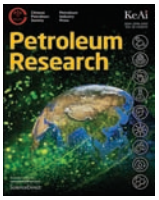
Petroleum Research 11 (2026) 84–91

Contents lists available at ScienceDirect

Petroleum Research

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Full Length Article

Enhancing oil recovery of gas flooding by regulating rock surface wettability



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ARTICLE INFO

Article history:
Received 25 December 2024
Received in revised form
22 July 2025
Accepted 23 July 2025
Available online 30 July 2025

Keywords:
Rock surface
Oil wettability
Gas flooding
Enhanced oil recovery

ABSTRACT

Rock surface wettability plays a crucial role in gas flooding for oil recovery. Natural rock surfaces are usually oleophilic, resulting in high flooding resistance and low recovery in gas flooding. This study explores the regulation of the wettability of sandstone rock surfaces by modifying them with 1H,1H,2H,2H-perfluorodecyltrimethoxysilane or its mixtures with hexyltrimethoxysilane to achieve oil contact angles ranging from 0° to 93.2°. Simulated gas flooding experiments, with N_2 as model gas, reveal that reducing the oleophilicity of rock surfaces improves gas flooding efficiency. Compared to the unmodified superoleophilic rocks, the final oil recoveries in the cores with neutral oil wettability significantly increases and the pressure differential for gas flooding significantly decreases. In cases of cores with permeability of 22 mD, 0.88 mD and 0.09 mD, the increases in oil recovery are from 28.5% to 37.5%, from 20.2 to 27.3% and from 9.1% to 15.4%, respectively; and the reductions of pressure differential for gas flooding are from 53 kPa to 41 kPa, from 323 kPa to 272 kPa, from 1054 kPa to 935 kPa, respectively. These results highlight the critical influence of wettability regulation on gas flooding efficiency, offering valuable insights for optimizing oil recovery strategies.

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1. Introduction

After primary recovery, such as water or gas are often injected into the reservoirs to maintain oil production levels (Alagorni et al., 2015; Liu et al., 2020; Wang, 2021; Wang et al., 2023a). However, many reservoirs with low permeability pose challenges to conventional water flooding techniques due to high capillary forces and the potential risk of water blockage (Hassan et al., 2019). Gas flooding, by contrast, is more effective in such reservoirs, because it reduces oil viscosity, lowers surface tension of oil, consumes less energy, and avoids damage in the formation (Jiang et al., 2010; Zhou et al., 2020).

Gas flooding has emerged as the predominant method for enhancing oil recovery in low-permeability fields due to its low energy consumption, broad adaptability, and operational simplicity (He et al., 2023; Qu et al., 2016; Wang et al., 2018). However, its effectiveness is limited when oil becomes trapped in the micropores of tight reservoirs (Wang et al., 2023b). During gas flooding, a three-phase boundary (gas, oil, and rock) forms at the displacement front, where capillary force within the rock's pores and fractures critically influence fluid dynamics (Wang et al., 2024; Yao et al., 2022). Capillary force which is a key determinant of oil recovery is governed by surface tensions of oil and rock surface, oil wettability on rock surface and pore radius (Zhang et al., 2024). Regulating rock surface wettability can optimize capillary force, thereby affecting recovery rates (Wan et al., 2022).

To achieve the goal of regulating oil wettability, various technological approaches have been explored, including microbiological, physical and chemical methods (Ding and Gao, 2021; Xia et al., 2023). For example, Soleimani et al. (2023) reported the feasibility of using asphaltene adsorption on rocks after microbial aging to keep rock samples in their water-wetted state. Jang et al. (2018) used 3-glycidoxypropyltrimethoxysilane to modify the wettability of silica nanoparticles in high-salinity and high-temperature conditions. Zhu et al. (2014) examined the influence of SiO_2 nanoparticles on the ability of hydrophobically associating polyacrylamide (HAHPAM) solutions to enhance oil recovery under high-salinity and high temperature conditions. Mohammed and Babadagli (2015, 2016) reported about the chemicals used to induce wettability changes and discussed their feasibility in heavy

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Peer review under the responsibility of Editorial Office of Petroleum Research.

https://doi.org/10.1016/j.ptlrs.2025.07.005
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oil sandstone and carbonate reservoirs. Kamal et al. (2017) mobilized residual oil by injecting surfactants to reduce the interfacial tension between oil and water and/or by changing the wettability, and reviewed the field application of surfactants for chemically enhanced oil recovery in carbonate and sandstone reservoirs. Rezaei et al. (2022) combined dodecylbenzenesulphonic acid with SiO_2 nanoparticles, showing that this synergy effectively reduced interfacial tension and promoted water-wet conditions, with surfactants playing a dominant role in wettability alteration. Despite these advances, significant limitations persist (Ali et al., 2023; Lin et al., 2018; Ratanpara and Kim, 2023): microbial methods are slow and constrained by reservoir temperature, salinity, and nutrient availability, making sustained activity difficult to control; surfactants risk pore blockage due to emulsification or precipitation; and physical methods suffer from surface abrasion or stress relaxation, leading to wettability reversal. To address these challenges, chemical surface modification has emerged as a precise and controllable alternative (Pothula et al., 2023). By reacting with surface functional groups, tailored chemical reagents can systematically tune rock wettability, thereby optimizing oil displacement efficiency (Purswani and Karpyn, 2019; Sakthivel et al., 2024). Pinto et al. (2020). Synthesized and evaluated some wettability modifiers, demonstrating their ability to regulate rock wettability, while also analyzing the effects of reagent dispersion stability and concentration on performance. Nevertheless, However, most prior studies focused on wettability alteration for water flooding, with limited systematic investigation into its impact on gas flooding efficiency.

In this study, by modifying superoleophilic natural sandstone rock surfaces (oil contact angle 0°) with 1H,1H,2H,2H-perfluorodecyltrimethoxysilane or its mixtures with hexyltrimethoxysilane, rocks with different oil wettability, including strongly oleophilic rock (oil contact angle 34.8°), weakly oleophilic rock (oil contact angle 62.1°), and neutral rock (oil contact angle 93.2°), were obtained. With these modified rocks and unmodified superoleophilic rocks, the effects of oil wettability on gas flooding efficiency have been systematically studied. The results indicated that decreasing rock surface oleophilicity could be a promising way to improve production and lower the difficulty in gas flooding, especially in tight reservoirs.

2. Materials and methods

2.1. Materials

1H,1H,2H,2H-perfluorodecyltrimethoxysilane (PFMS), hexyltrimethoxysilane (HTMS), hydrochloric acid, sodium hydroxide, sodium chloride and kerosene were purchased from Aladdin Reagents (Shanghai, China). Anhydrous ethanol was purchased from Chron Chemicals (Chengdu, China). All the chemicals were used as received without further purification. The cores used in this work were natural outcrop sandstone cores. The parameters of the cores are shown in Table 1.

Table 1
Parameters of cores with different permeabilities.

Core sample	22	0.88	0.09
Diameter (cm)	2.50	2.52	2.50
Length (cm)	7.03	7.02	7.02
Volume (cm^3)	34.51	35.01	34.45
Dry weight (g)	78.24	81.07	88.333
Permeability (mD)	22.03	0.88	0.09
Porosity (%)	15.65	9.25	6.41

2.2. Methodology

2.2.1. Rock surface modification

Fig. 1 shows the surface modification reaction. Briefly, ethanol solutions of 1H,1H,2H,2H-perfluorodecyltrimethoxysilane or its mixture with hexyltrimethoxysilane (total concentration of 3 wt%) were firstly prepared. In a setup as illustrated in Fig. 2, a cleaned and dried rock plug was put into the core holder. After the core was heated to 80 °C, the modifying solution was flowed into the core by N_2 bubbling for 6 h. After the reaction, the modified rock plug was collected and dried in vacuum at 60 °C overnight to obtain the modified rock sample. Rock powder samples were obtained by grinding the rock and modifying them in the modifying solutions.

In order to obtain rock surfaces with different oil wettability, the surface was modified with solutions composed of different ratio of 1H,1H,2H,2H-perfluorodecyltrimethoxysilane and hexyltrimethoxysilane (Table 2).

2.2.2. Simulated gas flooding

Gas flooding experiments were carried out in a setup as illustrated in Fig. 3. Firstly, core plug with different oil wettability was put into the core holder, and then it was saturated with kerosene as model oil. After weighing the amount of kerosene in the core, it was put into the core holder for simulated gas flooding. During gas flooding, the injection rate of N_2 was controlled to be 2 mL/min. The recovered oil was collected by a graduated cylinder. The oil recovery and pressure differential in the process were recorded. And the recovery was calculated according to the following equation:

$$\text{recovery ratio}\ (\%) = \frac{\rho_o V}{m_1 - m_0} \tag{1}$$

where ρ_o was the density of kerosene, V was the volume of collected kerosene, m_1 was the mass of core saturated with kerosene, and m_0 was the mass of dry core.

2.3. Characterizations

The structure and morphology of rock samples were analyzed with scanning electron microscopy (SEM) (Gemini SEM 300, Carl ZEISS), and X-ray diffraction (XRD) (Miniflex 600, Rigaku). Prior to SEM tests, the samples were fixed on the stage and sprayed with gold to improve the conductivity of the surface. XRD investigation was carried out with a scanning rate of 2°/min and a scanning range of 10°–80°. The chemical compositions of the rock surfaces were determined by Fourier transform-infrared spectroscopy (FT-IR) (Nicolet iS20, ThermoFisher) and X-ray photoelectron spectroscopy (XPS) (K-Alpha, ThermoFisher). FT-IR spectra were measured using transmission mode with KBr disks. XPS investigations were carried out with a tube voltage of 40 kV and a current of 40 mA. Contact angle measurements were made using a contact angle meter (SL250, KINO). For each measurement, a 2 μL drop was placed on the rock surface. After reaching a steady state, a side-view image was taken. The contact angle was obtained by measuring the angle between the liquid-rock surface and the liquid-air surface. To ensure the reliability of the measurements, three measurements were taken for each rock sample (Rezaei et al., 2020).

3. Results and discussion

3.1. Oil wettability regulation on rock surface

As shown in Fig. 4, the unmodified natural rock surface was superoleophilic, showing oil (kerosene) contact angle of 0°. After

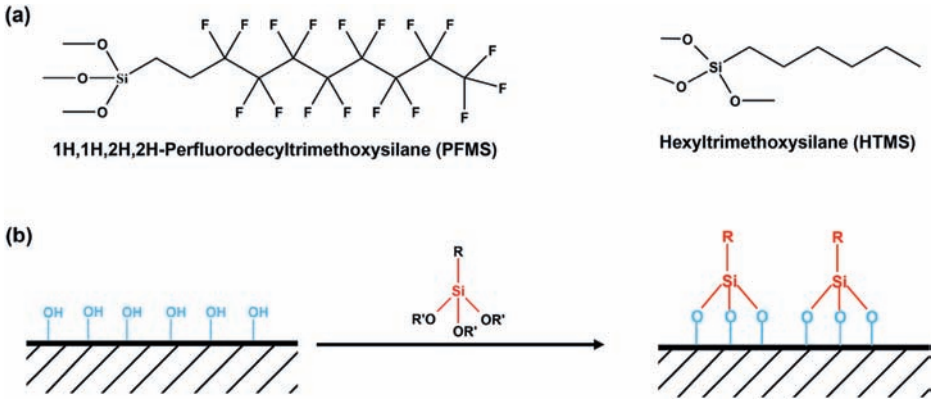


Fig. 1. (a) The reagents for rock surface modification; (b) The reaction of rock surface modification.

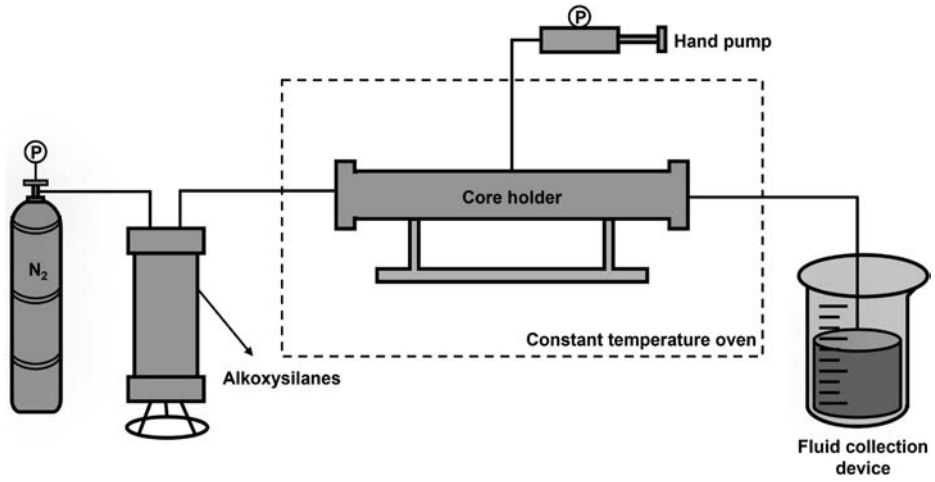


Fig. 2. Schematic diagram of experimental apparatus for rock surface modification.

Table 2
The composition of modifying solutions for different oil wettability.

Core oil wettability	1H,1H,2H,2H-perfluorodecyltrimethoxysilane (wt%)	hexyltrimethoxysilane (wt%)
Neutral	3	0
Weakly oleophilic	1.5	1.5
Strongly oleophilic	0.3	2.7
Superoleophilic (Unmodified)	–	–

modified with 1H,1H,2H,2H-perfluorodecyltrimethoxysilane or its mixtures with hexyltrimethoxysilane, rocks with different oil wettability were obtained. Hydrocarbon groups have low surface energy, while the surface energy of fluorocarbon groups is even lower. Therefore, after modification, the surface energy of rock surface was significantly decreased. And the more 1H,1H,2H,2H-perfluorodecyltrimethoxysilane, the lower the surface energy of rock surface, resulting in the higher oil contact angle. According to the oil contact angle on their surface, these rock samples were named as neutral rock (with oil contact angle of 93.2°), weakly oleophilic rock (with oil contact angle of 62.1°), and strongly oleophilic (with oil contact angle of 34.8°), respectively.

After modification, changes on the rock surfaces were analyzed. Fig. 5a illustrates the XPS spectra of the rock samples before and after modification. All the rocks showed more obvious peaks at binding energies of 101 eV (Si 2p), 152 eV (Si 2s), and 554 eV (O 1s), which were derived from SiO_2, the main component of rock substrate. As can be seen from the F 1s spectra in Fig. 5b, all the modified samples show a distinct F 1s peak around 688.5 eV compared to the unmodified natural rock. The intensity of the F 1s peak varied depending on the oil wettability of rocks. As the amount of 1H,1H,2H,2H-perfluorodecyltrimethoxysilane used in the modification increased, the fluorine content on the rock surface increased, accompanied by a corresponding rise in the oleophobicity of the rock surface. From the FT-IR spectra in Fig. 5c, it can be seen that the peak at 474 cm^{-1} was the symmetric bending vibration of the Si–O bonds, the absorption peaks at 1037 cm^{-1} and 807 cm^{-1} were the asymmetric stretching vibration peaks of the Si–*O*–Si bonds, and the vast absorption peak near 3428 cm^{-1} was the stretching vibration absorption peaks of the hydroxyl groups (Kurbanov et al., 2023; Rana et al., 2022). These absorption peaks were found in all rock samples because they were derived from the main component of the rock, i.e. silica. In addition, the modified rocks also showed other peaks that were not found in the

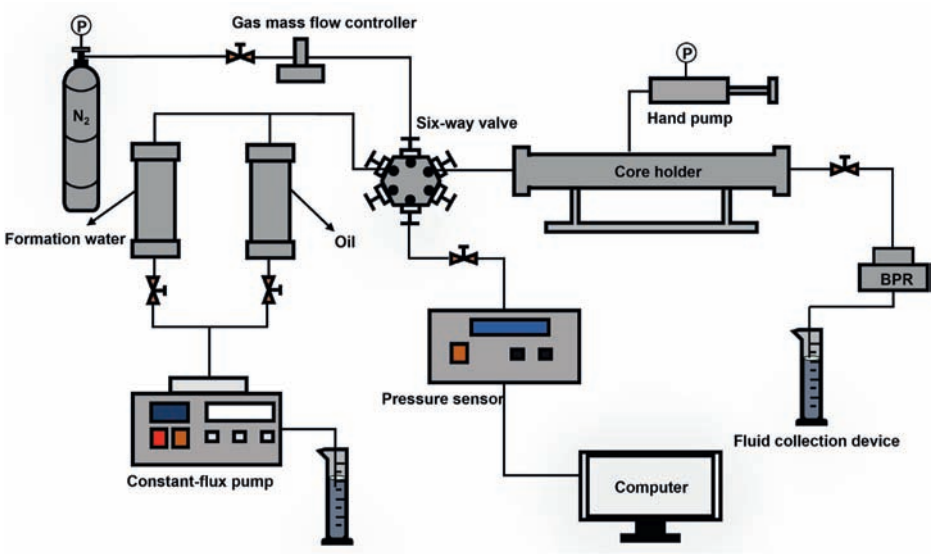


Fig. 3. Schematic diagram of experimental device for simulated gas flooding.

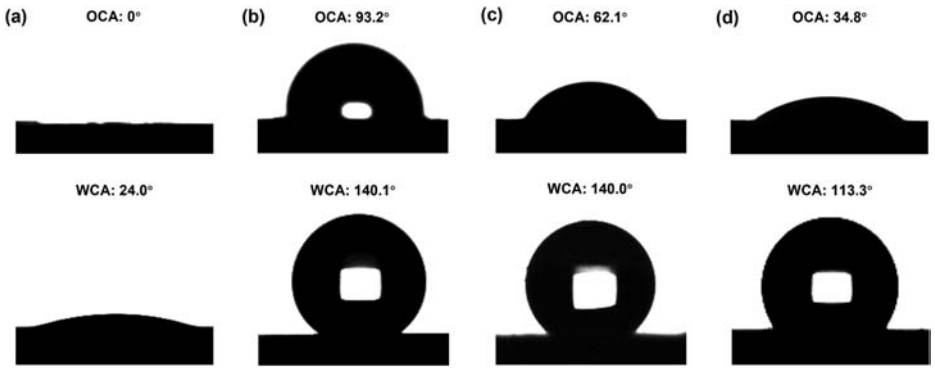


Fig. 4. The contact angles of oil (kerosene) (OCA) and water (WCA) on the surface of rocks. (a) unmodified superoleophilic rock; (b) neutral rock; (c) weakly oleophilic rock; and (d) strongly oleophilic rock.

unmodified natural rock sample: the peak of the C–H bonds at 2933 cm^{-1}; and the peaks related to the C–F bonds at 1249 cm^{-1}, 1132 cm^{-1}, and 1103 cm^{-1} for the neutral oleophilic rocks. The weakly oleophilic rock and neutral rock also showed a C–F bond-related peak at 1249 cm^{-1} (Su et al., 2023); however, it did not appear in the strongly oleophilic rock. This was because in the modification to prepare the strongly oleophilic sample, the amount of 1H,1H,2H,2H-perfluorodecyltrimethoxysilane used was much less than the other two rocks. These results indicate that new elements were successfully introduced onto the surface of the modified rocks, and the chemical composition of the rock surface was changed, making the oil wettability of the surface changed.

Although chemical composition changes occurred on the rock surface after modification, the structural changes were minimal. As shown in Fig. 5d, XRD investigations showed that only peaks related to SiO_2 could be observed for all these samples, meaning that the crystal structure of the modified rock remained identical to that of the unmodified natural rock. The primary component of the natural rock was SiO_2. The modification reaction happened only on the surface. Therefore, the inner crystal structure of the rock was not affected when the surface wettability altered.

Fig. 6 shows the SEM images of the cross-section of rocks before and after modification. The modification process introduced a layer of hydrocarbon or fluorocarbon to the surface of rock channels. The thickness of the introduced groups was about 2–3 nm, which was negligible compared to the size of rock channels which were on the level of micrometers. From these images, it could be seen that there was no significant change on the microscopic morphology of the modified rocks compared to the unmodified natural rock. These results suggest that the chemical modifications used to alter the wettability of the rock surface did not impact the rock's microscopic structure or internal morphology. This was beneficial for enhancing oil recovery, as the modification changed the surface wettability which could promote the stripping of oil from the surface of rock channels, while the maintenance of the microstructure could remain the pathway for oil to flow. Therefore, the stripped oil could be displaced from its original position to the production well more easily.

As the rock in the formation suffers high salt environment, the modification layer should have considerable tolerance. In the salt tolerance investigations, modified rock samples with neutral oil wettability were immersed in brines with salinity of 0.001, 0.01, 0.1, 1, 10, and 100 g/L for seven days, respectively. The test results shown in Fig. 7a demonstrate that the oil and water contact angles on the modified rock surface did not change significantly for all these samples. As the production of oil could result in high-speed scouring on rock surfaces, the modified surface should have good resistance to fluid scouring. Fig. 7b shows the surface wettability of

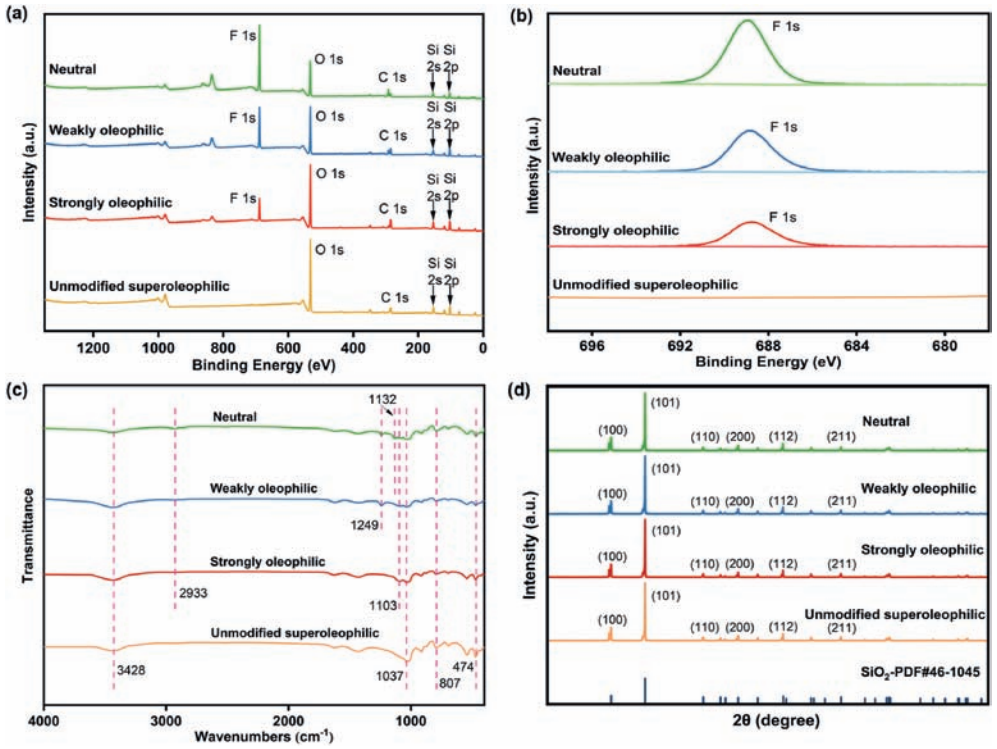


Fig. 5. The XPS full spectrum (a), high-resolution F 1s XPS spectrum (b), FT-IR spectra (c), and XRD spectra (d) of rock samples.

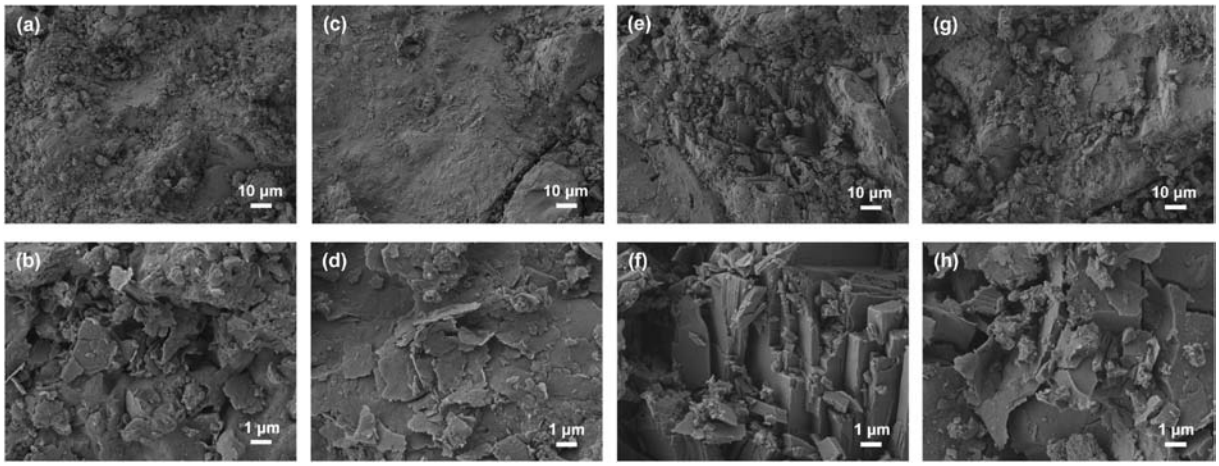


Fig. 6. SEM images of rocks. (a), (b) unmodified superoleophilic rock; (c), (d) strongly oleophilic rock; (e), (f) weakly oleophilic rock; (g), (h) neutral rock.

modified rock samples with neutral oil wettability after 10 m/min water flow scoured for up to 6 days. The oil/water contact angles on the rock surface remained stable with prolonged scouring time, indicating that the chemically modified layer was not significantly affected under long-term scouring conditions. In summary, chemical modification of rock surface could result in surface which were stable in harsh formation environments and could last for a long period of time to maintain oilfield production.

3.2. Enhancing gas flooding oil recovery

During gas flooding, gas phase injected into oil formation could reduce the viscosity of oil, and reduce the interfacial tension, thus promoting the flow of oil to the production wells to improve oil recovery. However, in reservoirs where the rock is usually oleophilic, oil tends to adhere to the rock surface, which leads to a decrease in the contact area between oil and gas and reduces the pumping effect of gas in oil. In addition, the oleophilicity of the rock surface may also lead to impeded gas transportation in the reservoir, resulting in gas barrier phenomenon that limits gas flooding efficiency.

The change of rock surface oil wettability could change the flow behaviors of oil phase and gas phase in gas flooding, therefore it could have significant effects on the efficiency of gas flooding. With N_2 as model gas, simulated gas flooding experiments were conducted with cylindrical core plugs with different oil wettability to systematically investigate the effects of rock surface oil wettability on gas flooding. During the experiments,

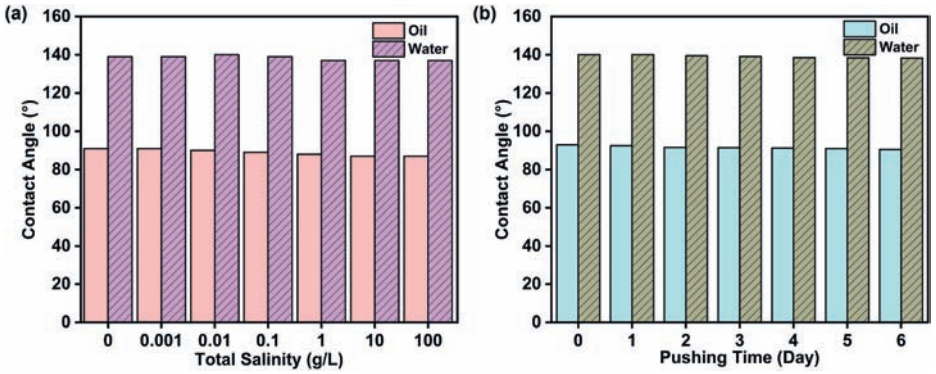


Fig. 7. Oil and water contact angles of modified rocks with neutral oil wettability. (a) After suffering high salt conditions. (b) After suffering water flow scouring.

the cores with different oil wettability were firstly saturated with kerosene, followed by N_2 flooding to harvest the oil inside the cores. The oil recovery and the flooding pressure during the process were investigated. The oil recovery reflects the effectiveness of gas flooding. The higher the oil recovery, the higher the oil production. The flooding pressure reflects the resistance of

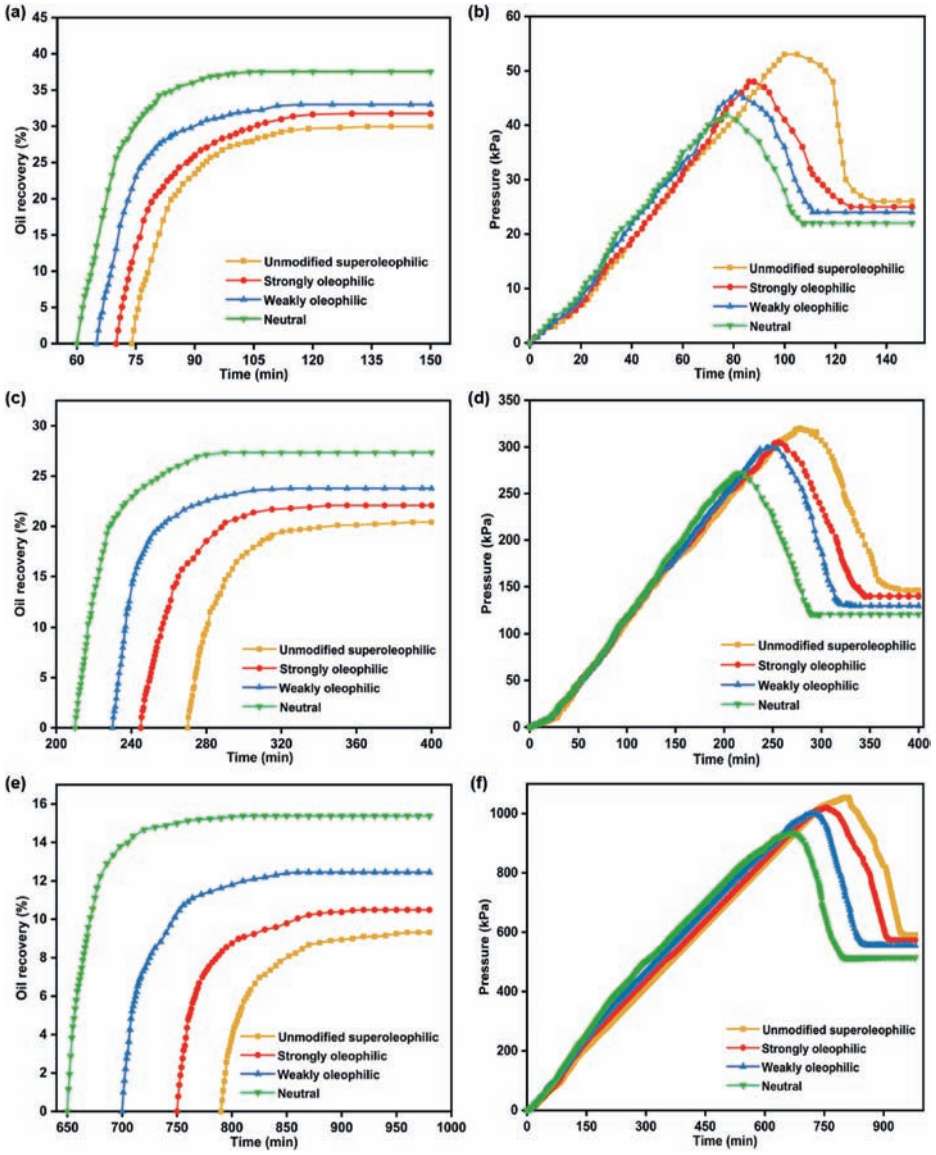


Fig. 8. Oil recovery and flooding pressure over time in simulated gas flooding. Oil recovery (a) and flooding pressure (b) of cores with permeability of 22 mD under different oil-wettability; Oil recovery (c) and pressure (d) of cores with permeability of 0.88 mD under different oil-wettability; Oil recovery (e) and pressure (f) of cores with permeability of 0.09 mD under different oil-wettability.

gas injection, i.e., the smaller the flooding pressure, the easier the gas injection.

Take the cores with a permeability of ~22 mD as an example (Fig. 8). For an unmodified superoleophilic natural core, the maximum flooding pressure during gas flooding was 53 kPa, and the final oil recovery rate was 28.5%. For strongly oleophilic, weakly oleophilic, and neutral cores, the maximum flooding pressures were 48 kPa, 45 kPa, and 41 kPa, respectively, and the final oil recoveries were 31.7%, 33.1%, and 37.5%, respectively. As the oleophilicity decreases, the maximum flooding pressure decreases, and the oil recovery increases. This means that gas flooding gradually became easier and oil production increased. It should be mentioned that the displacing pressure showed a trend of "peak first, then decline, and finally stabilize" (Fig. 8b). In the initial stage, the flooding gas needed to overcome capillary forces and viscous resistance of oil in rock channels and to push oil out. In this process, the flooding gas was gradually compressed, making the pressure gradually rise. As the flooding gas volume increased, it gradually broke the obstruction of oil to form continuous flow channels, therefore the flow resistance reduced. Moreover, the oil mobility also gradually increased as it was flooded by gas. Consequently, the flooding pressure dropped. In the last stage, the gas flow and oil flow reached a dynamic equilibrium, reaching a balance in flooding pressure.

Similar trends were observed in the cores with permeability of 0.88 mD and 0.09 mD (Fig. 8). Compared with the high permeability cores, the decrease in flooding pressure was larger in low permeability cores, which indicates that the difficulty of the gas flooding process reduced more. For instance, in the case of cores with a permeability of 0.88 mD, the maximum flooding pressure of the neutral core was 272 kPa, 51 kPa lower than that of the unmodified superoleophilic core (323 kPa). And their oil recovery was 27.3% and 20.2%, respectively. While in the cores with permeability of 0.09 mD, the maximum flooding pressure of the neutral core was 935 kPa, 119 kPa lower compared with the unmodified superoleophilic core (1054 kPa), and their oil recovery was 15.4% and 9.1%, respectively.

The reduction in displacement pressure observed during gas flooding can be explained through the fundamental capillary pressure equation:

$$P_c = 2\sigma \cos\theta / r \quad (2)$$

where P_c is the capillary pressure, σ is the interfacial tension between oil and gas, θ is the contact angle of oil on the rock surface, and r is the pore radius. The surface of unmodified rock was superoleophilic, the oil phase strongly wets the rock surface, making oil the wetting phase and gas the non-wetting phase. Gas flooding must overcome significant capillary resistance (P_c), where the magnitude of resistance scales with cos θ. Chemical modification increases the oil contact angle (θ), sharply reducing cos θ. This decreased the absolute value of P_c. If θ exceeds 90° (neutral-wet or oleophobic), the capillary pressure reverses direction, transforming resistance into a driving force that assists gas displacement. Even without full wettability reversal, reduced cos θ diminishes capillary resistance, lowering the critical pressure differential required for effective flooding.

In low-permeability cores, smaller pore radii (r) amplify capillary forces ($P_c \propto 1/r$). Wettability modification thus has a disproportionately greater impact in tight reservoirs. Stronger initial oil adhesion (higher cos θ) in small pores. Larger relative reduction in P_c for the same change in θ. Consequently, wettability regulation offers greater efficiency gains in low-permeability systems.

4. Conclusions

Natural rock surfaces are typically oleophilic, which adversely affects gas flooding efficiency. In this study, we successfully reduced surface oleophilicity through chemical modification using *1H,1H,2H,2H*-perfluorodecyltrimethoxysilane and its mixtures with hexyltrimethoxysilane. Comprehensive characterization via XPS, FT-IR, XRD, and SEM confirmed that the modification altered surface chemistry without affecting the crystalline structure or microscopic morphology of the rocks. The modified surfaces demonstrated excellent long-term stability under harsh conditions, as verified through salt resistance and scouring tests. Key findings from simulated gas flooding experiments revealed that reduced surface oleophilicity significantly enhanced final oil recovery while substantially decreasing the required flooding pressure differential. The improvement was particularly pronounced in low-permeability cores, where stronger initial capillary forces made wettability modification more impactful. This dual benefit (higher production with lower operational difficulty) suggests that surface engineering could transform gas flooding economics. These results provide a practical pathway for optimizing gas flooding performance, especially in challenging low-permeability reservoirs. The demonstrated stability and efficacy of our chemical modification approach offer strong potential for field-scale implementation to enhance.

CRediT authorship contribution statement

Chengxi Deng: Writing – original draft. **Min Chen:** Visualization, Validation. **Changsong Liu:** Funding acquisition. **Yinfeng Xu:** Data curation. **Yonghui Du:** Resources. **Hua Dong:** Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

This work was sponsored by CNPC Innovation Found (No.2024DQ02-0126).

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