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Synthesis and evaluation of a novel anti-water blocking agent for low-permeability reservoir

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) was prepared using double-hydrogen-terminated silicone oil (DHSO), allyl glycidyl ether (AGE), and triethylamine hydrochloride (TH) as raw materials. The structure was characterized by Fourier transform infrared spectroscopy (FTIR). The effect of the anti-water blocking agent on damage of water blocking of the brine solution in core pores was studied. The results showed that the anti-water-blocking agent significantly reduced the surface tension of the brine solution (less than 15 mN/m), and increased the water contact angle on the core surface to 85.3°, indicating that the anti-water blocking agent can transform the core surface from hydrophilicity to neutral and effectively reduce the viscous force of the water phase as it moves through the pores of the core. In addition, the anti-water blocking agent reduced the self-absorption and water blocking damage of the core, and improved the recovery rate of core permeability. A reasonable explanation of the results is that DAT-1 molecules are stably adsorbed in the sandstone core by the electrostatic force, the nonpolar segments are exposed to the sandstone surface to form a strong hydrophobic molecule layer, thereby changing in the wettability, and effectively reducing the invasion of water into the formation during drilling and completion operations. This is of great importance to the protection of the productivity of a low permeability reservoir.

Keywords: Low-permeability reservoir; Anti-water blocking agent; Contact angle; Surface tension; Water blocking damage; Reservoir protection

Синтез і оцінка нового антиводного блокуючого агента для низькопроникного пласта. *Сін-Бін Чжао, Янь-фей Лі, Чжао Хуан, Мін-бяо Сюй, Тао Ван, Да-лун Фен, Вей Хуан*

Отримані і досліджені властивості нового екологічно чистого засіба (DAT-1), що блокує воду, використовуючи силіконову олію з подвійним воднем (DHSO), аллілгліцидиловий ефір (AGE) і триетиламін гідрохлорид (TH) як сировину. Структуру охарактеризували інфрачервоною спектроскопією з перетворенням Фур'є (FTIR). Досліджено вплив антиводоблокуючого агента на пошкодження блокуванню води розсолу в порах керна. Результати показали, що агент, що запобігає блокуванню води, значно зменшив поверхневий натяг розсолу (менше 15 мН/м) і збільшив кут контакту з водою на поверхні ядра до 85,3°, що вказує на те, що блокування води агент може перетворити поверхню ядра з гідрофільної на нейтральну та ефективно зменшити силу в'язкості водної фази, коли вона рухається через пори ядра. Крім того, засіб, що блокує воду, зменшує самопоглинання та пошкодження серцевини, а також покращує швидкість відновлення проникності серцевини. Розумним поясненням результатів є те, що молекули DAT-1 стабільно адсорбуються в ядрі пісковику під дією електростатичної сили, а неполярні сегменти піддаються впливу поверхні пісковику, утворюючи міцний гідрофобний шар молекул, таким чином змінюючи змочуваність і ефективно зменшуючи проникнення води в пласт під час операцій буріння та завершення. Це має велике значення для захисту продуктивності пласта з низькою проникністю.

1 Introduction

China's low-permeability reservoirs contain a large amount of oil and gas, accounting for nearly 30% of the total oil and gas resources. However, its reserve abundance and formation pressure are low [1]. It is of strategic significance for the development of China's energy industry to improve the development efficiency of oil and gas resources in low-permeability reservoirs. Low-permeability reservoirs are characterized by poor physical properties and small pore throats, which can easily cause high resistance to oil and gas flow [2-3]. Additionally, low-permeability reservoirs are less resistant to damage from external fluids. Due to hydrophilicity of the pore throats, the external fluids are sucked into the pore throats via capillary force, causing water blocking damage and significantly reducing the relative permeability of the reservoir, which can seriously affect the productivity of low-permeability reservoirs [4-5]. Currently, there are various methods to overcome water blocking damage, including surfactants, acidification, fracturing, heating reservoir, and extending shut-in time [6]. Acidification and fracturing can alleviate water blocking damage by changing the shape of the channels. If the damage originates from external fluids, the effect of alleviating water blocking damage will be greatly reduced. In addition, procedures of acidification and hydraulic fracturing are complex and need longer well completion time [7-8]. Due to high energy consumption, providing heat to the reservoirs to mitigate water blocking damage is limited. Extending shut-in time increases the pressure gradient in the formation, thereby weakening water blocking damage. However, this method wastes a lot of time during the completion process. In summary, injecting appropriate chemical agents into the reservoir in the form of solution is an ideal way to alleviate water blocking damage by reducing capillary force in small pores and changing the wettability of core pores, which can increase core permeability [9]. At present, the common way to overcome water blocking damage is to add anti-water blocking agents to reduce the flowback pressure and surface tension of the water phase. Commonly used anti-water-blocking agents mainly include surfactants, microemulsions, organic alcohols, among which surfactants and organic alcohols are the most widely used in alleviating water blocking damage [10–11]. Alcohols that can lower the surface tension of water phase are prone to evaporation under high-temperature conditions, thereby accelerating the evaporation rate of water [12].

Fluorocarbon surfactant, due to its stable structure and strong surface activity, is widely used to reduce water blocking damage; it can reduce the intrusion of liquid phase in pore throats by reducing the surface tension of liquid phase and changing the wettability of cores. In [13] small-molecule of anti-water blocking agent was synthesized using perfluorooctanoic acid, thionyl chloride, sodium chloroacetate and N-Propylamine as raw materials. The above anti-water blocking agent can significantly change the shale wettability and alter the contact angles of water on the shale surface from 36° to 119°. A reasonable explanation of this phenomenon was that when the polar segments of the anti-water blocking agent were adsorbed in the shale by the electrostatic force, the nonpolar segments were exposed to the shale surface to form a strong hydrophobic molecule layer, thereby changing in the wettability of the shale surface. Tang et al. [14] studied the property of wettability alteration of a fluorochemical polymer as an anti-water blocking agent. The wettability of Berea sandstone before and after the above chemical treatment was studied at various temperatures from 25° to 93°. Application of a fluorochemical polymer as an anti-water blocking agent has revealed the possibility of its use in fields to improve the productivity and injectivity of wells by changing the wettability of rocks around the wellbore in formations from highly hydrophilic to neutral. Article [15] reported that the wettability of Berea and reservoir rocks can be permanently altered after treatment with the fluoropolymer surfactant at high temperature, and wettability alteration does not have a measurable effect on the absolute permeability of the rock. However, the fluorocarbon surfactant has many drawbacks such as difficulty in synthesis, multiple side reactions, and easy environmental pollution during degradation processes [16]. Obviously, these disadvantages limited the research and application of fluorocarbon surfactants. Therefore, developing alternatives to fluorocarbon surfactants is an important research direction for anti-water blocking agents. To develop a non-fluorine surfactant as an anti-water blocking agent, the environmentally friendly surfactant has been studied to meet the requirements of oil and gas production in the field. In [17] a novel, environmentally friendly anti-water blocking agent was developed and evaluated;

it was prepared using dimethyl silicone oil and octadecyltrimethylammonium chloride as raw materials. It was found experimentally that the surface tension of water solution with the anti-water blocking agent was reduced to 24 mN/m; this indicated that the capillary force of the water solution still existed in a low permeability reservoir. A reasonable explanation of the result was that the prepared molecule contained the structure of one-quaternary ammonium salt, which did not adsorb very stably on the rock surface. On this basis, the molecule prepared in this paper contained a double-quaternary ammonium salt structure, enhanced its adsorption ability on the sandstone surface. A novel, environmentally friendly anti-water blocking agent suitable for low-permeability reservoirs was developed, and its anti-water blocking performance and mechanism were studied.

2. Materials and methods

2.1 Materials

Double-ended hydrogen containing silicone oil (DHSO), allyl glycidyl ether (AGE), triethylamine hydrochloride (TH), isopropanol, anhydrous ethanol, and Karstedt's catalyst were all of analytical purity and supplied from Sinopharm Chemical Reagent Co., Ltd. Industrial grade NaCl was supplied by the Nanjing Chemical Reagent Co., Ltd. Field sandstones (gas permeability: 0.5-2.0 mD) were obtained from CNOOC China Ltd. Glass tubes (radius: 0.5 mm) were supplied by Shanxi Longxiang High Tech Glass Products Co., Ltd.

2.2 Preparation and structural characterization of the anti-water blocking agent

Based on addition reaction, double-ended hydrogen containing silicone oil (30 g) and allyl glycidyl ether (20 g) were dissolved with 200 mL isopropanol solution followed by passing through N_2 for 30 min. After that, Karstedt's catalyst (1g) was added to the mixture with continued stirring. The mixture was then refluxed and reacted at 70 °C for 6 h. After the reaction, the mixture was purified by distillation under reduced pressure to remove the isopropanol solution. A yellow reaction product was obtained, which was the intermediate.

Based on the epoxy ring-opening reaction, the intermediate (30 g) and triethylamine hydrochloride (10 g) were dissolved with 100 mL the anhydrous ethanol solution. Under continued stirring, the mixture was then refluxed and reacted for 5 h at 70 °C. After the reaction, the mixture was purified by pressure-reduced distillation to remove the anhydrous ethanol solution. Finally, a cationic organic silicon surfactant (DAT-1) was obtained.

FT-IR spectra of the purified and dried products (DAT-1) were measured with a WOF-520 Fourier Transform Infrared spectrometer (Beijing Jingke Ruida Technology Co, Ltd., Beijing, China) by the reflection method testing. The molecular structure of DAT-1 was characterized in the range of 500 ~ 4000 cm^{-1} to determine the functional group structure.

2.3 Performance evaluation of an anti-water blocking agent

2.3.1 Surface tension test

At room temperature, 0, 0.02wt%, 0.05wt%, 0.1wt%, 0.3wt%, 0.5wt%, and 1.0wt% anti-water blocking agents (DAT-1) were added to 3wt% NaCl brine solutions, respectively. The surface tension of the above brine solutions was measured with a BP100 Surface Tension Meter (Krüss Corporation, Hamburg, Germany). The solutions with different concentrations of DAT-1 were tested three times to evaluate the effect of anti-water blocking agent on the surface tension of the solution.

2.3.2 Contact angle test

The surface of the sandstone was smoothed with 200-300 mesh sandpaper. Then, the sandstone surface was soaked in the 3 wt% NaCl brine solution containing different concentrations of the anti-water blocking agent (DAT-1) for 4 h. After drying at room temperature, the contact angle of distilled water on the sandstone surface was tested by a JC2000DM Contact Angle tester (Beijing Zhongyi Kexin Technology Co, Ltd., Beijing, China). The effect of the anti-water blocking agent (DAT-1) on the wettability of the sandstone surface was evaluated.

2.3.3 Glass capillary test

The glass capillary test further assessed the effect of anti-water blocking agent on the wettability of reservoir pore throats. The capillary radius used in this experiment is approximately 0.5 mm. The glass capillaries were washed with distilled water and dried at high temperature. The cleaned glass capillaries were then inserted into the 3 wt% NaCl brine solutions with various concentrations of anti-water blocking agent for 2 h; then the height of the liquid rise or fall in the glass capillary was observed. The schematic is shown in Fig. 1. With a positive rise of the liq-

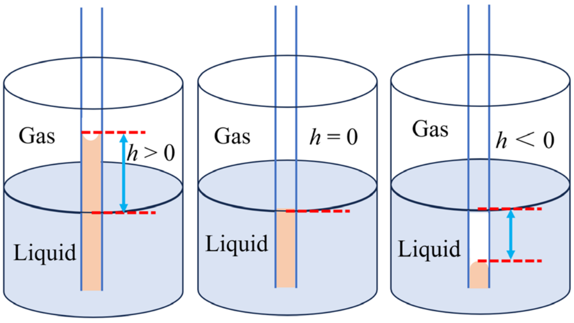

Fig.1 Simple device for the glass capillary test

uid in the capillary ($h > 0$), the contact angle between the liquid and the glass surface was less than 90°. When the liquid level in the tube was neutral ($h = 0$), the contact angle between the liquid and the glass surface was 90°. When the liquid level in the tube was negative ($h < 0$), the contact angle between the liquid and the glass surface was greater than 90°. The contact angle can be calculated by Equation 1.

$$\theta = arccos\left(\frac{\rho ghr}{2\sigma}\right) \tag{1}$$

Here σ is the surface tension of the solution (mN/m); ρ is the density of the solution (g/cm^3); r is the radius of the glass capillary (mm); g is the constant of gravitational acceleration (N/kg); θ is the contact angle (degrees); h is the height of the liquid in a glass capillary tube (mm).

2.3.4 The spontaneous impregnation test

Simple device for the spontaneous impregnation test is shown in Fig. 2. Referring to the China's petroleum and natural gas industry standard SY/T5336-2006, the sandstone cores (permeability: 0.5-2.0 mD, porosity: 7.21–11.85%, pore volume: 2.40-3.35 mL) were pretreated and dried. The cores were then soaked in 3 wt% NaCl brine solutions without or with 0.5% anti-water blocking agent respectively, and the mass of impregnation of the cores was checked at different times.

2.3.5 Evaluation of reservoir protection performance of anti-water blocking agents

Referring to the industry standard SY-T 6370–1998 "Core Gas Permeability Tester", the core permeability before and after the spontaneous impregnation test was measured by a HKY-200 Gas Permeability meter (Haian Petroleum Scientific Research Instrument Co, Ltd., Nantong, China). Referring to the industry standard SY/T 6540-2002 "Indoor Evaluation Method for Damage to Oil Reservoir by Drilling Fluid and Completion Fluids", a reverse displacement of kerosene was carried out on the self-priming cores. A constant injection rate was set at 0.5 mL/min during the kerosene displacement experiments. The breakthrough pressure is defined as the displacement pressure that can drive the first drop of kerosene passing through the self-priming cores [18]. And the smaller the breakthrough pressure, the smaller the capillary force of the liquid in the core pore throats.

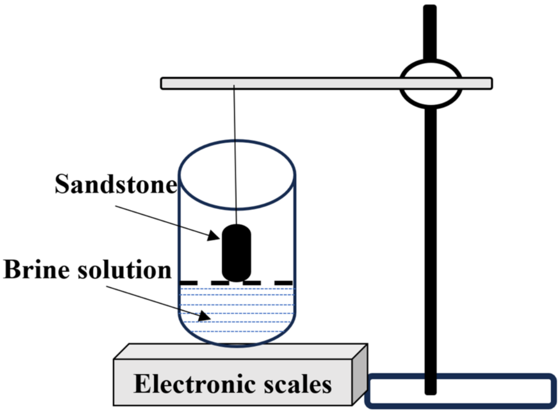

Fig.2 Simple device for the spontaneous impregnation test

2.3.6 Nuclear magnetic resonance（NMR）experiment

After pretreatment and drying, the cores absorbed 3 wt% NaCl brine solutions without or with 0.5% anti-water blocking agent respectively. The self-priming time was 60 min. Then, the cores were analyzed by a DPX-400 nuclear magnetic resonance spectrometer (Bruker Corporation, Massachusetts, USA) and the T_2 spectra were tested. The magnetic field strength was 2 MHz and the echo time was 100 μs.

2.3.7 Zeta potential test

At room temperature, 0, 0.1wt%, 0.3wt%, 0.5wt%, and 1.0wt% anti-water blocking agent (DAT-1) were added to 3wt% NaCl brine solutions. Then, the Zeta potential of the above brine solutions was measured using a JS94H Zeta potentiometer (Shanghai Zhongchen Digital Technology Equipment Co, Ltd., Shanghai, China)

2.3.8 Surface microscopic analysis

The surface of the sandstone was smoothed with 200-300 mesh sandpaper. Then, the sandstone surface was soaked in 3 wt% NaCl brine solutions with 0.5 wt% DAT-1 for 4 h. After drying at room temperature, the SEM images of the surface were observed via a Quanta3D FEG Field Emission Scanning Electron Microscope (American FEI Corporation, Oregon, USA).

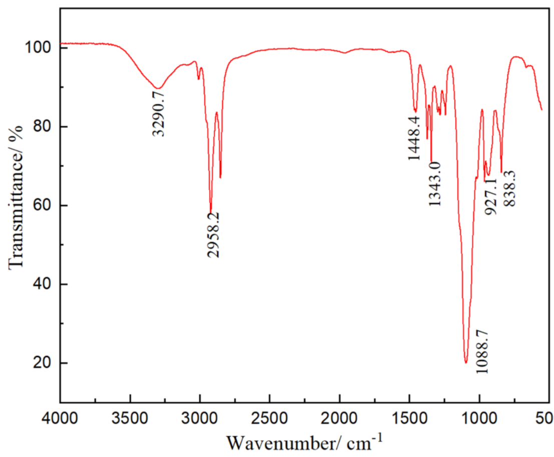

Fig.3 FTIR spectrum of anti-water blocking agent (DAT-1)

3. Results and Discussion

3.1 Structural characterization of the anti-water blocking agent

The infrared spectrum of the anti-water blocking agent (DAT-1) is shown in Fig. 3. From Fig. 3, it can be seen that the spectrum absorption peak at 2958.2 cm^{-1} was the stretching vibration of C-H in methyl and methylene groups. The strong absorption peak at 1088.7 cm^{-1} corresponded to the stretching vibration of Si-O-Si. The peak at 838.3 cm^{-1} is characteristic of $-Si-(CH_3)_2$. The peak at 1448.4 cm^{-1} is characteristic of $-N^+-(CH_3)_3$, indicating that the epoxide group in AGE was successfully opened. The spectrum absorption peak at 3290.7 cm^{-1} represents the stretching vibration of -OH, and the formation of -OH is due to the opening of the epoxy group. The spectrum absorption peak of Si-H was not found in the range of 2095 ~ 2157 cm^{-1}, indicating that there was addition reaction between AGE and DHSO. Therefore, the infrared spectrum confirmed that the anti-water blocking agent was successfully synthesized.

3.2 Surface tension test

According to the Laplace equation, the capillary force of the liquid phase in the pore throats is proportional to its surface tension, indicating that the greater the capillary force, the greater the flow resistance of the liquid phase in the pore throats. The effect of anti-water blocking agent at various concentrations on the surface tension of the brine solution is shown in Fig. 4. From Fig. 4, it can be seen that the surface tension of the brine solution significantly decreased when the dosage of anti-water blocking agent increased from 0 to 0.5 wt%, which could effectively reduce the flow resistance of the liquid phase in the core pores; at the dosage of anti-water blocking agent of 0.5 wt%, the surface tension of the brine solution was less than 15 mN/m, indicating that the anti-water blocking agent (DAT-1) can effectively reduce the capillary force of water phase in the core pores. In addition, with a further increase in the DAT-1 concentration, the decrease in surface tension slowed down, indicating that the critical micelle formation concentration (CMC) of the surfactant was exceeded.

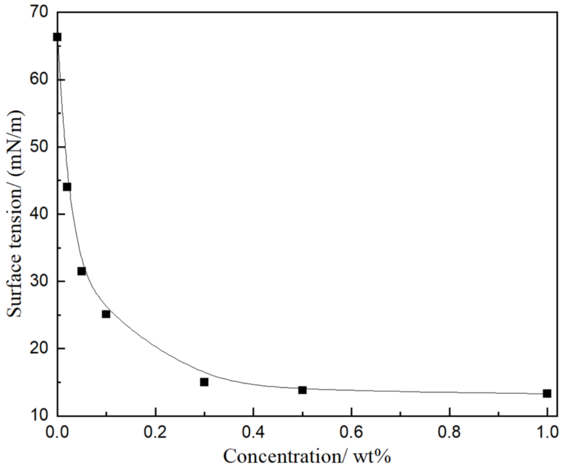

Fig.4 Effect of DAT-1 at various concentrations on the surface tension

3.3 Contact angle testing

The effect of the anti-water blocking agent at various concentrations on the water contact angle of the sandstone core is shown in Fig. 5. As can be seen from Fig. 5, the water contact angle on the surface of the sandstone core after treatment with the brine solution was only 16.8°, indicating that the original sandstone core was hydrophilic. After being treated with the brine solution containing the anti-water blocking agent (DAT-1), the water contact angle on the core surface significantly increased. Notably, the water contact angle on the core surface gradually increased with increasing the amount of anti-water blocking agent (DAT-1) added to the brine solution. When the dosage of anti-water blocking agent (DAT-1) in the brine solution was 0.5 wt%, the water contact angle on the core surface increased to 85.3°. According to the Laplace equation, an increase in the water contact angle reduced the capillary force of the liquid phase in the pore throats. In addition, the developed anti-water blocking agent (DAT-1) can alter the wettability of the core

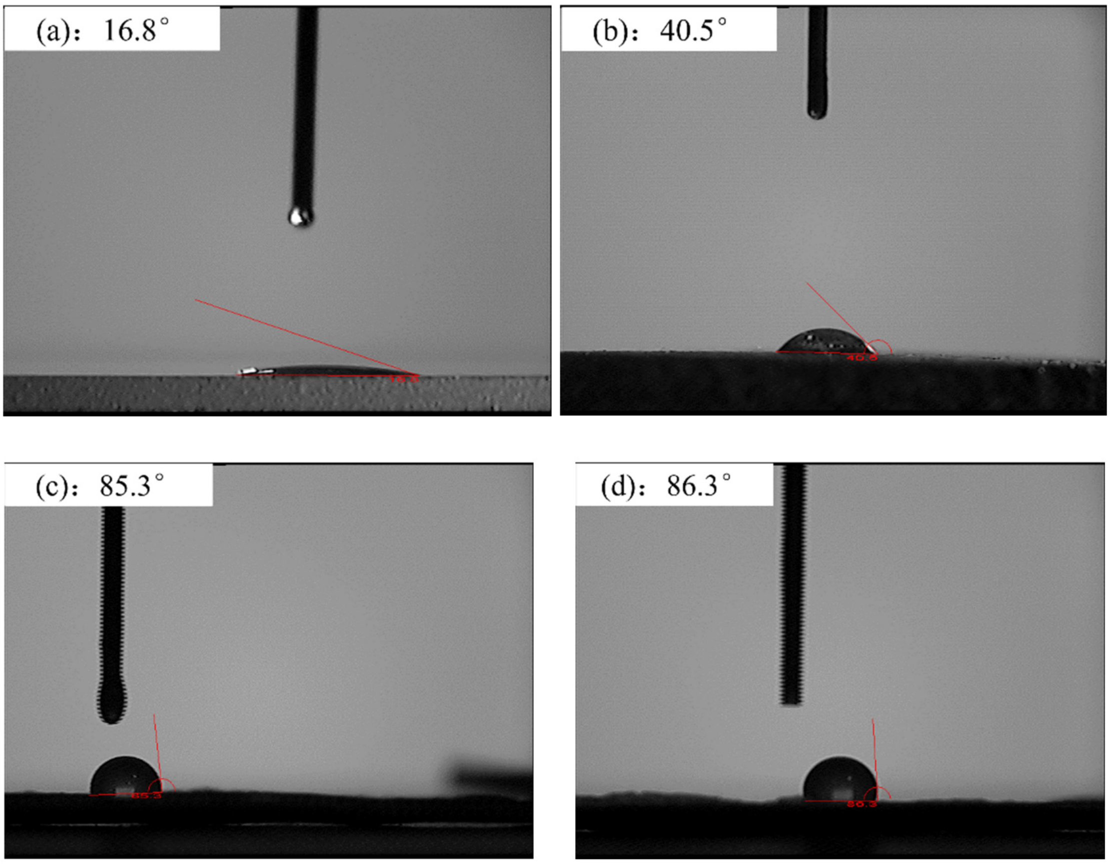

Fig.5 Effect of DAT-1 at various concentrations on the water contact angle of the core: (a) untreated, (b) 0.1 wt%, (c) 0.5 wt%, and (d) 1.0 wt%.

surface from hydrophilicity to neutral wetness, which can effectively reduce the viscous force of the water phase during migration within core pores.

3.4 Evaluation of Glass Capillary Experiment

The wettability of the core surface before and after treatment was verified by glass capillary experiment, and the experimental results are shown in Fig. 6. It can be seen from Fig. 6 that as the amount of the anti-water blocking agent (DAT-1) added to the brine solutions increased, the rise height of the brine solutions in the capillary gradually decreased. When the amount of anti-water blocking agent (DAT-1) added to the brine solution was 0.5 wt%, the rise height of the brine solution in the capillary decreased from 28.1 mm to 0.2 mm, indicating that the developed anti-water blocking agent (DAT-1) can effectively prevent the water phase from entering the deep throats of the reservoir. According to the capillary formula (equation 1) mentioned above and the test data of surface tension, the calculated contact angle was consistent with the result of the water contact angle measured by the contact angle tester.

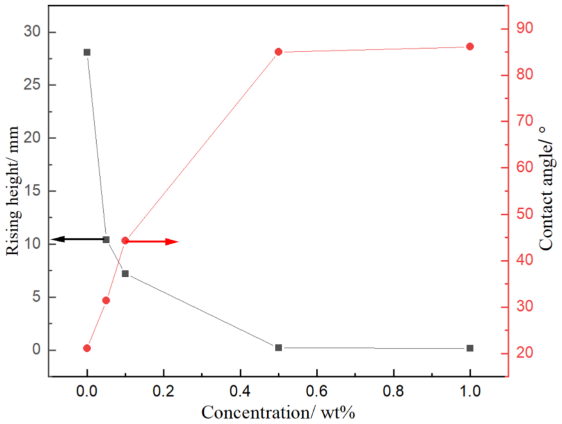

Fig.6 Rise height of the brine solutions in capillary at various concentrations of DAT-1

3.5 The spontaneous imbibition measurement results

The impregnation mass of the sandstone cores in the brine solutions without or with 0.5 wt% anti-water blocking agent (DAT-1) are shown in Fig. 7; and the basic information on the cores is presented in Table 1. From Fig. 7,

Table 1- The basic information on the cores

No.	Gas permeability/ mD	Core porosity/ %	Pore volume/ mL
	0.89	8.43	3.04
13#	0.71	7.26	2.86
15#	1.33	9.64	3.15
19#	1.14	8.84	3.08

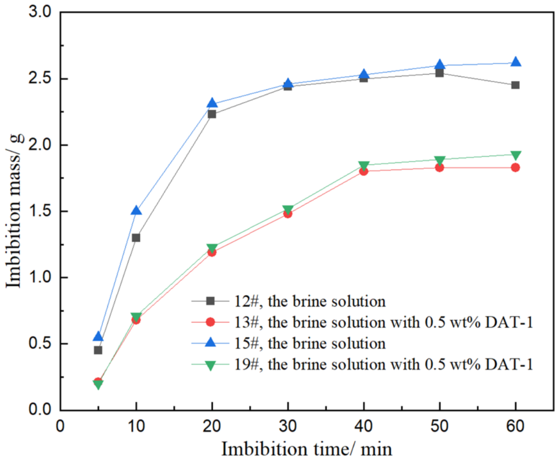

Fig.7 Relation between imbibition mass and time

it can be seen that the 0.5 wt% anti-water blocking agent (DAT-1) significantly reduced the mass and rate of water absorption by the cores; this indicates that the anti-water blocking agent (DAT-1) reduced the surface tension of the brine solution and changed the wettability of the pore throats, resulting in a sharp decrease in capillary force. Molecules of the anti-water blocking agent (DAT-1) can be adsorbed on the surface of the pore throats, changing their wettability. Therefore, the anti-water blocking agent (DAT-1) can significantly reduce the water-blocking effect of water phase in the pore throats, which is of great importance for protecting the productivity of a low-permeability reservoir.

3.6 Core permeability testing

The results of tests for gas permeability of cores 12 # and 13 # are shown in Fig. 8. The results of tests for breakthrough pressure of cores 15 # and 19 # treated with the brine solutions without or with 0.5 wt% anti-water blocking agent (DAT-1) are shown in Fig. 8. It can be seen that the gas permeability of the core contaminated with the brine solution was only 0.33 mD, while the gas permeability of the core contaminated with the brine solution containing 0.5 wt% anti-water blocking agent (DAT-1) was 0.49 mD. From Fig. 9, it can be seen that compared to the polluted core 15 #, the breakthrough pressure of the polluted core 19 # was smaller. From the results of gas permeability and the breakthrough pressure, it can be seen that the addition of anti-water blocking agent (DAT-1) to the brine solution improved the recovery rate of core permeability, reduced the binding ability of core pore throats to water phase.

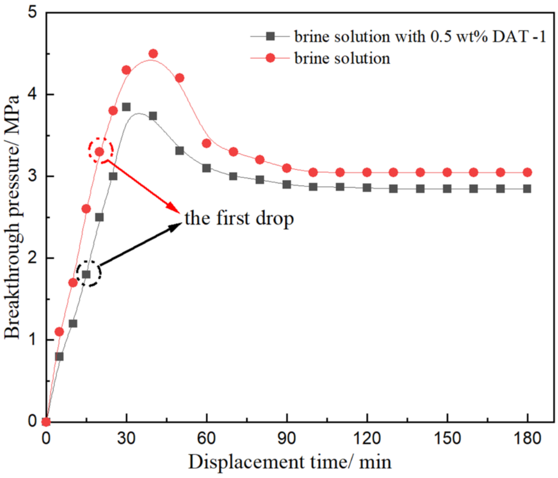

Fig.8 Gas permeability of two cores before and after imbibition

3.7 Nuclear Magnetic Resonance (NMR) Experiment

Due to the different degree of water binding with different sizes of the pore throats, the corresponding NMR T_2 relaxation time is also different. The relaxation time is longer in large pore throats and shorter in small pore throats. In addition, the NMR signal amplitude is proportional to the amount of water signal. The NMR T_2 spectra of the cores (12 # and 13 #) after impregnation are shown in Fig. 10. From Fig. 10, it can be seen that the signal amplitude from the small pores increased rapidly, while the signal amplitude from the large pores also increased, but slowly, which was due to the strong capillary force in small pores. Accord-

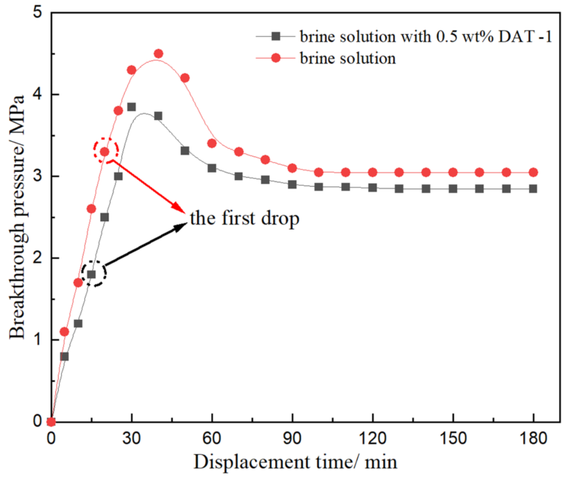

Fig.9 Breakthrough pressure of the contaminated cores

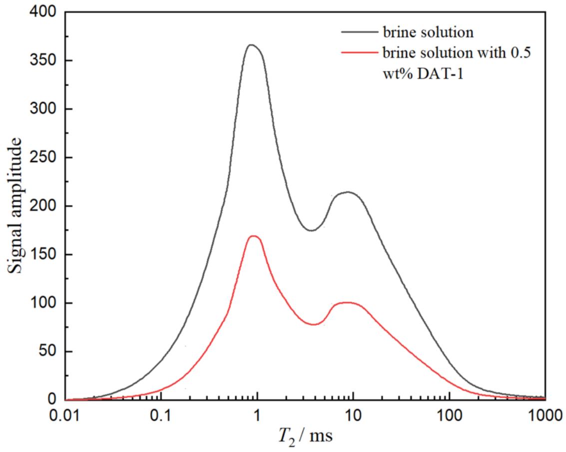

Fig.10 NMR T_2 spectra of the cores after impregnation

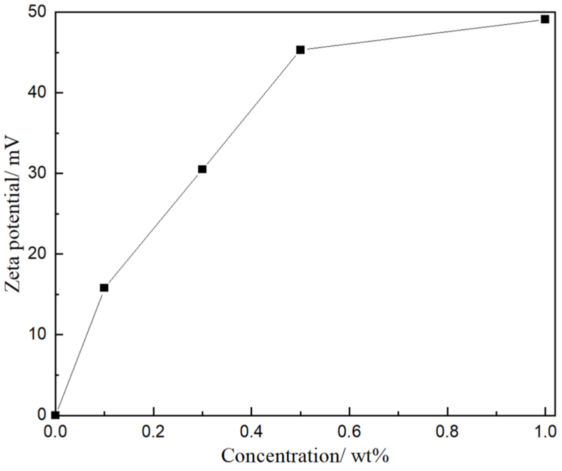

Fig.11 Effect of DAT-1 at different concentrations on Zeta potential

ing to the observed NMR signal amplitude, the difference between the two solutions in the impregnation of large pores in the cores was insignificant, while the impregnation of fine core pores with the brine solution containing 0.5 wt% anti-water blocking agent (DAT-1) was significantly reduced. The explanation of the results is that the anti-water blocking agent (DAT-1) can reduce the surface tension of the brine solutions and increase the water contact angle on the surface of the cores, so that the capillary force is reduced.

3.8 Zeta potential test

The Zeta potential of the brine solutions with different concentrations of the anti-water blocking agent (DAT-1) was measured. The experimental results show (Fig. 11) that with increasing DAT-1 concentration, the Zeta potential of the brine solutions gradually increased. When the dosage of the anti-water blocking agent (DAT-1) was 0.5 wt%, the Zeta potential of the brine solution reached 45.3mV. The explanation of the result is that the DAT-1 molecule has a quaternary ammonium salt structure with a positive charge. The surface of cores is usually negatively charged, and DAT-1 molecules can be adsorbed on the surface of cores through the electrostatic force.

3.9 Surface microscopic analysis

The SEM images of the sandstone surface before and after treatment are shown in Fig. 12. The sandstone surface before treatment was rough and there were many large particles. Under the action of hydrophilic minerals, the sandstone surface became hydrophilic. The sandstone surface after treatment was smooth, because DAT-1 molecules were adsorbed onto the surface by electrostatic action to form a dense film. In addition, the polysiloxane chain in the middle of DAT-1 molecules had good rotational flexibility, allowing the molecules to tightly cover the sandstone surface. At the same time, the flexibility of the polysiloxane chain segment also allowed the ethyl groups on both sides to accumulate at a high density, which resulted in wettability alteration of the sandstone surface. And it can reduce water blocking damage.

4. Conclusions

1) In this paper, the double-ended hydrogen containing silicone oil (DHSO), allyl glycidyl ether (AGE), and triethylamine hydrochloride (TH) were used as raw materials to prepare a new anti-water blocking agent (DAT-1), significantly reducing the surface tension of the brine solutions, increasing the water contact angle on the sandstone surface, which can reduce

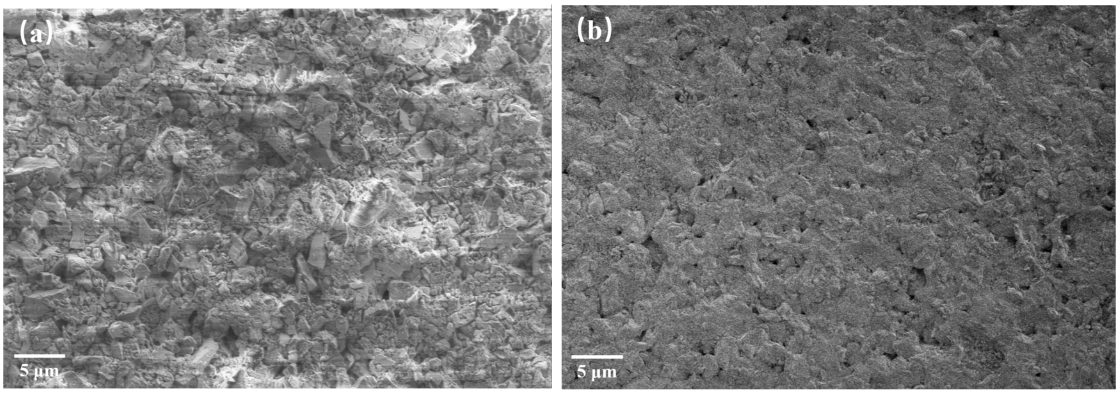

Fig.12 SEM images of the sandstone surface: (a) before treatment, and (b) after treatment.

the capillary force of external fluids in the pore throats.

2) The impregnation experiments and NMR indicated that the anti-water blocking agent (DAT-1) reduced the binding capacity of brine in pore throats, which can alleviate water blocking damage of low permeability reservoirs. The results of core permeability study showed that the addition of the anti-water blocking agent (DAT-1) to the brine solution reduced the backflow pressure of water phase in the pore throats and improved the recovery rate of core permeability.

3) The DAT-1 molecules were adsorbed on the surface by electrostatic action to form a dense film. In addition, the polysiloxane chain and the ethyl groups can improve the density of the adsorption film, resulting in wettability alteration of the sandstone surface, which reduce water blocking damage.

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