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Effects of fracturing fluid composition and other factors on improving the oil imbibition recovery of shale reservoir

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Abstract:
Imbibition is an important mechanism of shale reservoir development. In exploring the factors affecting the enhanced recovery of shale reservoirs by imbibition, laboratory spontaneous and forced imbibition experiments were conducted using outcrop cores of shale reservoirs. The effects of imbibition agent composition, fracture, and pressure on imbibition are obtained in this work based on imbibition recovery test findings and imbibition theory. Results show that the imbibition curve includes three stages, namely, imbibition, transition, and stability. Among the components of compound fracking fluid, surfactants have the greatest impact, whereas emulsifiers have the least impact. Complex crack structures and high-temperature environments can improve imbibition recovery. Pressure is inversely proportional to imbibition recovery in the highly stress-sensitive shale reservoir. In addition, the throughput time of the imbibition agent has an optimal value in the shale reservoir. After the huff-n-puff time exceeds the optimal value, the imbibition agent should be replaced to continuously improve the imbibition effect. The research results can serve as a basis for enhancing oil recovery through imbibition.

1. Introduction

Imbibition occurs in porous media and is an effective method for extracting fractured and tight reservoirs (Chakraborty et al., 2017; Andersen, 2019; Guo et al., 2020; Qu et al., 2022). Following hydraulic fracturing, infusing fluids into ultra-low permeability reservoirs like shale can clearly result in oil-water imbibition, which can improve oil recovery even further, according to recent studies (Kathel and Mohanty, 2013; Tokunaga et al., 2021; Zheng et al., 2021; Xu et al., 2022). Therefore, understanding the factors that affect imbibition has become a key focus, with the potential to further enhance recovery through imbibition mechanisms (Zaeri et al., 2018; Cai, 2021; Sakthivel and Elsayed, 2021).

There are many factors affecting imbibition, including reservoir physical properties, mineral composition, and boundary conditions (Graue and Fernø, 2011; Cai et al., 2014; Li et al., 2020). The three primary physical characteristics of a reservoir are wettability, permeability, and porosity, which have an immediate impact on the reservoir's permeability, storage capacity, and level of oil and gas adsorption on the rock surface (Yang et al., 2017; Lai et al., 2019; Liang et al., 2020; Dou et al., 2021). The pore structure, porosity, surface charge, and chemical reactions of a reservoir can all be impacted by the mineral makeup of the rocks that make up the reservoir (Yang et al., 2016, 2019; Cai et al., 2020; Liu et al., 2021). The adsorption behaviour of liquid or gas molecules on the

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Fig. 1. Imbibition bottle.

surface of reservoir rocks will be influenced by the boundary conditions. (Yildiz et al., 2006; Lyu et al., 2019; Meng et al., 2019; Li et al., 2022). The three elements mentioned above, however, are just the effects of the initial reservoir environment on the imbibition process. After volume fracturing of horizontal wells, the reservoir environment will undergo certain changes, and the injected fracturing fluid will also cause a series of reservoir changes. The impact of these factors on the permeability and suction effect still needs to be studied urgently (Guo et al., 2021b; Zhou et al., 2022).

There are two types of imbibition: spontaneous and forced (Hammond and Unsal, 2009; Wang et al., 2018; Cao et al., 2022). Currently, researchers examine imbibition's affecting elements mostly through spontaneous imbibition experiments, using forced imbibition to study less (Hamidpour et al., 2015; Gao and Hu, 2016; Gao et al., 2019). However, spontaneous and forced imbibition coexist during the oil displacement process, so it's equally important to research the components that influence the forced imbibition process.

In this study, the focus was on exploring the impact of factors such as composite fracturing fluid composition, fractures, temperature, surfactant concentration, pressure, and the huff-n-puff times of the imbibition agent in the imbibition process. To do so, the Jimsar shale reservoir in the Junggar Basin, Xinjiang, is chosen as the research subject. By employing a combination of static and dynamic imbibition experiments, the experimental results closely mimicked the actual imbibition process in the reservoir. By studying the influence of these factors on imbibition, this work can deeply understand the mechanism of imbibition, optimize imbibition oil recovery technology, improve recovery efficiency, and offer theoretical backing for shale oil and gas field development and exploration.

2. Materials and methods

2.1 Equipment and materials

Table 1. Core parameters.

No.	Length (cm)	Diameter (cm)	Permeability (mD)	Porosity (%)	Saturated oil (mL)
1	5.0175	2.5243	0.1412	12.3	0.3315
2	5.0036	2.5246	0.1423	12.5	0.3545
3	5.0144	2.5263	0.1515	13.1	0.3675
4	4.9741	2.5250	0.1463	12.9	0.3352
5	5.0033	2.5281	0.1329	11.5	0.3344
6	4.9833	2.5211	0.1503	13.2	0.1028
7	4.9712	2.5230	0.1511	13.5	0.3540
8	5.0021	2.5214	0.1368	11.3	0.3467
9	5.0103	2.5224	0.1489	12.9	0.3734
10	5.0174	2.5230	0.1478	12.6	0.2857
11	4.9913	2.5216	0.1522	13.4	0.3049
12	5.0110	2.5227	0.1468	12.3	0.3589
13	5.0127	2.5216	0.1539	13.6	0.3220
14	4.986	2.5223	0.1524	13.5	0.3057
15	5.0102	2.5221	0.1335	11.4	0.3439
16	8.0231	3.8102	0.1422	12.6	3.7060
17	7.9901	3.8113	0.1565	13.7	4.2984
18	7.9854	3.8105	0.1356	11.4	3.2691

The following is a list of the equipment used: Brookfield DV-III viscosimeter, Brookfield Company, USA; ME204E electronic balance, Mettler Toledo Instruments (Shanghai Co., Ltd.); TST101-1B electric thermostatic drying oven, Chengdu Tesite Instrument Co., Ltd; DF-101S type heat-collecting constant temperature heating magnetic stirrer, Gongyi Yuhua Instrument Co., Ltd; IKA RW20 digital display top mounted mechanical mixer, Shanghai Jiuxi Scientific Instrument Co., Ltd; DSA30S drop shape analyzer, KRUSS, Germany; TX-500C spinning droplet tensiometer, USA KINO Industry Co., Ltd; TY-2 core holder (it has a diameter of 2.5 cm), JB-3 manual metering pump and ZR-3 piston type high-pressure intermediate vessel (the volume is 100 ml, and the maximum withstand voltage is 32 mPa), Haian Core Petroleum Instrument Co., Ltd; pressure transducer (0-10 MPa), Omega Engineering, USA; 2FY-4C-N vacuum pump (the ultimate vacuum is 0.2 Pa), Zhejiang Value electromechanical Co., Ltd; ISCO 1000DX ram pump (the flowrate is 0.001-50 mL/min), Peian Co., Ltd; fabricated imbibition container in laboratory, the imbibition bottles shown in Fig. 1.

Chemicals and materials used are as follows: $(NH_4)_2S_2O_8$, SDBS, $CaCl_2$, $MgCl_2$, KCl, NaCl, Na_2CO_3, $NaHCO_3$, Na_2SO_4 (analytically pure, Chengdu Kelong Chemical Reagent Factory), and lotion fracturing fluid YTH-1 (Xinjiang Baoshu New Energy Technology Co., Ltd.); there are 18 shale reservoir cores in Jimsar Sag, and specific parameters are presented in Table 1, while core images are displayed in Fig. 2; the crude oil in the shale reservoir of Jimsar Sag has an apparent viscosity of 9.54 mPa·s at 90 °C, and the crude oil density is 0.89 g/cm^3; the particular content of the simulated

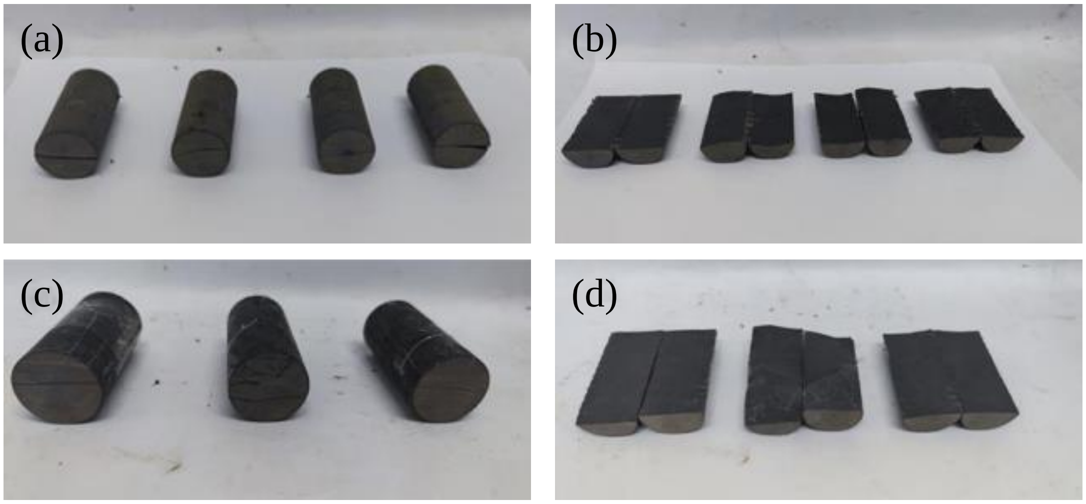

Fig. 2. Artificially fractured cores (the dimensions of (a) and (b) are 2.5 cm × 5 cm, and the dimensions of (c) and (d) are 3.8 cm × 8 cm).

Table 2. Composition of simulated brine.

Inorganic salt	$CaCl_2$	$MgCl_2$	KCl	NaCl	Na_2CO_3	$NaHCO_3$	Na_2SO_4	Total salinity
Concentration (g/L)	0.2349	0.0793	0.0385	3.8916	0.1498	7.1236	0.2254	1.1701

saline trial water is displayed in Table 2.

2.2 Methods

2.2.1 Spontaneous imbibition

With the core size of 2.5 cm × 5 cm as the material and the imbibition bottle as the experimental instrument, the preparation work is first carried out: i) Artificially fracturing the outcrop shale core and recording the weight (m_1); ii) Next, the oil was aged for a month at 90 °C after being saturated (returning the core to its original state), and the wet weight (m_2) was recorded before the imbibition experiment. In Fig. 3, the experimental procedure is displayed. The density of crude oil is 0.89 g/cm^3, and the volume, imbibition recovery rate, and imbibition efficiency of saturated crude oil can be computed by:

$$V = \frac{(m_2 - m_1)}{\rho} \tag{1}$$

$$R = \frac{(V_i - V_{i-1})}{V} \tag{2}$$

$$A = \frac{(R_i - R_{i-1})}{R} \tag{3}$$

where V is the amount of crude oil that has the core saturated, m_1 and m_2 are respectively the weights of the core before and after saturation with crude oil, ρ is the experimental crude oil's density, R is the final recovery rate, V_i and $V_{i\text{-}1}$ are respectively the volumes of crude oil extracted at time i and i-1, A is the imbibition efficiency, R_i and R_{i-1} are respectively the crude oil recovery rates at time i and i-1.

1) Composition of composite fracturing fluid
Four solutions of YTH-1 emulsion (0.9 wt%), $(NH_4)_2S_2O_8$ (0.08 wt%), SDBS (0.2 wt%), and a composite system were configured and aged at 90 °C until the fracturing fluid broke. Then add the solution to four imbibition bottles, using one bottle filled solely with saline water. Subsequently, immerse the five cores in the five imbibition bottles. At last, the bottle was heated to 90 °C in an oven. The effects of each composition on imbibition were explored.
2) Surfactant concentration
With SDBS concentrations of 0.05 wt%, 0.1 wt%, 0.15 wt%, and 0.3 wt%, four different kinds of composite fracturing fluids were made. These four solutions were placed in an oven and heated to 90 °C. The upper clear solution was collected after 24 hours. Subsequently, the extracted solution was poured into four imbibition bottles, and the core was immersed in the liquid before placing it together with the imbibition bottles in a 90 °C oven. The effect of different surfactant dosages (different interface tensions and hydrophilic wetting levels) on imbibition was explored.
3) Work of adhesion
i) The 5 cores were sliced and polished, then soaked in crude oil and aged at 90 °C for a week;
ii) Fracturing fluids with different SDBS contents (0.05 wt%, 0.1 wt%, 0.15 wt%, 0.2 wt%, and 0.3 wt%) were configured and aged at 90 °C for 24 hours;
iii) Conduct an interfacial tension test between water and oil using the solution set up in step ii);
iv) Using the barb approach, the water-solid interface contact angle of the oil droplets on the initial five core

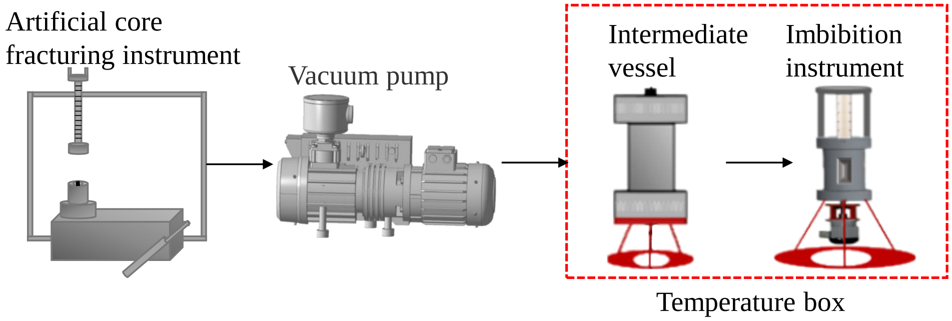

Fig. 3. Spontaneous imbibition flow chart.

slices was measured;

v) Put the core slices into the liquid specified in step ii) and soak them at 90 °C for 2 weeks (surfactant adsorption equilibrium);

vi) For every core slice, measure the contact angle at the oil droplets' water-solid interface. Then, compute and contrast the adhesion work done before and after the glue-breaking solution is soaked. The adhesion work can be computed by:

$$W = \sigma_{ow}(1-\cos\theta) \tag{4}$$

where W is the adhesion work, σ_{ow} is the interface tension of crude oil-water, θ is the size of the contact angle.

4) Temperature
The upper-layer clear fluid was recovered after ageing at 90 °C for 24 hours using the composite system fracturing fluid (SDBS 0.2 wt%). Subsequently, the solution was poured into each of the five imbibition bottles, and the core was submerged. The bottles were placed in ovens and heated at 20, 40, 60, 80, and 90 °C. The effects of different temperatures on imbibition were explored.

5) Core cracks
The upper-layer clear fluid was recovered after ageing at 90 °C for 24 hours using the composite system fracturing fluid (SDBS 0.2 wt%). Two distinct imbibition bottles were then filled with the solution. Finally, the fractured and unfractured cores were immersed. The imbibition bottles were placed in a 90 °C oven. The impact of cracks on permeability before and after fracturing (different specific surface areas) was explored.

2.2.2 Forced imbibition

The core with a size of 3.8 cm × 8 cm is used as the material, and the core holder of the oil displacement device is used as the experimental instrument. The following are specific experimental steps:

i) An artificial fracture was made on the natural outcrop core.

ii) Vacuum was pumped on the core, and the saturated saline was pressed (porosity is recorded).

iii) Confining pressure is exerted after the core is inserted into a core holder. Saturated oil was displaced at 90 °C (the volume of saturated oil was recorded) and aged for 48 hours (the core was restored to the original state of the reservoir).

iv) A composite fracturing fluid was prepared, and after ageing at 90 °C for 24 hours, the upper-layer clear liquid was collected and poured into a high-pressure intermediate vessel.

v) Saline flooding was performed on the saturated oil core in a 90 °C oven. After the water cut reached 98%, the saline drive was stopped, and heat preservation was maintained for 24 hours.

vi) A 0.5 PV fracturing fluid gel breaker was continuously used for displacement.

vii) For a duration of 28 hours, closed-well imbibition and oil recovery were carried out with the core holder's outlet valve closed and the injection pressure of the core maintained.

viii) Saline displacement was continuously carried out, and a calculation of the imbibition oil recovery rate was made. The displacement flow rate is 0.01 mL/min. In Fig. 4, the experimental procedure is depicted.

1) Confining pressure
The influence of different confining pressures (a stress-sensitive process) on imbibition was tested. Change the confining pressure in step iii) above. Perform dynamic imbibition oil recovery under confining pressures of 8, 16, and 24 MPa to test the final imbibition oil recovery.

2) Number of times the imbibition agent huffs-n-puffs
The effects of different huff and puff times on imbibition were tested. Above steps vi), vii), and viii) were repeated to compare the final recovery rate after 1, 2, and 3 huff-n-puff imbibition times.

3. Results and discussion

3.1 Spontaneous imbibition

3.1.1 Composition

The complex system consists of an integrated emulsion fracturing fluid, including YTH-1, $(NH_4)_2S_2O_8$, and SDBS. The imbibition results of YTH-1 emulsion (0.9 wt%), $(NH_4)_2S_2O_8$ (0.08 wt%), SDBS (0.2 wt%), and composite systems were studied at 90 °C using Jimsar shale outcrop cores. Three sections make up the imbibition curve: the imbibition stage (imbibition efficiency increasing rapidly), the transition stage (imbibition efficiency decreasing gradually), and the steady stage (imbibition saturation and imbibition reaching equilibrium). Due to its capacity to alter wettability

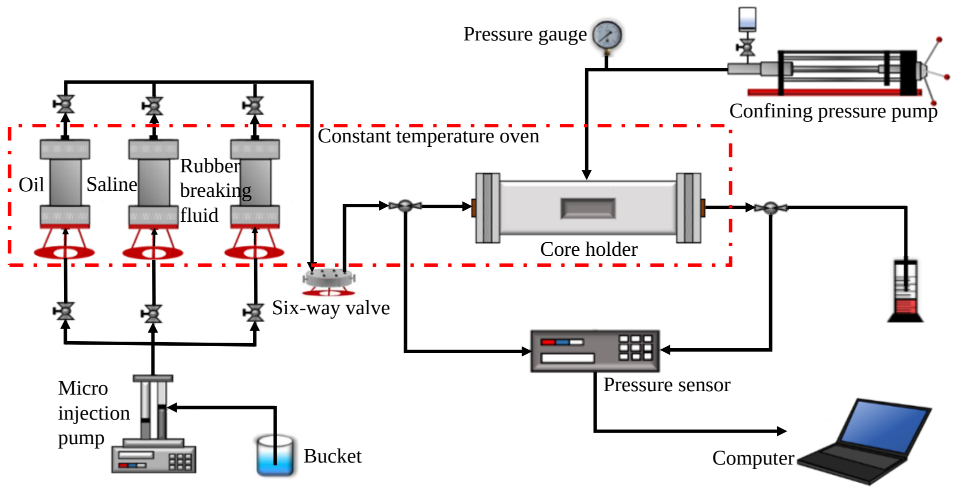

Fig. 4. Dynamic imbibition flow chart.

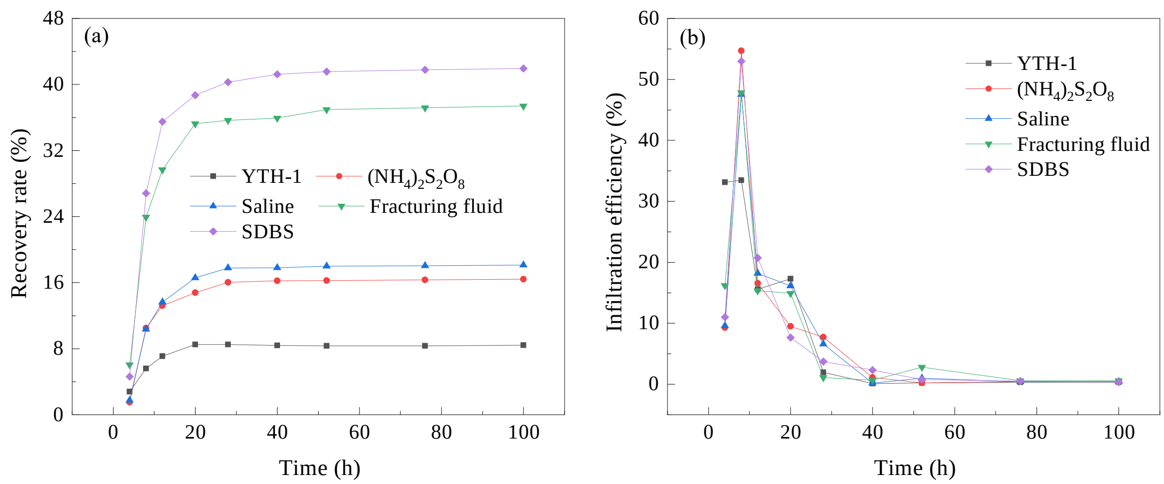

Fig. 5. Imbibition results of Saline, YTH-1, $(NH_4)_2S_2O_8$, SDBS and Fracturing fluid; (a) imbibition recovery and (b) imbibition efficiency.

and diminish interfacial tension, the anionic surfactant SDBS has a maximum absorption efficiency of 41.77%, as illustrated in Fig. 5(a). The imbibition recovery of a compound fracturing fluid with SDBS is 37.38%, and the imbibition recovery of $(NH_4)_2S_2O_8$ (16.325%) is close to that of brine (18.1%). In addition, the YTH-1 solution still has a certain viscosity (small molecular chain lengths), which results in the lowest oil recovery (only 8.463%).

3.1.2 Concentration

Anionic surfactant is more surfactant than the other three (Liu et al., 2019; Souayeh et al., 2021; Hou and Sheng, 2022). Concentration determines how much anionic surfactant SDBS can alter the interfacial tension between oil and water and the wettability of rocks. However, increasing oil recovery through imbibition is significantly impacted by wettability and interfacial tension. Further research is therefore necessary to determine how surfactant concentration affects imbibition.

As Fig. 6(a) illustrates, the imbibition effect increases with surfactant SDBS content. The oil-water interface steadily diminished as the concentration of SDBS increased, and the oil-wet surface eventually transformed into a water-wet surface. Therefore, enhancing the flow of crude oil will increase the capillary force and imbibition power, thereby producing a better imbibition effect. The imbibition power and peak value of imbibition efficiency increase with surfactant content, as Fig. 6(b) illustrates.

There is a thermodynamic transition process (adhesion work) in the process of oil droplet removal, and with the process of oil droplet removal, the contact competition gradually decreases and the adhesion work gradually decreases. Consequently, the more adhesion work reduction there is, the more oil is stripped off, and the more robust the capacity to improve oil recovery.

Utilizing the barb approach as illustrated in Fig. 7, determine the oil's contact angle at the water-solid interface. With the increase in SDBS concentration, the contact angle of oil droplets decreases gradually. After the critical micelle concen-

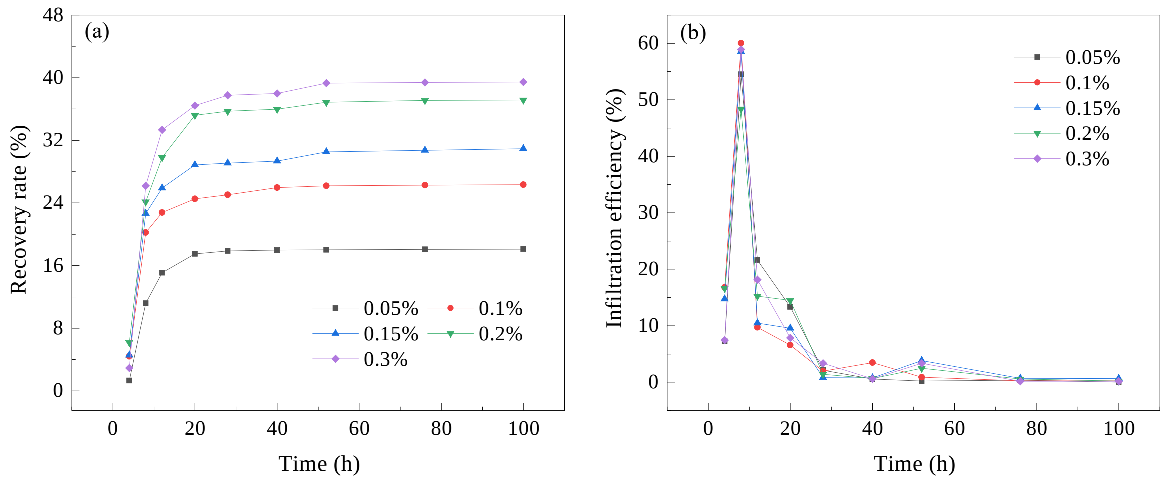

Fig. 6. Imbibition results at different surfactant concentrations; (a) imbibition recovery and (b) imbibition efficiency.

Table 3. Calculation results of adhesion work with different SDBS content.

Content (%)	Interface tension (mN/m)	Contact angle (°)	Work of adhesion (mJ)	Recovery efficiency
Initial state	9.14	117.77	13.3983	
0.05	0.854	101.43	1.0232	18.11
0.1	0.226	87.25	0.2151	26.39
0.15	0.084	66.92	0.0511	30.89
0.2	0.096	43.05	0.0258	37.38
0.3	0.084	46.69	0.0264	39.25

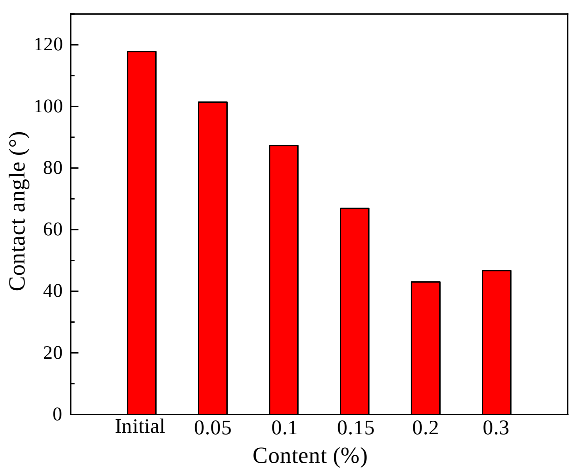

Fig. 7. Spontaneous imbibition flow chart.

Table 4. Relationship between temperature and viscosity of shale oil.

Temperature (°C)	Viscosity (mPa·s)
20	72.72
40	52.88
60	38.51
80	15.63
90	9.54

tration is reached, the ability to change wettability begins to level off. Table 3 lists the adhesion work calculated by Eq. (4). 0.2 wt% SDBS can greatly reduce the adhesion work, and the ultimate adhesion work is 0.0258 mJ, and the final imbibition recovery rate is 37.38%. In conclusion, adhesion work decreases and imbibition's potential to improve oil recovery increases with reservoir wettability's hydrophilicity. There exists an ideal concentration for the surfactant concentration; a higher concentration is not always preferable.

3.1.3 Temperature

Temperature affects many characteristics of reservoirs and fluids, such as capillary contraction (the temperature effect). When the temperature rises, the capillary force and imbibition power increase (Feng et al., 2019; Guo et al., 2021a). Furthermore, it is also easier to activate crude oil and convert an oil-wet reservoir to a water-wet one at higher temperatures. With the increase in temperature, the fracturing fluid can break the gel more easily, which increases the imbibition rate. Furthermore, crude oil's viscosity reduces, viscous resistance diminishes, and fluidity improves as temperature rises. Therefore, studying the effects of different temperatures on imbibition recovery and efficiency is necessary. Table 4 shows the change in shale oil viscosity with temperature.

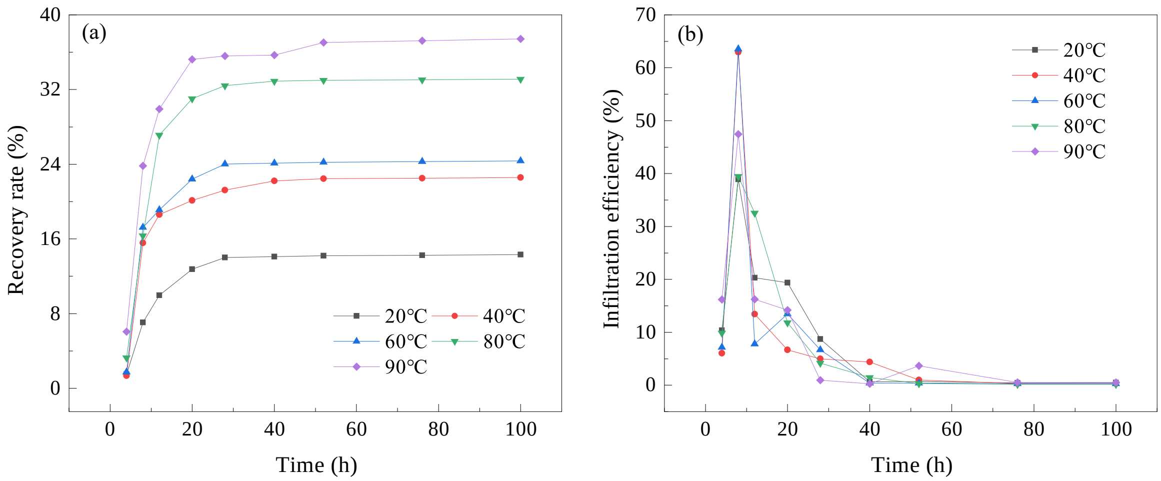

Fig. 8. Imbibition results at different temperatures; (a) imbibition recovery and (b) imbibition efficiency.

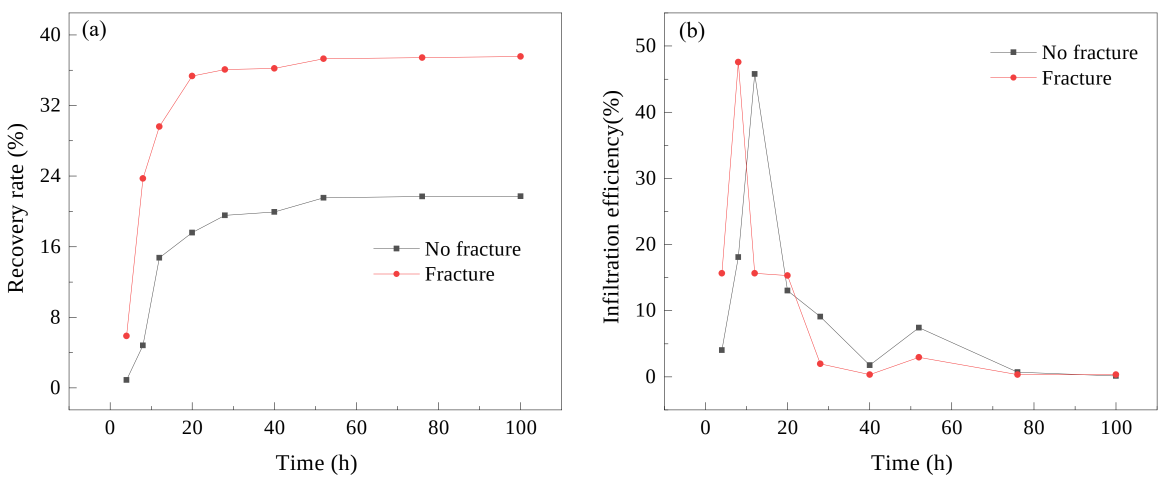

Fig. 9. Imbibition results of fractured and unfractured cores; (a) imbibition recovery and (b) imbibition efficiency.

The viscosity of crude oil drops from 62.72 to 9.54 mPa·s as the temperature rises from 20 to 90 °C, and there is an improvement in oil recovery from 5.98% to 37.38%. The imbibition recovery rises as the temperature rises, as Fig. 8(a) illustrates. As shown in Fig. 8(b), the peak value of imbibition efficiency is lower at low temperatures, and the decrease in imbibition efficiency is slower at the transition stage in imbibition. This outcome is caused by the fact that the capillary force and primary force for gravity absorption become less at lower temperatures, which slows down absorption efficiency.

3.1.4 Cracks

After the shale reservoir is fractured, it is divided into matrix and cracks to compare the imbibition characteristics of matrix and matrix crack systems. The imbibition experiment of composite fracturing fluid between the fractured core and unfractured core was conducted at 90 °C. The fractured core's permeability recovery is 37.38%, as seen in Fig. 9(a). The permeability recovery of the unfractured core is 21.4%. When compared to the unfractured core, the imbibition recovery is about twice as high in the fractured core due to its huge specific surface area of imbibition. As shown in Fig. 9(b), the time period of the best imbibition efficiency of the fractured core is advanced because the large specific surface area of imbibition after fracture causes rapid changes in core wettability; therefore, the peak value and time interval of absorption efficiency are improved. Therefore, the imbibition efficiency and ultimate recovery factor can be improved by reservoir reconstruction, complex crack network structure, and good imbibition.

3.2 Forced imbibition

3.2.1 Confining pressure

There are two main categories of imbibition: forced and spontaneous. Forced imbibition is a conventional method for actual reservoir imbibition development. Most shale oil reservoirs are highly stress sensitive (Cui et al., 2018; Zhang et al., 2018; Jing et al., 2019; Wang et al., 2019); thus, exploring the effect of changes in effective stress (changing confining pressure) on imbibition recovery is necessary.

The confining pressures of cores 1, 2, and 3 are 8, 16, and

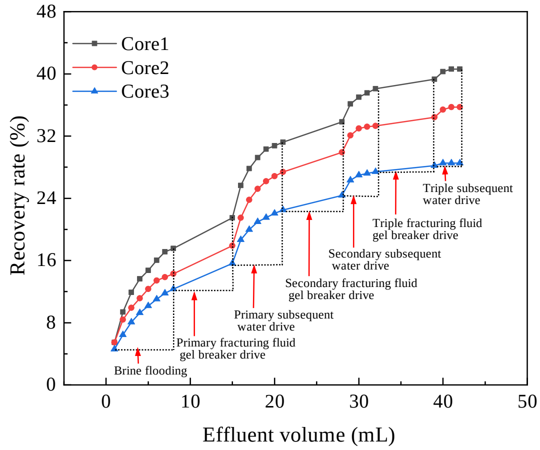

Fig. 10. Effect of different confining pressures on dynamic imbibition.

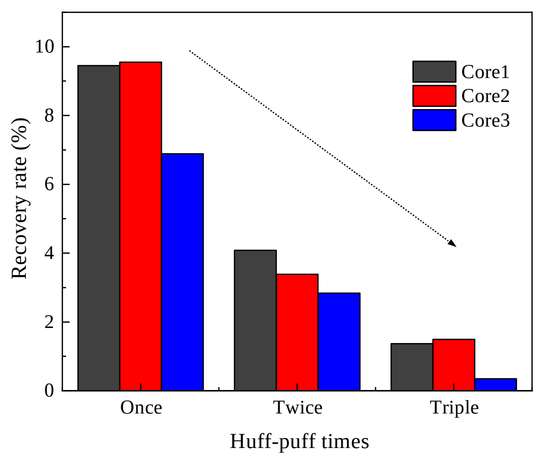

Fig. 11. Effect of different confining pressures on dynamic imbibition.

24 MPa, respectively. The water drive recovery and subsequent water drive recovery following the imbibition of the closed well decrease as confining pressure increases, as Fig. 10 illustrates. Furthermore, when confining pressure increases, the core's permeability reduces significantly, the crack closes, the fluid flow space gradually shrinks, the specific surface area shrinks, and the recovery from imbibition and water drive declines.

3.2.2 Times

The imbibition recovery factor with different times of imbibition is important for fracturing reservoir reconstruction and imbibition agent replacement. The comparison of imbibition recovery with different times of throughput is shown in Fig. 11. Oil recovery gradually declines as imbibition times rise. The throughput's ability to improve oil recovery has decreased to less than 2% at the third huff-puff. Furthermore, puff-puff imbibition times have a significant impact on the low porosity and low permeability core. After the third huff-puff, almost no increase in oil recovery is observed. Consequently, two times of huff-n-puff is the ideal amount of time for the YTH-1+SDBS compound fracturing fluid. To improve shale oil recovery, the imbibition agent needs to be changed after the second huff-n-puff.

4. Conclusion

In order to investigate the impact of temperature, confining pressure, and fracturing fluid composition on increased oil recovery by imbibition, a number of forced and spontaneous imbibition experiments were carried out in the current study. The following are the study's primary findings:

1) Imbibition curve analysis: The imbibition process has been shown to be separated into three stages: imbibition (imbibition efficiency increases rapidly), transition (imbibition efficiency decreases gradually), and stability (water absorption saturation, imbibition equilibrium). And the imbibition efficiency increases rapidly first and then decreases gradually, with an optimal imbibition time period.
2) Fracturing fluid composition comparison: The enhanced oil recovery ability of fracturing fluid components is shown as follows: SDBS > fracturing fluid > saline > $(NH_4)_2S_2O_8$ > YTH-1. This helps determine the most effective fracturing fluid composition and provides guidance for fracturing fluid proportioning.
3) Effects of temperature and cracking: Crude oil has a decreasing viscosity as temperature rises. I As oil viscosity rises, enhanced oil recovery through imbibition diminishes, and imbibition efficiency declines as water saturation rises. Moreover, reservoir reconstruction and complex fracture networks are helpful to improve imbibition efficiency and recovery efficiency.
4) Effect of effective stress: Pressure increases cause the crack to close, the core permeability to significantly drop, the fluid flow space to gradually contract, and the specific surface area to decrease. Furthermore, water drive recovery and dynamic imbibition recovery have decreased.
5) The imbibition agent's best number of huff-n-puffs: The best number of huff-n-puffs is two times; not the more times, the better the effect.

In conclusion, the study offers insightful information on the variables influencing imbibition and increased oil recovery and offers recommendations for the choice of fracturing fluids and operating parameters in the development of shale reservoirs.

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