shale oil per well[13]. However, the geological conditions of shale reservoirs in the Ek_2 differ from those in other basins[28], such as high temperature (120 ~ 150 °C), high pressure, deep burial, poor oil quality, etc. High temperatures often cause the surfactant to be inactivated or even broken down. Thus, the application of conventional surfactants poses challenges for the Ek_2 reservoirs. Additionally, the incompatibility between surfactants and fracturing fluids presents a significant challenge. Hence, this study aims to prepare a multifunctional slickwater fracturing fluid system that is tailored for the shale reservoir of the Ek_2. In our previous work, we reported a high-temperature-resistant surfactant (PSG) with a high interfacial activity that can emulsify shale oil in the Dagang oilfield[29]. However, it is not clear whether it can be used as an additive to prepare functional fracturing fluids. In this work, using the previously reported PSG surfactant, we leverage its environmentally friendly and cost-effective attributes inherent in the SWFF. PSG is added to the existing SWFF system to achieve the versatility of emulsification, viscosity reduction, oil washing, and wettability improvement, thereby achieving the purpose of enhancing oil recovery for shale reservoirs. Meanwhile, the use of this functional fracturing fluid has the potential to reduce the frequency of re-fracturing, thereby reducing development costs over the life of the shale reservoir. Furthermore, this study provides a theoretical foundation and technical support for the highly efficient development of shale oil in the Bohai Bay Basin.

Experimental sections

Chemicals and materials

GY743H shale oil was obtained from the second member of the Kongdian formation (Ek_2), Cangdong Sag, China. At 50 °C, the viscosity of crude oil is 2220 mPa·s, and its density is 0.9084 g/cm^3. The wax content is 23.54%, and the freezing point is 42 °C. Petroleum ether was purchased from Tianjin Kemiou Chemical Reagent Co., LTD, China. PSG surfactant was synthesized in the laboratory, and it is a kind of catanionic surfactant (Fig. 1)[29]. The critical micelle concentration (CMC) and hydrophilic-lipophilic Balance (HLB) values of the PSG surfactant are 0.43 mmol/L and 13.8, respectively. The filed water was obtained from the Third Oil Production plant of Dagang Oilfield, with a total salinity of 2520.46 mg/L and water type of $MgCl_2$, including 600.01 mg/L $Na^+ + K^+$, 90.18 mg/L Ca^{2+}, 133.71 mg/L Mg^{2+}, and 456.28 mg/L SO_4^{2-}, 994.98 mg/L Cl^-, and 245.30 mg/L HCO_3^-

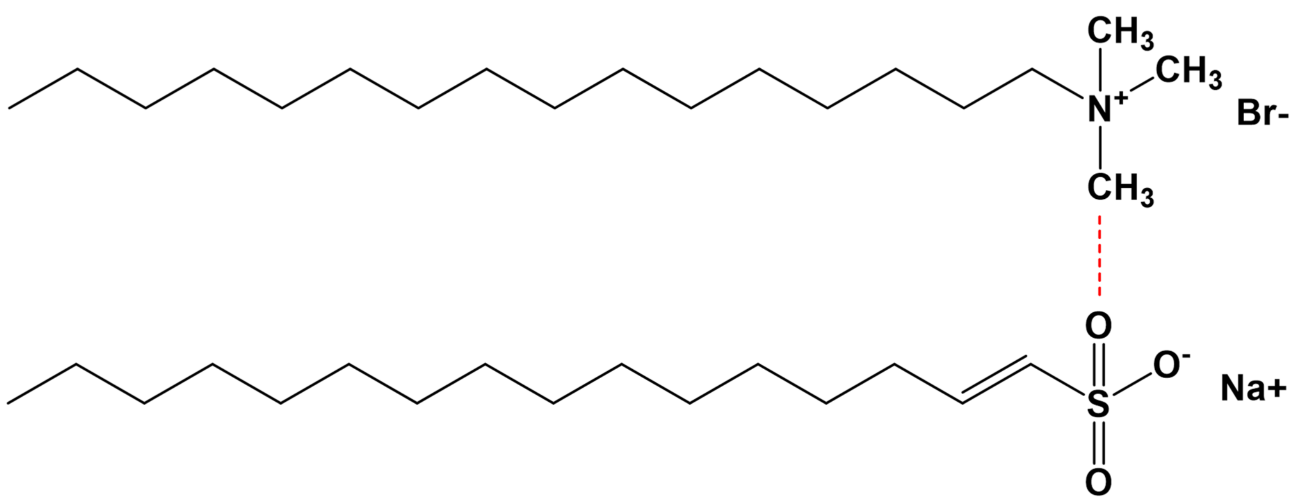

Fig. 1. Molecular structure of PSG surfactant.

. The SWFF was provided by the Petroleum Engineering Research Institute of Dagang Oilfield, which mainly includes slickwater, anti-swelling agent, and bactericide. Natural shale core is taken from the EK_2 reservoir of Dagang Oilfield.

Preparation of functional fracturing fluids

First, the SWFF was prepared using field water with a composition of 0.5% slickwater, 0.5% anti-swelling agent, and 0.01% bactericide. Then, various concentrations of PSG surfactants were added to this fracturing fluid. The mixture was stirred at 500 rpm for 2 h at ambient temperature to ensure thorough blending and finally formed a functional slickwater fracturing fluid (SWFF + PSG).

Evaluation of temperature and shear resistance

According to the test method of temperature and shear resistance reported by Chieng et al.[30], the viscosity of the above fracturing fluid system was measured by the HAAKE rheometer (RS6000, Germany) at a fixed shear rate of 170 s^{-1} within a temperature range of 20–100 °C. The temperature was raised to 100 °C at a rate of 3.5 °C/min, and then the viscosity of the fracturing fluid was maintained at this temperature for 60 min. The temperature and shear resistance were evaluated during this process.

Interfacial activity evaluation

The dynamic IFT between the fracturing fluid system and GY734H shale oil was measured by the CNG rotating drop interfacial tensiometer (CNG701, USA) at 90 °C and a rotational speed of 5000 rpm for 20 min.

Wettability evaluation

The natural shale core was first cut into three core columns (Diameter × height = 2.5 cm × 1 cm), and the measuring surface was polished with 500-mesh sandpaper to ensure smooth, flat, and consistent. Subsequently, the shale core columns were soaked in field water, shale oil, and fracturing fluids at 80 °C for 24 h, respectively. Whereafter, the cores were washed and then dried. Their contact angles with water were measured using a DataPhysics automatic contact angle measuring instrument (OCA50AF, Germany) before and after soaking to evaluate the wettability of the samples. Specifically, a drop of 5 μL of field water droplet was placed on the core surface, and the image was taken after the drop for 5 s. The contact angle between the core and water was calculated using the software provided with the instrument. Each sample was tested three times, and the average value was recorded.

Evaluation of emulsifying and viscosity reduction

Viscosity reduction properties of SWFF and SWFF + PSG were evaluated. First, the fracturing fluid was mixed with GY734H shale oil at an oil-water ratio of 1:1 and kept in a 70 °C water bath for 1 h. Subsequently, the mixture was manually stirred with a glass rod for 2 min to ensure complete emulsification. The micro-morphological characteristics of the emulsified mixture were observed by an optical microscope (80i, Nikon, Japan). The droplet particle size and distribution were then analyzed by Image J software[31]. Additionally, the viscosity of the mixture was determined using a Brookfield RV-DV3T rotary viscometer at 50 °C, and the viscosity reduction rate was calculated according to Eq. (1).

$$\varnothing = \frac{\eta_0 - \eta}{\eta_0} \times 100\% \tag{1}$$

Where, $\varnothing$ is viscosity reduction rate, %. η_0 is the initial viscosity of GY734H shale oil, and η is the viscosity of GY734H shale oil after emulsifying and viscosity reduction at 50 °C, mPa·s.

The prepared emulsions were transferred to a 50 mL stoppered measuring cylinder. Their percentages of separated water at 70 °C were observed to evaluate the stability of the emulsion, and the demulsification rate after 1 h was calculated using Eq. (2).

$$\phi = \frac{V_0 - V}{V_0} \times 100\% \tag{2}$$

Where, ϕ is the demulsification rate of the emulsified mixture after standing at 70 °C for 1 h, %. V_0 is the volume of adding fracturing fluid, mL. V is the volume of the separated fracturing fluid after standing at 70 °C for 1 h, mL.

Oil washing performance evaluation

According to the evaluation method of static oil washing efficiency reported by industry-standard Q/SH1020 2191 – 2021 and Zhao et al.[32]. A mixture of 15 g GY734H shale oil and 85 g quartz sand was aged in an oven at 80 °C for 48 h, and the mixture was stirred every 12 h to finally obtain oil sand. Then, petroleum ether is used to thoroughly extract the shale oil from the oil sand. The mass of the oil sand before and after extraction is weighed to determine the oil content of the oil sand. Subsequently, the oil sand and fracturing fluid were weighed in a conical bottle, maintaining a mass ratio of 1 : 5. These substances were then placed in a constant temperature oven at 80 °C for 48 h. After the shale oil in the oil sand was washed out, and the efficiency of static oil washing was calculated according to Eq. (3).

$$\omega = \frac{m_s - m}{m_0 \bullet C} \times 100\% \tag{3}$$

Where, ω is static oil washing efficiency, %. The m_0 is the mass of oil sands, g. The m_s and m are mass of conical bottles and oil sands before and after washing, g. The C is the oil content of oil sands, %.

Results and discussion

Temperature-resistance and shear-resistance

Temperature and shear resistance are important parameters of fracturing fluid performance. They are one of the key factors that determine the success or failure of a fracturing operation[33]. As shown in Fig. 2, compared to the SWFF system, the viscosity of the functional fracturing liquid system after adding 0.2%PSG surfactant is almost unchanged, while the viscosity gradually decreases with further increasing PSG concentration. Furthermore, the viscosity of the SWFF + 0.2%PSG system remains at 4.65 mPa·s after shear at 100 °C and 170 s^{-1} for 60 min, which is close to that of the SWFF system (4.62 mPa·s), indicating that the addition of 0.2% PSG will not reduce the temperature and shear resistance of SWFF. Nevertheless, high concentrations may lead to a decrease in temperature resistance and shear resistance. PSG is a kind of mixed-charge surfactant[29], and it combines cationic and anionic functionalities, enabling simultaneous reduction of interfacial tension (IFT) and alteration of wettability because the opposing charges promote electrostatic interactions at the oil-water-rock interface[34,35]. Additionally, this surfactant is generally conducive to the formation of wormlike micelle structure[36], but excess may cause the wormlike micelle to form a branched structure or transform into layer structure[37,38], thereby destroying the wormlike network structure, resulting in a decrease in apparent viscosity[20,39].

Oil/water interfacial tensions

Many studies have found that the addition of surfactants can improve oil recovery by the mechanism of reducing IFT, altering wettability toward more water-wet, and detaching adsorbed oil from rock surfaces[24,40,41]. Consequently, it is very important to investigate the oil-water IFT after adding a surfactant to the fracturing fluid. As shown in Fig. 3, the equilibrium IFT between filed water and GY734H shale oil is maintained at about 10 mN/m, while the equilibrium IFT between SWFF and shale oil is about 3.53 mN/m due to the presence of drag reducer components. In contrast, the equilibrium IFT decreased to 0.14 mN/m after adding 0.2% PSG surfactant to SWFF, and the equilibrium IFT first decreased and then slightly increased with further increasing PSG concentration to 0.6%. This can be attributed to the surfactant molecule reaching a state of saturation

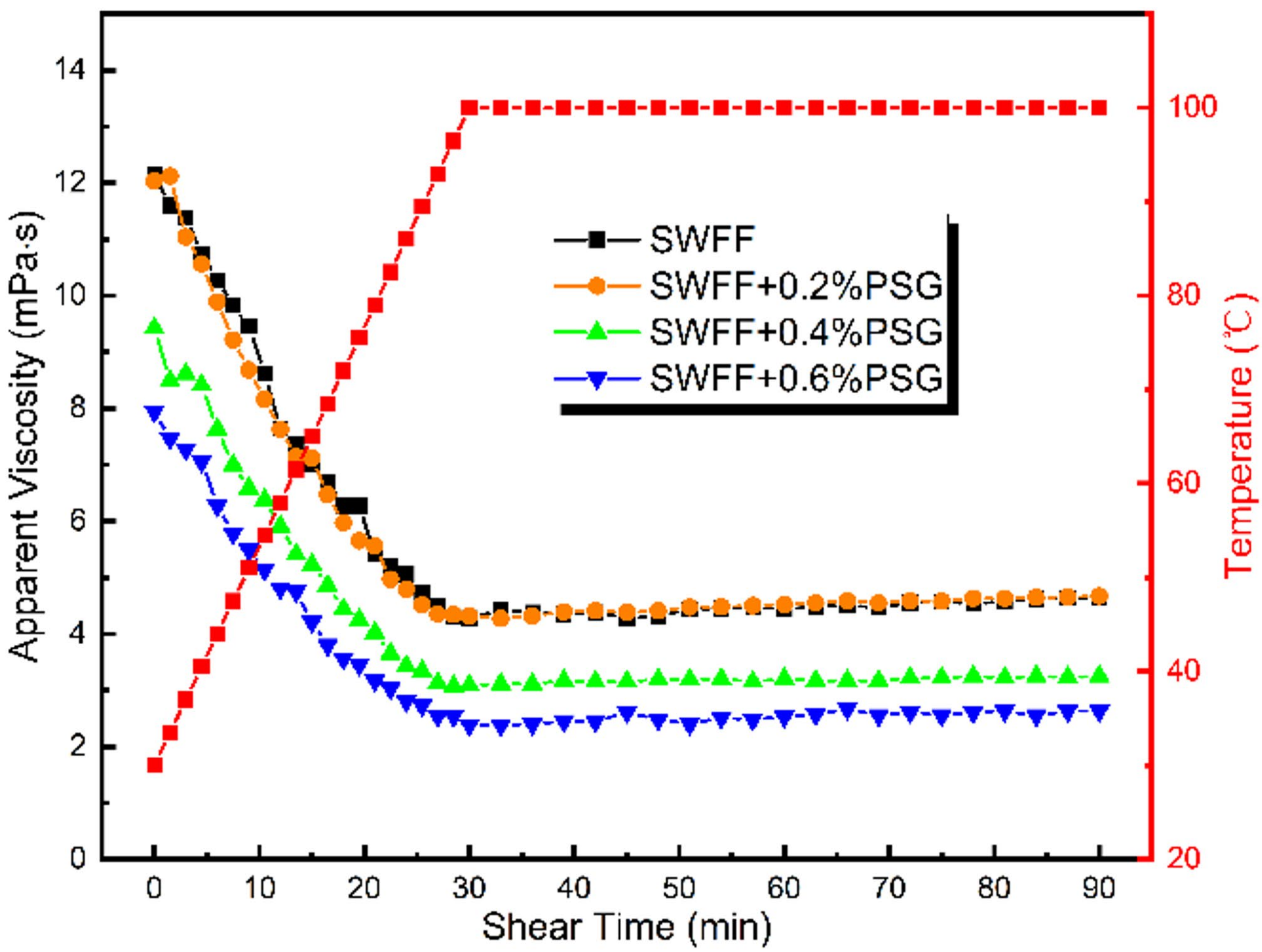

Fig. 2. Temperature and shear resistance of SWFF and SWFF + PSG systems.

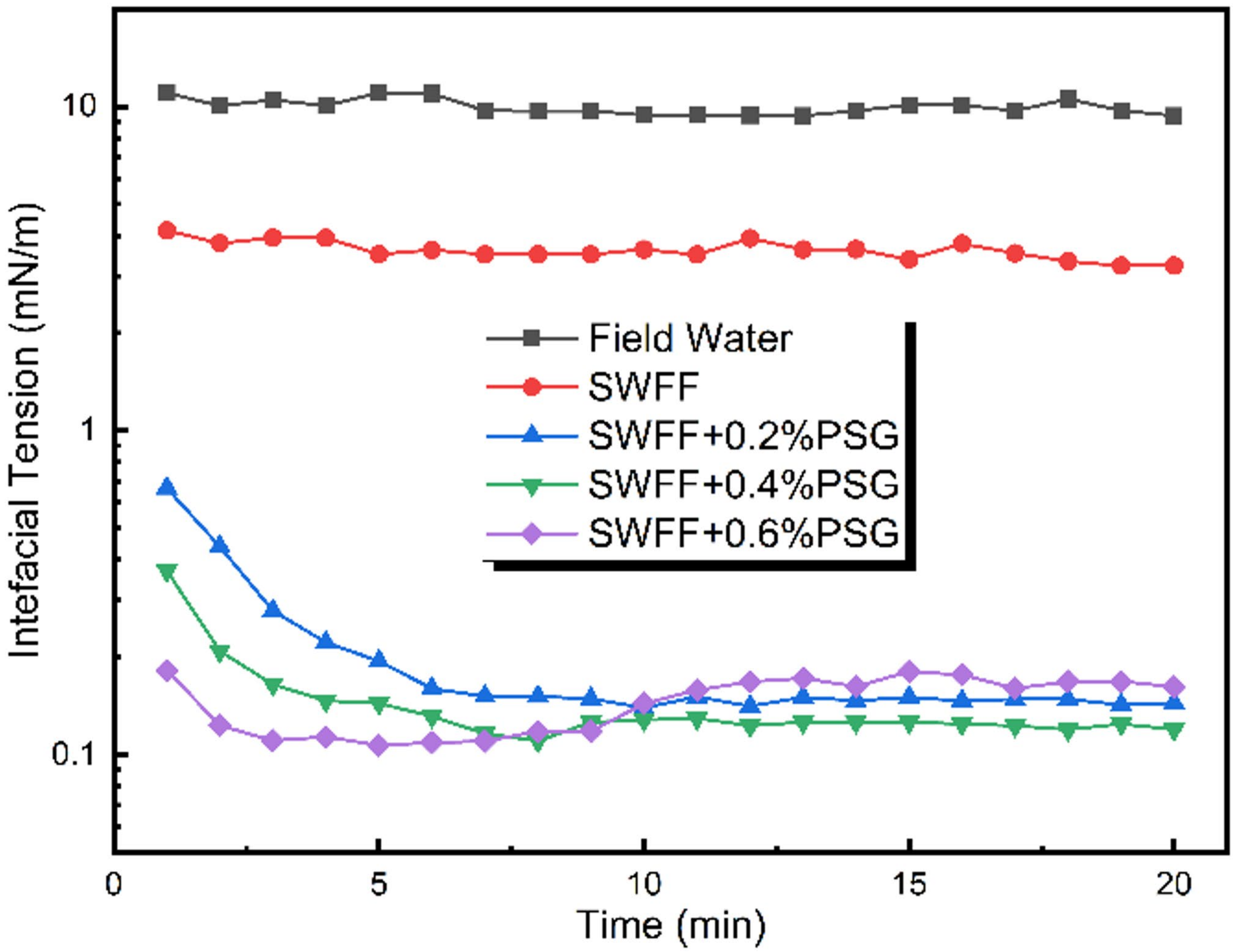

Fig. 3. The IFT between the fracturing fluid system and GY734H shale oil.

adsorption at the oil-water interface. Upon further increase in surfactant concentration, the surfactant molecule would be transformed into micelles, resulting in a partial desorption of surfactant molecules[42]. Thus, the IFT slightly increased. The findings suggest that the functional fracturing fluid system formed after adding a small amount of PSG to SWFF exhibits high interfacial activity, which is beneficial to increase the imbibition and oil-washing effect during the primary fracturing development for shale reservoirs.

Emulsifying and viscosity reduction of shale oil

Although horizontal well fracturing technology has effectively increased the macro sweep volume and substantially improved the production efficiency of shale reservoirs[23], the viscosity of Ek_2 Shale oil in Cangdong Sag is generally higher than 50 mPa·s at 50 °C, resulting in a limited flow of shale oil through pores and microfractures, which is hard to achieve ideal imbibition displacement[43]. Therefore, using fracturing fluid to reduce the viscosity of shale oil is the key to enhancing oil recovery. As shown in Fig. 4a, shale oil and fracturing fluid appear as two separate phases before emulsifying, with the upper layer being shale oil and the lower layer being fracturing fluid. After emulsifying, SWFF and shale oil were not completely emulsified, and a small amount of fracturing fluid was precipitated. However, after adding PSG to SWFF, shale oil and fracturing fluid were completely emulsified, and no fracturing fluid was precipitated (Fig. 4b). The emulsified system was completely phase-separated after standing at 70℃ for 2 h (Fig. 4c). From a viscosity standpoint (Fig. 5), the initial viscosity of shale oil is 2220 mPa·s at 50℃, while SWFF and shale oil are emulsified to form water-in-oil (W/O) emulsion (Fig. 6a), resulting in an increase in the viscosity of the system to 2780 mPa·s. Because of the low interfacial activity of SWFF, shale oil cannot be reversed to oil-in-water (O/W) emulsion. In contrast, the addition of PSG with high interfacial activity can emulsify shale oil to form an oil-in-water (O/W) emulsion (Fig. 6b-d), thereby significantly reducing the viscosity of the system with a viscosity reduction rate exceeding 93.45% (Fig. 5). Furthermore, the higher the amount of PSG added, the smaller the particle size of the corresponding O/W droplets (Fig. 6b-d), resulting in a better viscosity reduction effect (Fig. 5). The mechanism of viscosity reduction is the emulsification effect of PSG (Fig. 7). The initial shale oil forms a W/O emulsion due to the adsorption of components such as resins, asphaltenes and waxes at the oil-water interface during the flow process. However, after adding 0.2% PSG surfactant, PSG molecule rapidly adsorbs at the oil-

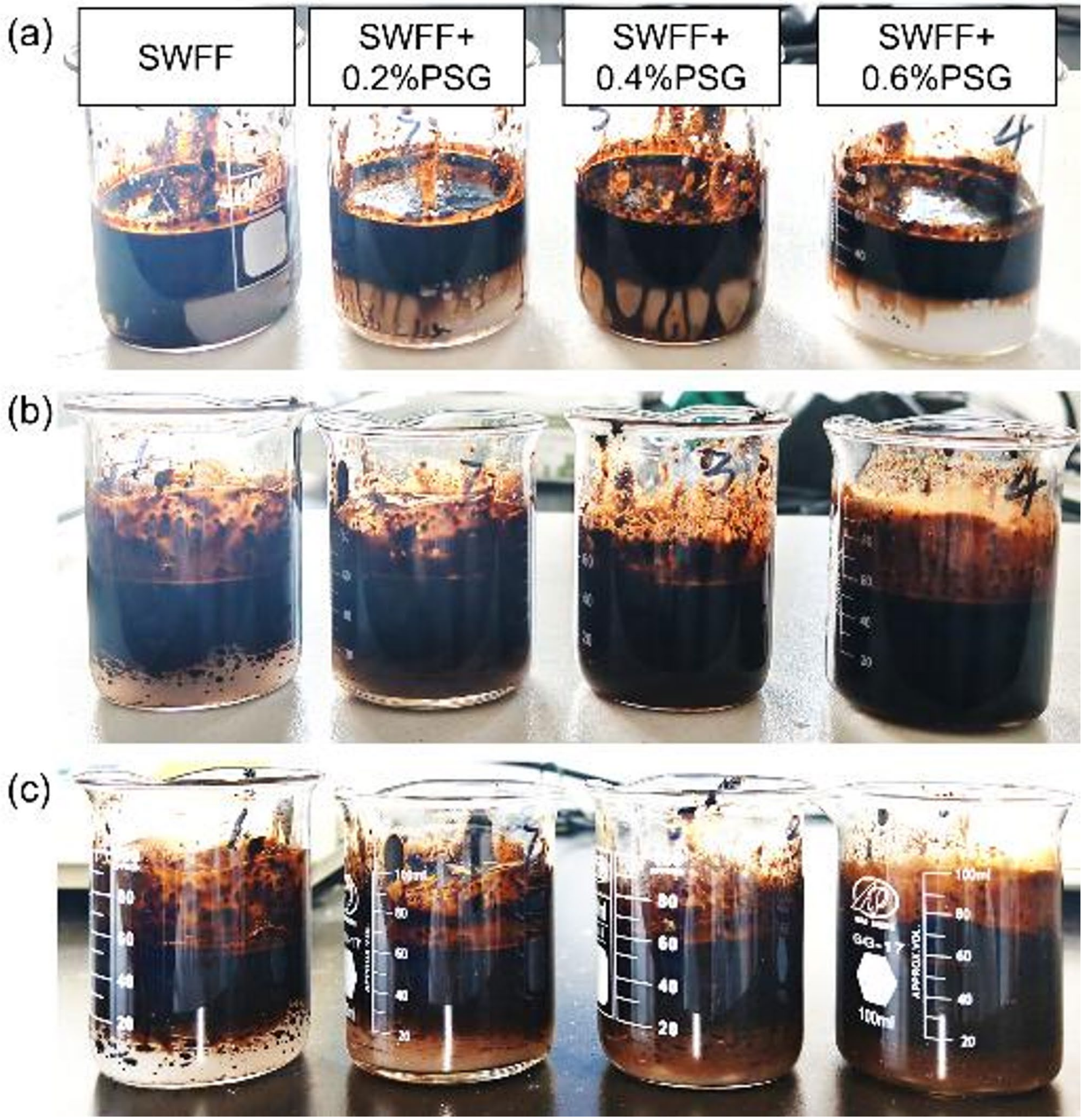

Fig. 4. Images of the emulsifying and viscosity reduction process of shale oil by using fracturing fluid at 70 °C: (**a**) at the beginning and un-emulsified, (**b**) after emulsifying for 0 h, and (**c**) after standing for 2 h.

water interface, simultaneously reducing the interfacial tension between shale oil and water, reversing the W/O emulsion into an O/W emulsion, thereby reducing the viscosity of the system. Besides, after adding PSG to SWFF, the demulsification rate of the mixed system was significantly reduced, suggesting an enhancement in emulsion stability. Moreover, the demulsification rate of all systems exceeded 80% after standing at 70 °C for 1 h, and the system was completely broken after 2 h (Fig. 8). These results also indicate that the addition of PSG does not influence the pipeline transportation or terminal demulsification processes of shale oil. Considering the temperature and shear resistance, cost, and demulsification efficiency, the SWFF + 0.2%PSG was selected as the optimal formulation for the functional fracturing fluid.

Surface wettability of shale

In shale reservoirs, the flow of fluid is confined under the nanoconfinement of shale pores due to capillary pressure[3]. As described by the Young-Laplace equation, the capillary pressure (P_c) is mainly related to the capillary diameter (r), interfacial tension (σ), and wettability ($P_c = 2\sigma \cdot \cos\theta \ / \ r$)[26]. As the σ or water contact

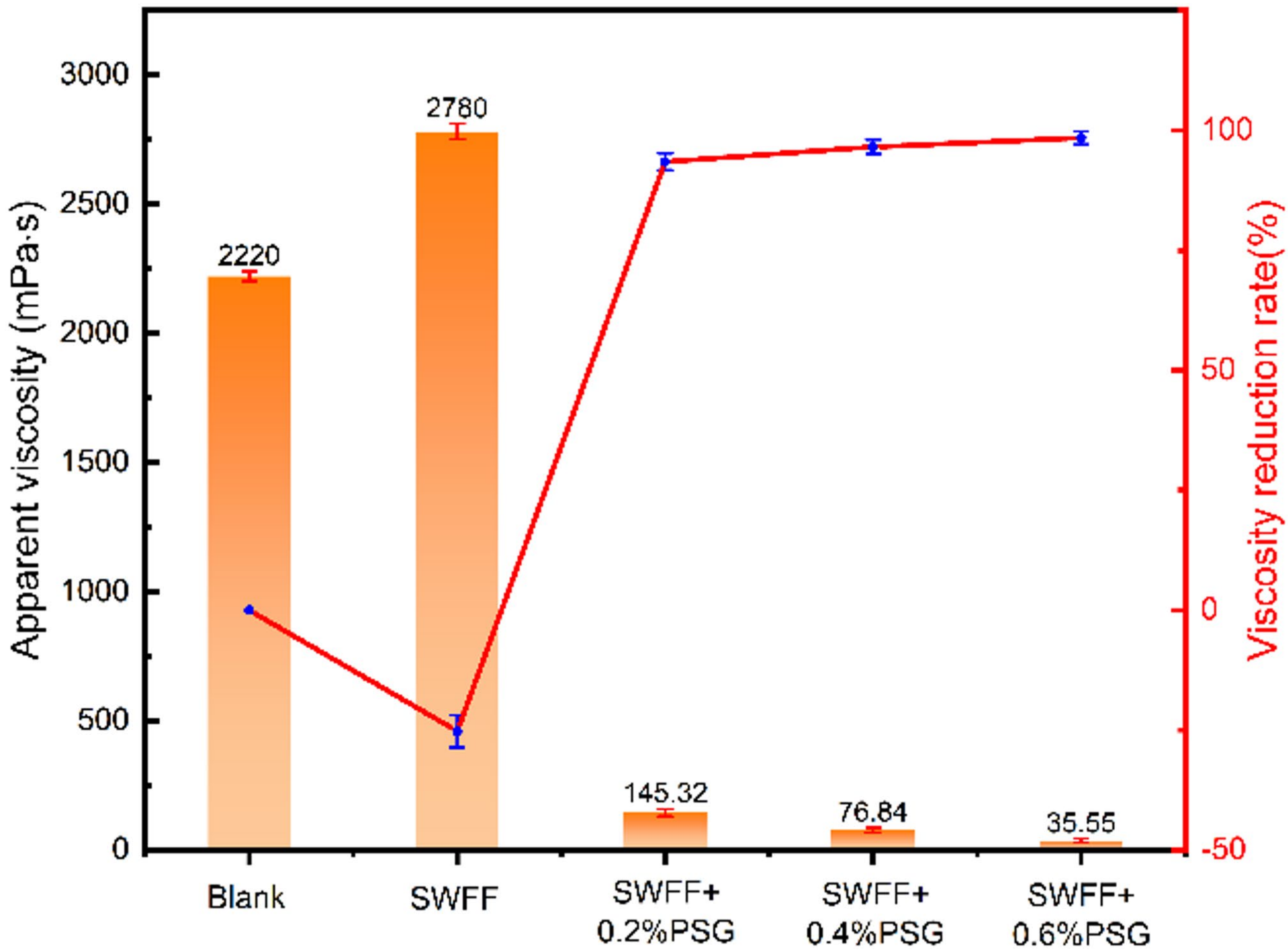

Fig. 5. Viscosity and viscosity reduction rate of shale oil after emulsifying and viscosity reduction.

angle (θ) decreases, the P_c decreases. Capillary pore size is an inherent characteristic of shale, whereas IFT and wettability can be modified by adding surfactants[23,44]. As shown in Fig. 9, the initial contact angle between shale core and field water is 73.8°, while the contact angle increases to 99.3° after soaking in GY734H shale oil for 24 h, indicating that the hydrophobicity of shale increases significantly. In contrast, the contact angle decreases to 50.8° after soaking in SWFF for 24 h, showing a water-wetting state, but the ability to change the wettability is limited. The contact angle is further reduced to 29.1° when 0.2% PSG is added to SWFF, showing a strong water-wetting state, reducing greatly the capillary resistance, which is more conducive to shale oil recovery by imbibition. Notably, when the concentration of PSG is further increased, the contact angle increases because the adsorption of PSG molecules on the shale surface reaches saturation. Therefore, SWFF + 0.2%PSG is the most effective functional fracturing fluid, and it can significantly improve shale wettability.

Static washing oil efficiency

In fact, adding surfactants to the fracturing fluid is a common field practice for enhanced oil recovery in low permeability reservoirs[40], while this approach has only just begun in shale reservoirs. The study outlined above demonstrates that this functional fracturing fluid system has the function of reducing IFT, emulsifying and viscosity reduction, and changing shale wettability. Nevertheless, the most critical criterion for evaluating this functional fracturing fluid system for practical application is its oil washing efficiency. As shown in Fig. 10, the oil-washing efficiency of the filed water is only 2.89%, while the SWFF increases the oil-washing efficiency to 11.65%, attributed to the presence of drag reducer components. After adding 0.2% PSG to SWFF, the oil washing efficiency increased to 20.66%, indicating a notable synergistic effect between SWFF and PSG. Because the IFT of the system is further reduced, and the shale wettability performance is improved, resulting in the shale surface turning into a strong water-wet state, thereby reducing the viscous resistance between the shale oil and the shale itself. However, it's worth noting that the amount of PSG added should not exceed an optimal level. The excessive PSG will self-aggregate in the SWFF, thereby reducing the efficiency of the oil washing. From the perspective of benefit and cost, the SWFF + 0.2%PSG system is the most suitable for the oilfield. Due to its excellent performance, it can not only reduce the frequency of re-fracturing but also reduce the development cost of the whole life cycle, which is both economic and technical promotion value.

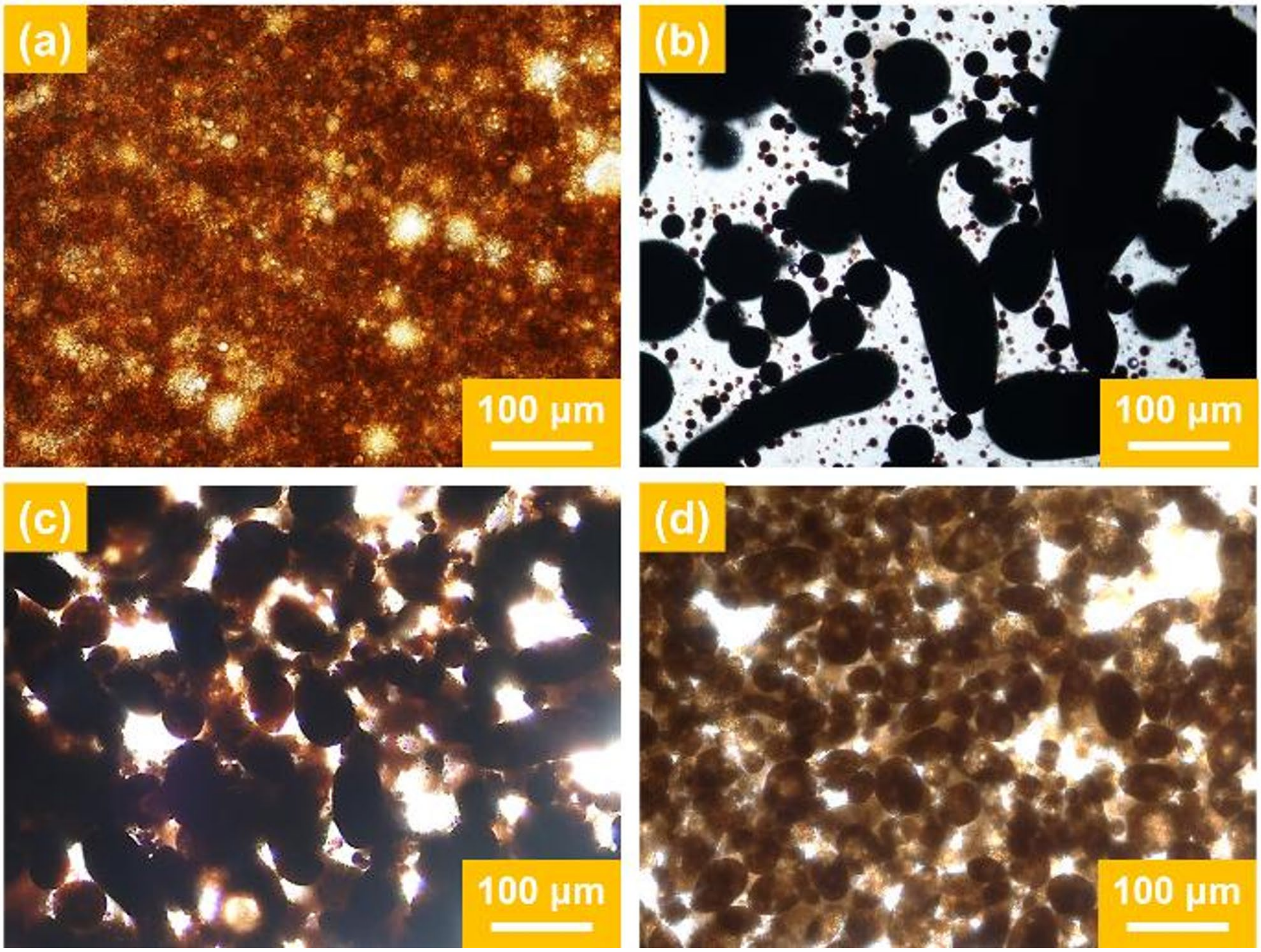

Fig. 6. Microscopic images of shale oil after emulsifying and viscosity reduction: (**a**) SWFF, (**b**) SWFF + 0.2%PSG, (**c**) SWFF + 0.4%PSG, and (**d**) SWFF + 0.6%PSG.

Proposed mechanism

Fracturing fluids play a key role in shale oil development technology. Conventional slickwater fracturing fluids have the advantages of increasing fracture conductivity and reducing reconstruction cost, but they have low oil washing efficiency. As shown in Fig. 11, the mechanism of the functional fracturing fluid system constructed in this work is proposed. By adding PSG surfactant to the SWFF system, it can reduce oil-water interfacial tension, emulsifying and viscosity reduction, change shale wettability, and improve oil washing efficiency. Compared with the existing systems in the literature[19–21], our system has more advantages in changing the interface properties. These characteristics make it potentially more applicable in enhancing the recovery rate of shale oil and the single well production.

Conclusion

This study demonstrates the successful development of a multifunctional fracturing fluid system by adding 0.2% PSG surfactant into conventional SWFF. The optimized SWFF + 0.2%PSG formulation exhibited robust compatibility, thermal stability (≤ 100 °C), and shear resistance (170 s^{-1} for 90 min), while maintaining dynamic IFT below 1.5 mN/m. The systematic evaluation revealed its dual functionality. For GY734H shale oil, the emulsification and viscosity reduction rate are higher than 93.45% by using the optimized SWFF + 0.2%PSG fracturing fluid system. Additionally, this system enhanced shale wettability modification, transitioning reservoir surfaces to strongly water-wet states (contact angle < 30°) with a static oil-washing efficiency of 20.66%, attributed to surfactant-mediated capillary force reversal. These synergistic effects position the system as a promising candidate for improving primary fracturing recovery in shale oil reservoirs. However, while laboratory-scale results validate technical feasibility, field-scale implementation requires further optimization of pumping protocols, long-term compatibility assessments, and economic viability analyses. Subsequent research should focus on pilot testing under simulated reservoir conditions to establish operational parameters for the cost-effective development of shale oil in the Bohai Bay Basin, located in East China. In a word, this work provides a methodological framework for developing smart fracturing fluids that integrate stimulation and enhanced oil recovery functions.

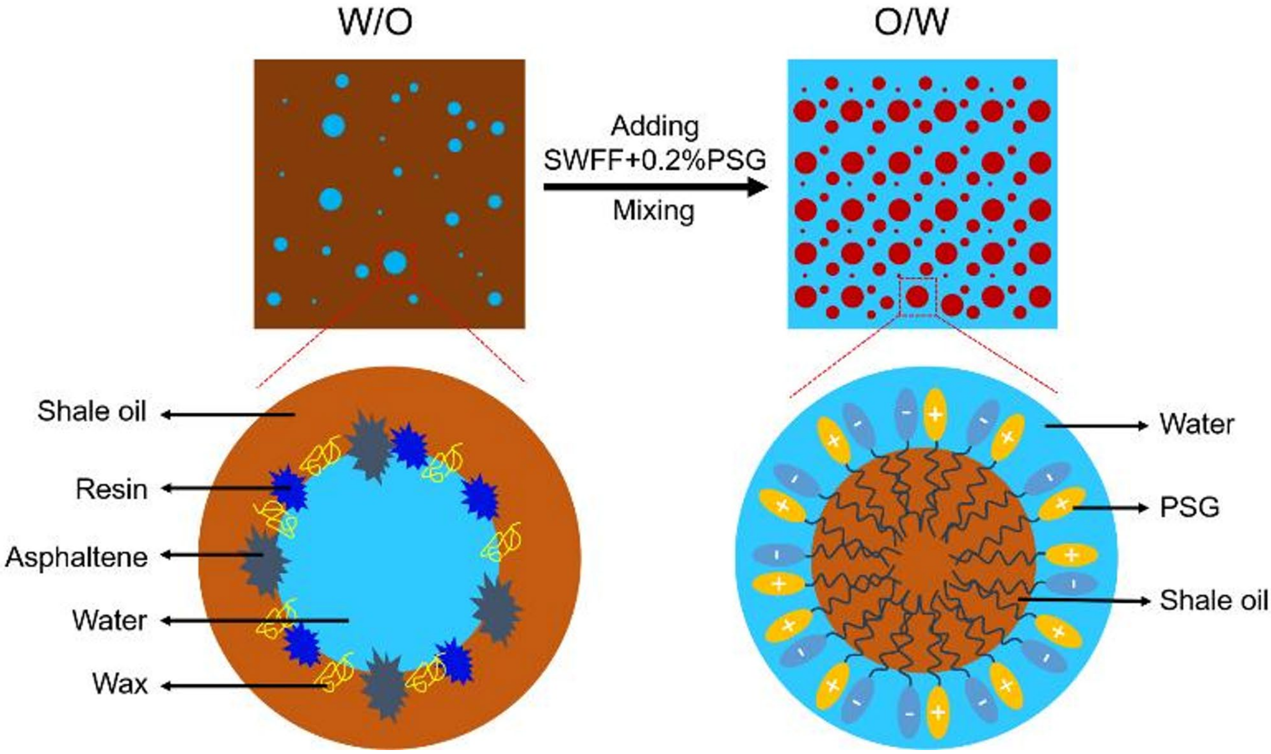

Fig. 7. Mechanism diagram of W/O transforming into O/W emulsion.

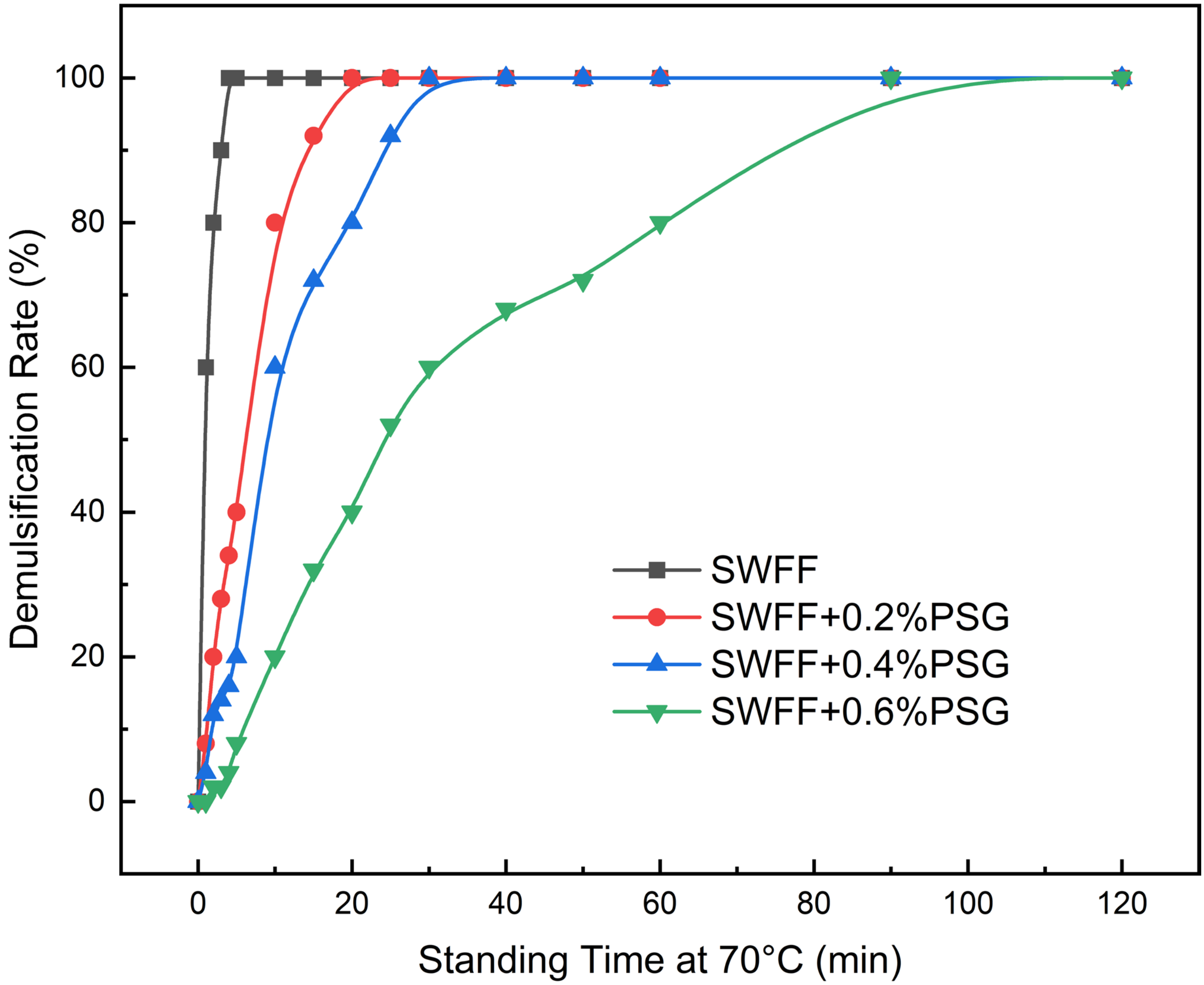

Fig. 8. Relationship between demulsification rate of shale oil emulsions after viscosity reduction and time at 70 °C.

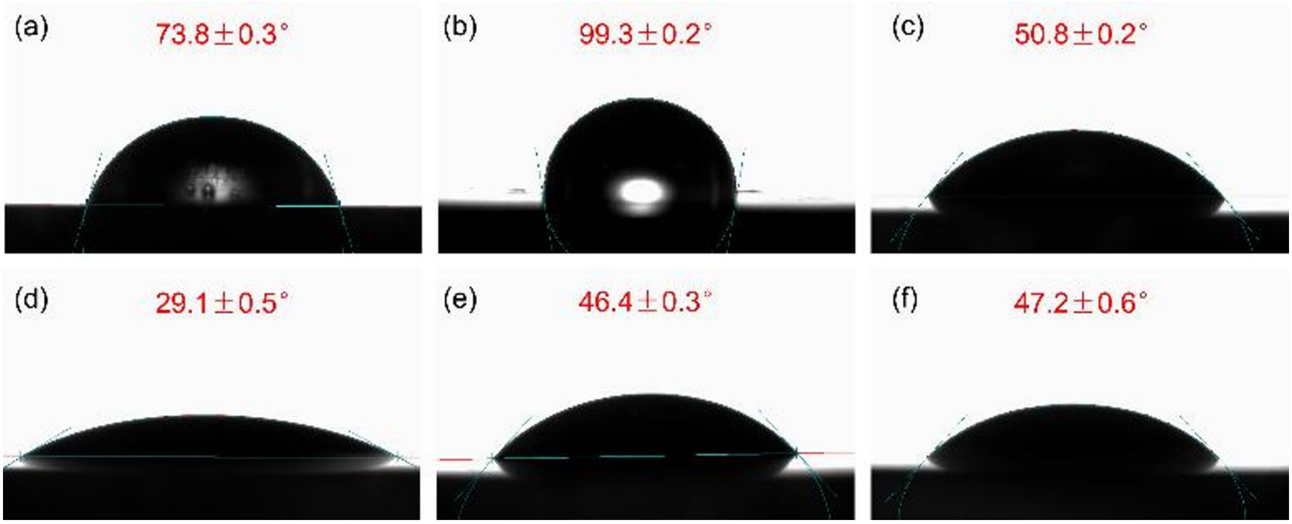

Fig. 9. Effect of fracturing fluid system on contact angle between shale and water: Shale core immersed in (**a**) field water, (**b**) GY734H shale oil, (**c**) SWFF, (**d**) SWFF + 0.2%PSG, (**e**) SWFF + 0.4%PSG, and (**f**) SWFF + 0.6%PSG for 24 h, respectively.

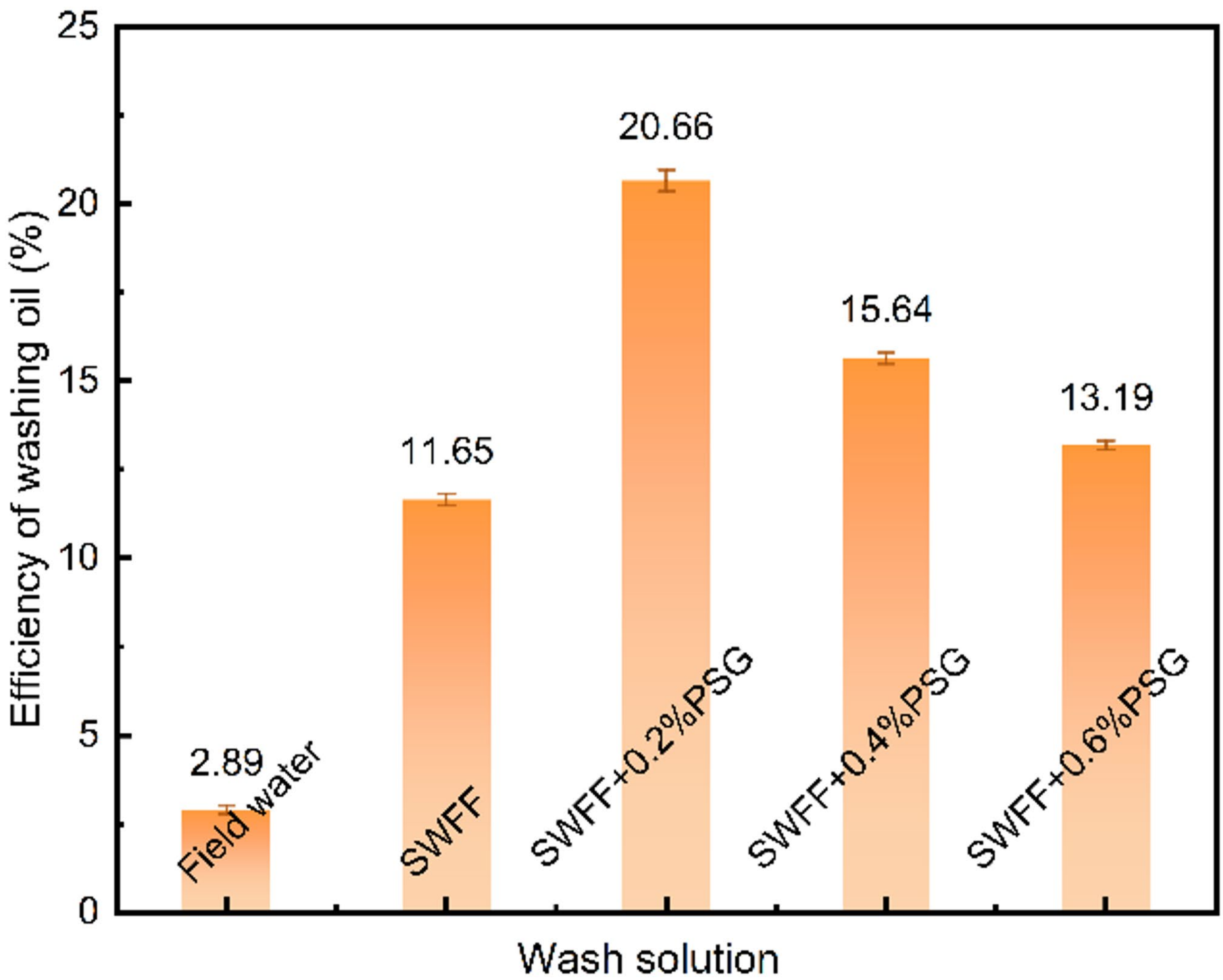

Fig. 10. Static oil washing efficiency of fracturing fluid system on GY734H oil sand at 80 °C.

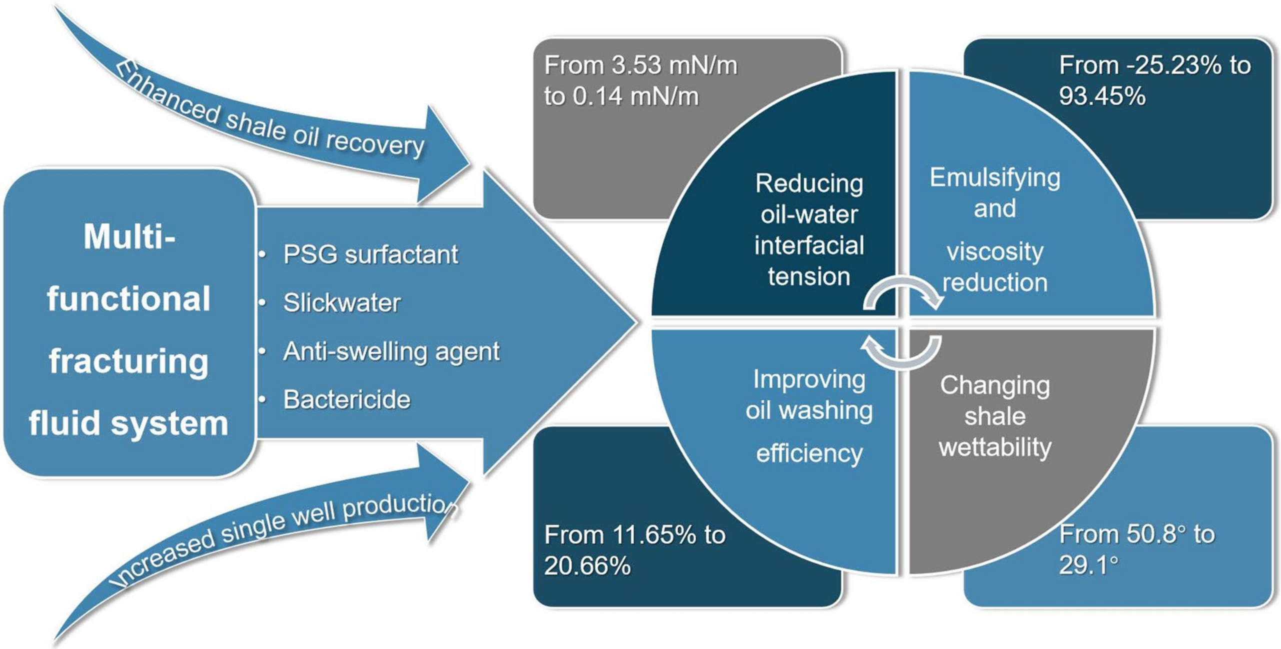

Fig. 11. Proposed mechanism of multifunctional fracturing fluid to enhance shale oil recovery.

Data availability

The authors declare that the data supporting the findings of this study are available within the paper and its Supplementary Information files. If any raw data files are needed in another format, they are available from the corresponding author upon reasonable request.

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Declarations

Competing interests
The authors declare no competing interests.

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