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Original Paper

Synthesis of temperature and salt resistance silicon dots for effective enhanced oil recovery in tight reservoir

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ABSTRACT

The intensive development of tight reservoirs has positioned them as a strategic alternative to conventional oil and gas resources. Existing enhanced oil recovery (EOR) methods struggle to effectively exploring reservoir oil, resulting in low recovery rates. Novel and effective means of developing tight reservoirs are urgently needed. Nanomaterials have shown promising applications in improving water flooding efficiency, with in-depth research into mechanisms that lower injection pressure and increase water injection volumes. However, the extent of improvement remains limited. In this study, a silicon quantum dots (Si-QDs) material was synthesized via a hydrothermal synthesis method and used to prepare a nanofluid for the efficient recovery of tight reservoir. The Si-QDs, with an approximate diameter of 3 nm and a spherical structure, were surface functionalized with benzenesulfonic acid groups to enhance the performance. The developed nanofluid demonstrated stability without aggregation at 120 °C and a salinity of 60000 mg/L. Core flooding experiments have demonstrated the attractive EOR capabilities of Si-QDs, shedding light of the EOR mechanisms. Si-QDs effectively improve the wettability of rocks, enhancing the sweeping coefficient of injected fluids and expanding sweeping area. Within this sweeping region, Si-QDs efficiently stripping adsorbed oil from the matrix, thus increasing sweeping efficiency. Furthermore, Si-QDs could modify the state of pore-confined crude oil, breaking it down into smaller particles that are easier to displacement in subsequent stages. Si-QDs exhibit compelling EOR potential, positioning them as a promising approach for effectively developing tight oil reservoirs.

1. Introduction

The rapid increase in global demand for petroleum resources has presented significant challenges to the petroleum industry (Teklu et al., 2017; Wang et al., 2017). As oilfield development enters its mid-to-late stages, enhancing oil recovery from medium to high permeability reservoirs has become increasingly challenging (Neshat et al., 2018). The development of unconventional oil and gas resources holds paramount significance in supplementing oil and gas production (Liu et al., 2023, 2024). Tight reservoirs play a pivotal role in unconventional hydrocarbon production (Xiong et al., 2018). They are considered a strategic alternative to conventional oil and gas resources (Jia et al., 2018). Tight reservoirs are characterized by the presence of micro-pores (< 1 μm), low porosity (< 10%), and low permeability (< 0.1 mD) (Alharthy et al., 2013). During water flooding processes, the high flow resistance poses substantial challenges in supplying energy to the reservoir through water injection (Ye et al., 2019; Zhang et al., 2020). Traditional waterflooding approaches often fall short of achieving desirable results, resulting in a recovery rate as low as 10%–15% (Zhou et al., 2016). Consequently, effective strategies for enhancing the development of such reservoirs have been extensively

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researched in the field of petroleum engineering.

Chemical methods (Li et al., 2017; Liu et al., 2017), such as surfactant flooding, polymer flooding, gas flooding (Sheng, 2015), and foam flooding (Zhang et al., 2015), have been explored as alternative approaches. However, challenges encountered during the using of these approached, damage to formations from large polymer molecules, limited effectiveness of surfactants in a small near-wellbore area (Hirasaki et al., 2011; Zhang et al., 2016), gas channeling during gas EOR (enhanced oil recovery) (Duan et al., 2016; Ren et al., 2011), and potential reservoir damage from high injection pressures in foam flooding have been encountered (Yu et al., 2008). Consequently, there is a pressing need to explore novel EOR methods for efficient development of tight reservoirs.

Nanomaterials (El-Diasty and Ragab, 2013; Fakoya and Shah, 2017; Wang and Wu, 2013) have emerged as promising means for efficient petroleum development and have undergone extensive research (Liu et al., 2022; Suleimanov et al., 2011; Zhang et al., 2014). Nano-silica has shown ability in reducing interfacial tension and improving recovery rates (Wasan et al., 2011). Nanomaterial adsorption on rock surfaces enhances wettability and displacement efficiency (Almahfood and Bai, 2018; El-Diasty, 2015; El-Diasty and Aly, 2015). However, conventional nanomaterials face challenges in crossing the fine pores of tight reservoirs (Nourafkan et al., 2018; Nwidee et al., 2017). Meanwhile, tight reservoirs tend to exhibit high temperature and high salt characteristics (Aghaeifar et al., 2015; Zhong et al., 2019). Conventional nanomaterials tend to aggregate in such environments and reduce the effectiveness of their ability (Kim et al., 2015).

Quantum dot materials (Demir et al., 2011; Ihn et al., 2010), with their ultra-small size, present a novel solution to these challenges (De and Karak, 2017). Compared with conventional nanomaterials, quantum dots possess smaller volume and provide a larger surface area for modification. Moreover, their stability in high-temperature and high-salt environments makes it a promising candidate for efficiently developing tight reservoirs. Quantum dots are typically synthesized through small-molecule polymerization (De and Karak, 2017; Yang et al., 2015), resulting in a surface rich in grafting sites that can be functionalized to enhance specific performance aspects.

Quantum dot-based nanofluids synergistically enhance oil recovery through various mechanisms, with structural separation pressure and wettability improving being considered crucial mechanisms in quantum dot materials' EOR in tight reservoirs. Due to the increase in ordered entropy, wedge-like structures form between the nanofluid-oil droplet-solid interfaces, exerting forward separation pressure on oil droplets and facilitating the stripping of oil films from rock surfaces. The diffusion of nanofluids driven by structural separation pressure on rock surfaces promotes the improvement of rock wettability and further effectively removes oil films (Boda et al., 1999; Jiang et al., 2023; Karimi et al., 2012; Nazari et al., 2015). Abundant researches indicate that surface modification of quantum dot materials significantly impacts oil film detachment performance. Higher volume fractions, smaller quantum dot sizes, and higher surface charge densities all enhance the structural separation pressure of quantum dot materials, thereby improving oil film stripping performance (Chengara et al., 2004; Foroozesh and Kumar, 2020; Hendraningrat et al., 2013; Kondiparty et al., 2011).

In this study, a new type of nanomaterial silicon quantum dots (Si-QDs) was developed. A two-step synthesis process was employed to functionalize Si-QDs with benzenesulfonic acid groups. The EOR mechanism of Si-QDs was studied through microfluidic flow experiments and micro-CT scanning. Nanofluids based on Si-QDs exhibit excellent temperature and salt resistance. Compared to conventional water flooding, Si-QDs based nanofluids can achieve a 17% EOR effect. Si-QDs improve reservoir wettability, increase the sweeping coefficient of injected water, efficiently strip oil films from rock surfaces, and thus improve oil sweeping efficiency. They demonstrate attractive oil recovery performance. Si-QDs possesses ultra-small particle size (~3 nm), and demonstrates attractive EOR potential by improving the wettability and strip oil films from rock surfaces. Core flooding experiments showed promising EOR potential of Si-QDs in ultra-low permeability (0–1 mD) reservoirs, indicating that Si-QDs serve as a promising EOR agent for effectively developing tight reservoirs.

2. Experimental

2.1. Chemicals and materials

3-aminopropyl-trimethoxysilane (APTMS), sodium ascorbate (L-SA) and sodium para-amino benzenesulfonate were purchased from Shanghai Aladdin Biochemical Technology Co., Ltd. Glutaraldehyde was purchased from Sinopharm Chemical Reagent Co., Ltd. Outcrop sandstone scores were obtained from Hai'an Petroleum Scientific Research Instrument Co., Ltd. Ultra-pure water was prepared by using reverse osmosis unit (UPT-II-5T, Chengdu ULUPURE Ultrapure Technology Co., Ltd).

2.2. Synthesis of Si-QDs

Silicon quantum dots (Si-QDs) are prepared through a two-step synthesis method. Precursor A is prepared by hydrothermal synthesis of APTMS and L-SA in a round-bottom flask, while precursor B is obtained by stirring sodium para-amino benzenesulfonate and glutaraldehyde at room temperature. Si-QDs are then synthesized by stirring precursor A and precursor B at room temperature. Afterward, Si-QDs were purified and dried.

2.3. Characterization of Si-QDs

A high-resolution transmission electron microscope (HRTEM) was employed to examine the morphology of precursor A and Si-QDs. Fourier-transform infrared spectroscopy (FT-IR) and X-ray photoelectron spectroscopy (XPS) were utilized to analyze the synthesis of Si-QDs. A laser particle size analyzer was used to assess the dispersion stability of Si-QDs in water; three sets of data were collected for each test, with each set consisting of one hundred data points. A rotating drop interfacial tensiometer and a contact angle measurement device were employed to evaluate the oil displacement ability of Si-QDs. 2.5D homogeneous microfluidic module was designed to investigate the EOR mechanism of Si-QDs. Micro-CT scanning was conducted to investigate the EOR mechanism of Si-QDs, with scanning parameters set at 140 kV voltage, 130 mA current, and a resolution of 1 μm (see Fig. 1).

2.4. Core flooding tests

The experimental setup (Fig. 2) involved a displacement apparatus with two intermediate containers, employing three artificial cores cut from the same long core to ensure experimental accuracy. The cores were cut to approximate lengths to guarantee that they had very similar properties in all three sets of experiments. The physical properties of the cores are listed in Table 1. To facilitate the displacement process, a simulated oil mixture of kerosene and crude oil was used as the displacing oil phase, with the crude oil sourced from a Chinese oilfield. The viscosity of the simulated oil mixture was 2.15 mPa s at 25 °C, with a density of 0.78 g/cm^3.

The nanofluid based on Si-QDs and a commercial nanofluid were injected into the cores, and the enhanced oil recovery from

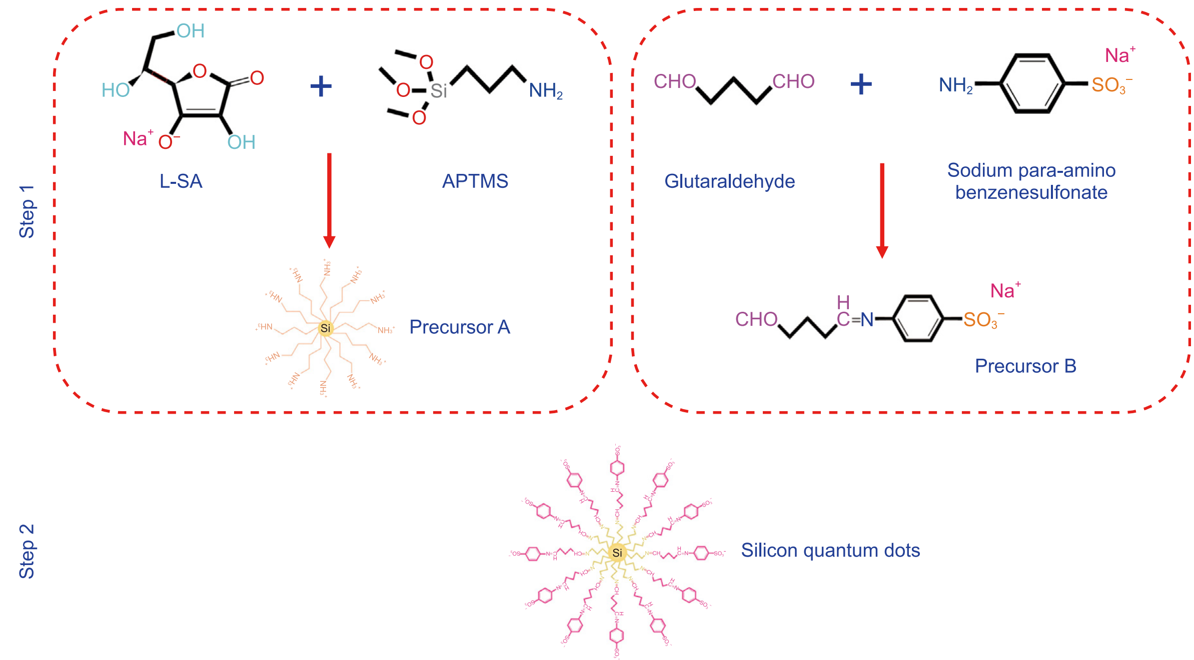

Fig. 1. Synthesis of silicon quantum dots.

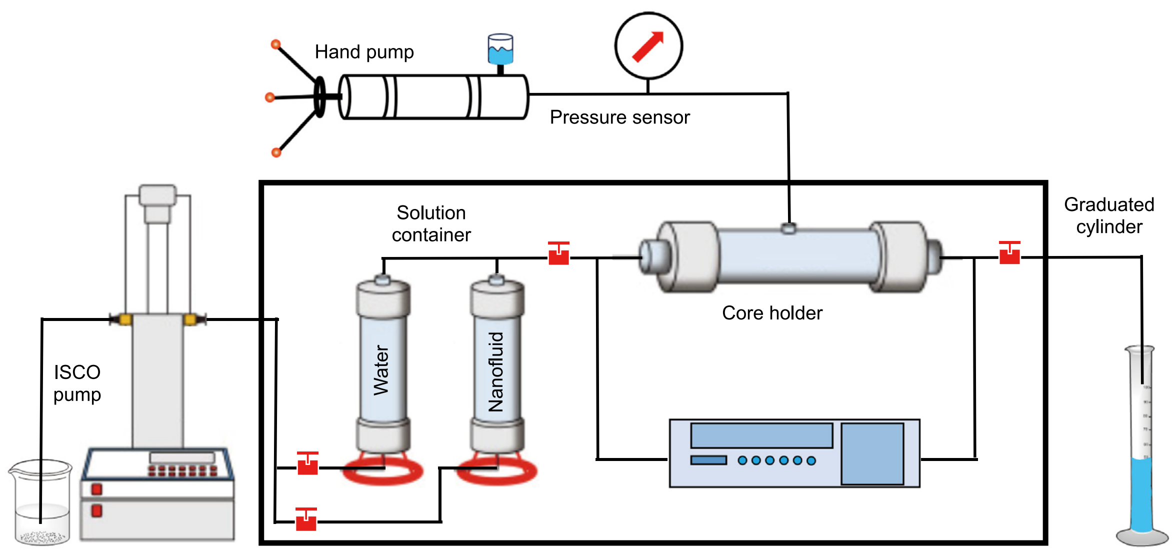

Fig. 2. Schematic diagram of core flooding device.

Table 1
Physical properties of the core samples.

Sample	Length, cm	Diameter, cm	Porosity, %	Permeability, mD	Pore volume (PV), cm^3
1	3.32	2.51	14.63	0.879	2.403
2	3.25	2.51	14.37	0.864	2.311
3	3.41	2.51	14.81	0.887	2.499

the cores was measured. The core displacement experiment proceeded with the following steps: core cleaning and drying, oil saturation, injection of a certain volume of brine into the core, followed by injection of a certain volume of nanofluid, and finally, injection of a certain volume of brine. The oil production volume was recorded, and the core's recovery factor was calculated.

2.5. Analytical methods

Si-QDs based nanofluids are considered to have attractive EOR potential, which was investigated by microfluidic (Fani et al., 2022; Gogoi and Gogoi, 2019; Xu et al., 2017; Yu et al., 2019) and CT approach in this study. Fluid flow channels were etched on the glass substrate using the principle of HF etching of glass, the oil phase was injected into the channels during the pre-experimental period, and the oil phase was driven using nanofluid after high temperature aging, the nanofluid-oil behaviors were observed using a microscope to study the mechanism of oil recovery of the Si-QDs. CT scanning utilized the different absorption and transmission of X-rays by water, oil, and rock solids. By scanning the target area, the pore fluid utilization was investigated. Scan the core before and after the use of nanofluid displacement to analyze the fluid distribution in the core and to form the oil recovery mechanism of the Si-QDs based nanofluid.

3. Results and discussion

3.1. Synthesis and characterization of Si-QDs

Precursor A and Si-QDs (Fig. 3) are the subjects of this section's research. The solution of precursor A appeared reddish-brown and emitted yellow-green fluorescence under 365 nm ultraviolet light, in agreement with previous reports. Si-QDs appeared deep red and turned into a red solid after purification and drying.

In the XPS analysis (Fig. 4(a)), both precursor A and Si-QDs exhibited peaks at C1*s*, O1*s*, N1*s*, and Si2*p*. By contrast, Si-QDs showed an additional peak at S2*p* due to surface modification. In the FT-IR analysis (Fig. 4(b)), both precursor A and Si-QDs displayed peaks around 3400, 1640, 1030, 1130, and 2930 cm^{-1}, which could be attributed to NH_2 amino groups, Si—O—Si silicon-oxygen bonds, and $-CH_2-$ methylene groups. Furthermore, the FT-IR spectrum of Si-QDs clearly indicated the presence of signals corresponding to the benzenesulfonic acid group (820 and 1350 cm^{-1}) (Coates, 2000; Von et al., 2000), confirming the grafting of benzenesulfonic acid groups onto the surface of precursor A.

Fig. 5 shows the microstructures of precursor A and Si-QDs. Precursor A, after surface functionalization, exhibited a slight increase in particle size. In the HRTEM images, precursor A exhibited a tendency to aggregate, while Si-QDs, which underwent surface functionalization with benzenesulfonic acid groups, demonstrated significantly improved dispersion stability.

3.2. Concentration optimization of Si-QDs base nanofluid

The improvement of wettability and the stripping of oil film are the main EOR mechanisms of nanomaterials. To determine the optimal concentration of Si-QDs, the wettability improvement effect

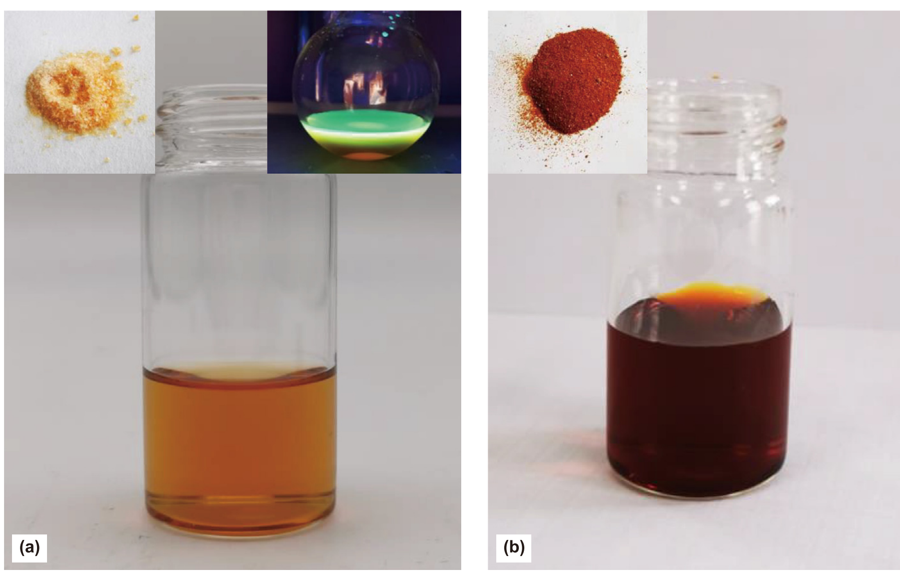

Fig. 3. **(a)** Aqueous solution, solid particles and fluorescence of precursor A; **(b)** aqueous solution and solid particles of Si-QDs.

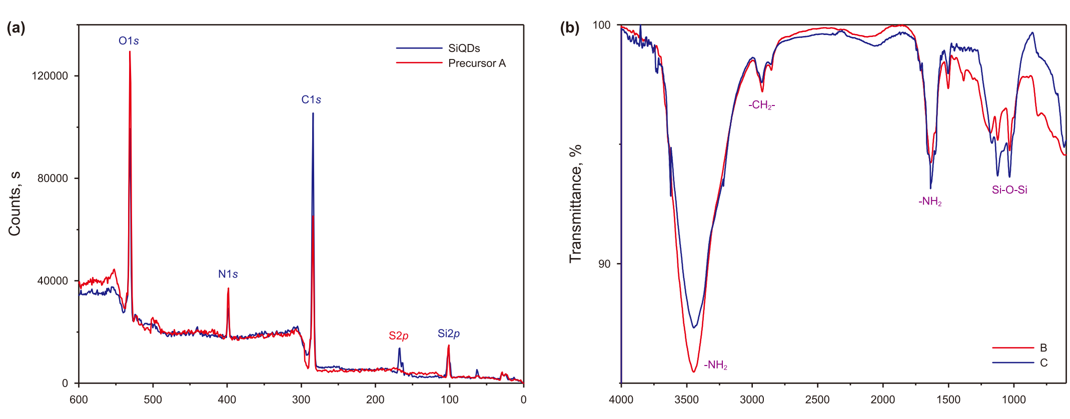

Fig. 4. **(a)** XPS photoelectron spectra; **(b)** FT-IR spectra.

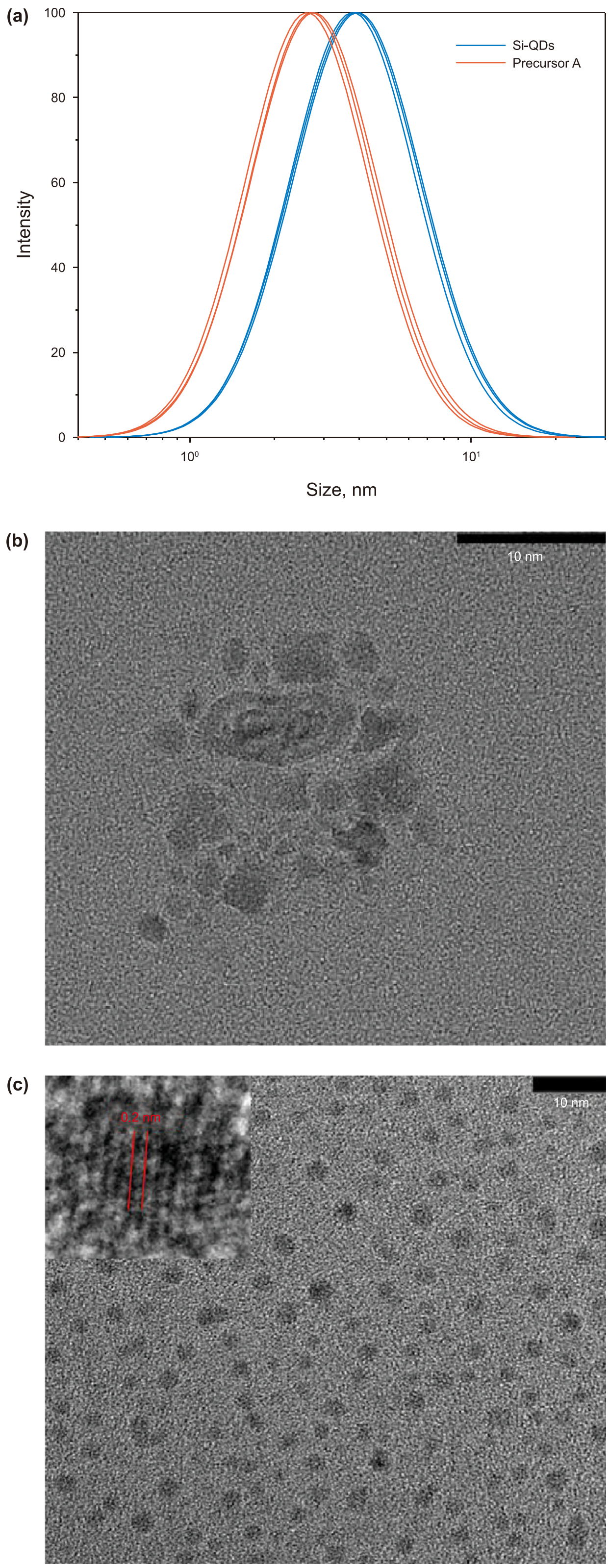

Fig. 5. **(a)** DLS size measurement results; **(b)** HRTEM image of precursor A; **(c)** HRTEM image of Si-QDs.

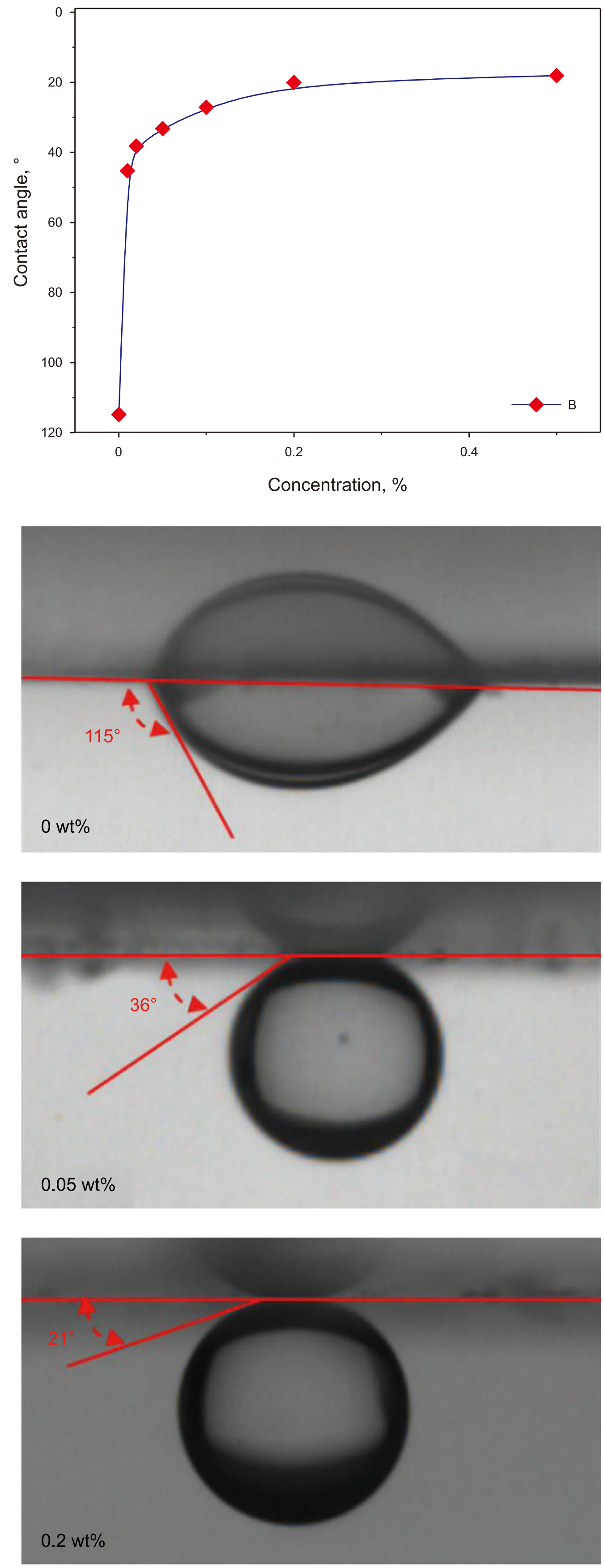

Fig. 6. Wettability improvement effect of Si-QDs at different concentrations.

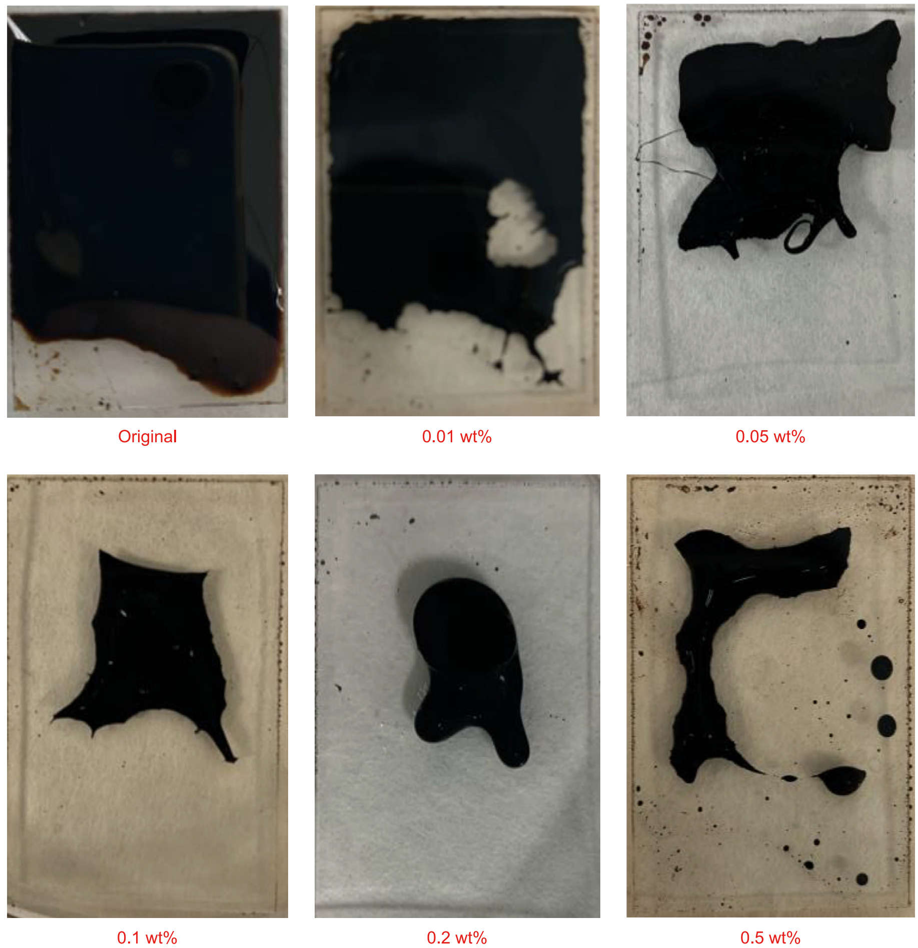

Fig. 7. Experimental results of oil washing.

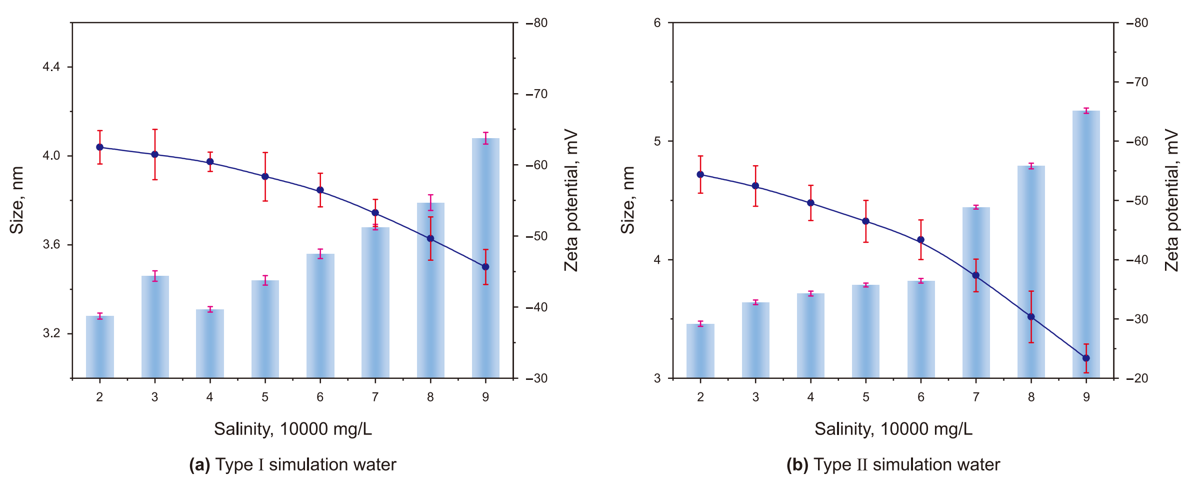

Fig. 8. Experimental results of temperature and salt resistance.

and the oil film peeling effect were used as evaluation indexes to optimize the use concentration of Si-QDs. Quartz slides were hydrophobically modified and then treated with different concentrations of Si-QDs for 24 h, as shown in Fig. 6. The oil film washing efficiency of different concentrations of Si-QDs was studied using quartz slides covered with heavy oil. The use of heavy oil ensures the stability of the oil on the surface of the quartz slide during the experiment. Quartz slides were immersed in Si-QDs with different concentrations, and the oil film washing efficiency was recorded after 24 h. The experimental results are shown in Fig. 7.

Fig. 6 illustrates the results of different concentrations of Si-QDs on improving wettability, with Si-QDs effectively changing

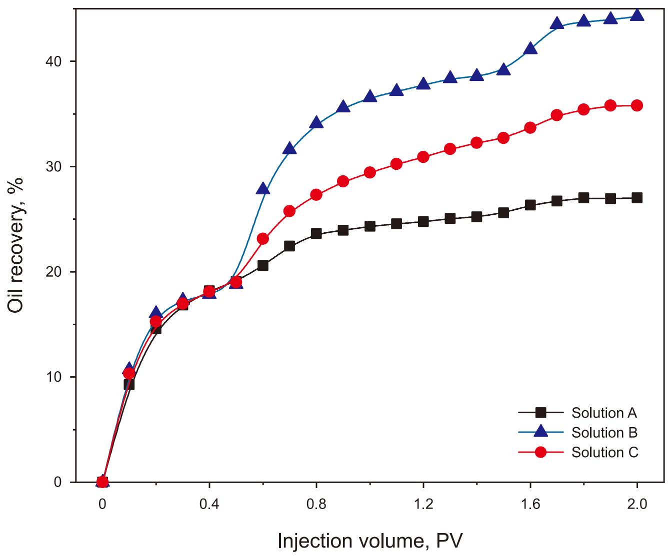

Fig. 9. Dynamic production curve of low-permeability core sample at 80 °C.

wettability from oil-wet to water-wet. The higher the concentration of Si-QDs, the more hydrophilic the quartz slides were after the experiment.

As is shown in Fig. 7, the oil-washing effects of nanofluids vary with different concentrations. At a concentration of 0.2 wt%, a significant portion of the oil phase on the quartz can be washed off. As the concentration increases to 0.5 wt%, there is no significant change in the oil-washing effect. This observation, combined with wettability improvement experiments, led to the determination that the optimal Si-QDs concentration is 0.2 wt%.

3.3. Stability evaluation of Si-QDs based nanofluid

The stability of nanofluids prior to injection into reservoirs is crucial for their effective performance in the formation. Conventional nanomaterials (aluminum oxide and silica) tend to aggregate in high salinity reservoirs and do not provide satisfactory results for EOR. To evaluate the stability of Si-QDs in high temperature and high salt conditions, two types of simulation water were used to conduct temperature and salt resistance stability experiments: type I only contained Na^+ and Cl^-, and type II contained Ca^{2+}, Na^+ and Cl^-, and Na^+:Ca^{2+} = 9:1. After placing the Si-QDs in a constant temperature oven at 100 °C for 7 days, the particle size and zeta potential were measured.

Fig. 8(a) illustrates the stability of Si-QDs in Type I simulation water. The results indicate excellent temperature and salt resistance of Si-QDs in Type I simulation water. With increasing salinity, there is a slight increase in particle size, and a certain decrease in zeta potential is observed, but it remains above −45 mV (highly stable). Fig. 8(b) presents the stability of Si-QDs in Type II simulation water. It can be observed that Si-QDs exhibit good temperature and salt resistance when the salinity is less than 6 w. During this process, there is a slight decrease in zeta potential; however, when the salinity exceeds 6 w, there is a significant increase in particle size of Si-QDs, accompanied by a substantial decrease in zeta potential, indicating a transition from stability to instability in the nanofluid.

The electrostatic repulsion between nanoparticles is considered a source of stability in nanofluids. During the modification process of quantum dot materials, there is a redistribution of charge on the surface of the quantum dots (Wu et al., 2023). The benzenesulfonic acid groups possess strong electronegativity, leading to a greater net negative charge on the quantum dots, thus enhancing the electrostatic repulsion between Si-QDs and improving the stability of the nanofluid. In a saline environment, positively charged ions tend to aggregate near the quantum dots, and higher temperatures accelerate this aggregation process. The aggregation of positively charged ions leads to a decrease in electrostatic repulsion, resulting in a decrease in system stability at a macroscopic level. Calcium ions carry more net positive charges compared to sodium ions, therefore, under the same salinity conditions, calcium-containing saline water has a greater impact on the stability of nanofluids compared to sodium-containing saline water.

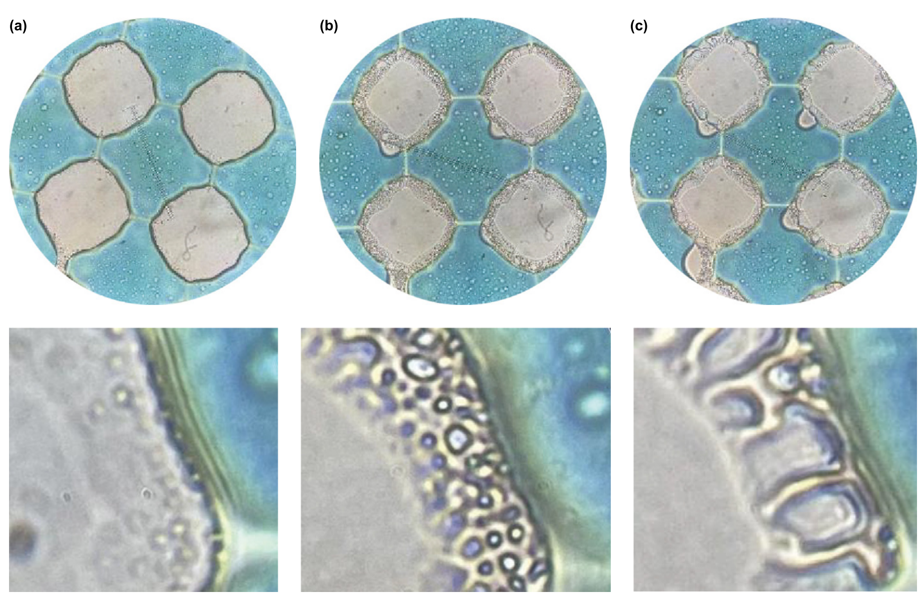

Fig. 10. Oil sweeping in microfluidic module: **(a)** 1 d; **(b)** 5 d; **(c)** 9 d.

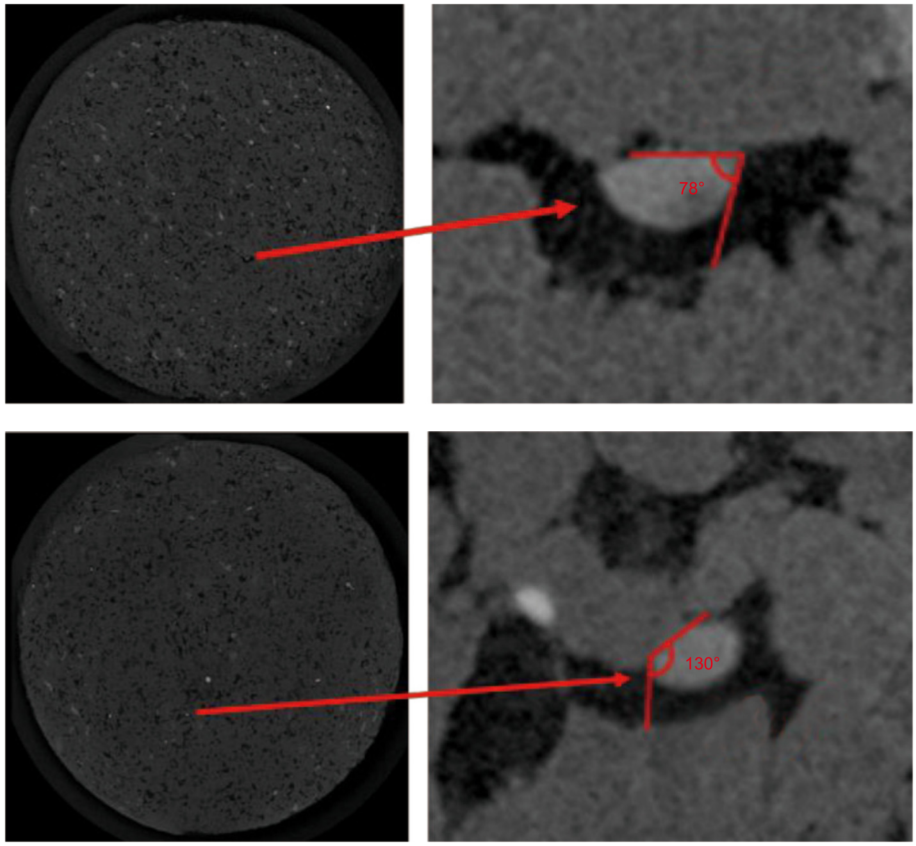

Fig. 11. Oil-water contact angle of core injection end and production end.

3.4. EOR potential of Si-QDs based nanofluid

Three different displacement fluids were prepared for the experiment: A (brine), B (0.2 wt% nanofluid based on Si-QDs), and C (0.2 wt% commercial nanofluid). Fig. 9 shows the core flooding experiment results. After displacing with 0.5 PV of brine, the oil recovery percentages for the three sets of cores were 19.07%, 18.98%, and 18.77%, respectively. Subsequently, after displacing with 1 PV of experimental solution, the oil recovery percentages increased by 6.51%, 20.32%, and 13.72% for the three fluids. Following a subsequent 0.5 PV water flooding, the oil recovery percentages for the three fluids increased by 1.43%, 5.17%, and 3.07%, respectively. The final recovery rates for three types of fluids are 27.01%, 35.77% and 44.26%, respectively. When compared to brine and commercial nanofluid, the nanofluid based on Si-QDs effectively improved core oil recovery.

3.5. Analysis of EOR mechanism

After injecting the fluid into the reservoir, the sweeping area and sweeping efficiency both influence the EOR capability of the injected fluid. We studied the behavior of the nanofluid within a 2.5D module and the fluid occupancy states in the core before and after nanofluid flooding, summarizing the EOR mechanisms of the Si-QDs based nanofluid. The research results indicate that the Si-QDs based nanofluid exhibits multiple EOR mechanisms.

After saturating the microfluidic model with red-dyed oil phase (a mixture of kerosene and mineral oil), the nanofluid, prepared using blue-dyed ultrapure water, was injected uniformly into the model. Subsequently, the model was placed under a microscope for observation. Fig. 10 illustrates the dynamic distribution of oil and water within the model at different time intervals. With the continuous injection of the nanofluid, the oil phase adsorbed on the model matrix is stripped away, leading to the formation of noticeable large oil droplets in the image. The effective removal of the oil phase adsorbed on the matrix, thus enhancing the oil displacement efficiency, is a significant EOR mechanism of Si-QDs.

The initial core was scanned to obtain the baseline rock structure. Subsequently, after saturating the rock core with oil, another scan was performed to capture the initial fluid distribution. Following this, the nanofluid based on Si-QDs was injected into the rock core until oil production ceased at the outlet end. Subsequent scans of the rock core were conducted. Due to limitations in scanning device, the CT equipment couldn't entirely capture the oil-water distribution within the standard-sized rock core. As a result, small samples were taken from both the injection end and the outlet end for reconstruction and modeling purposes.

Fig. 11 illustrates the oil-water contact angles at the injection end and the production section. At the end where the nanofluid displacement experiment concluded, the rock surface at the injection end had prolonged contact with the nanofluid, resulting in a hydrophilic contact angle. Conversely, in the production section, this region was not effectively influenced by the nanofluid, exhibiting hydrophobic behavior. Wettability is the property of fluid diffusion on a solid surface, and nanoparticles stabilize the water layer through adsorption on the surface of the rock, so that the wettability of the rock changes to water-wet. In this process, the wedge-shaped structure formed between the nanofluid, the oil phase and the rock propel the diffusion of the nanofluid on the rock surface and improves the wettability improvement (Karimi et al., 2012; Nazari et al., 2015; Nikolov et al., 2010; Zhao et al., 2022).

Subsequently, the CT scan results were used to identify the

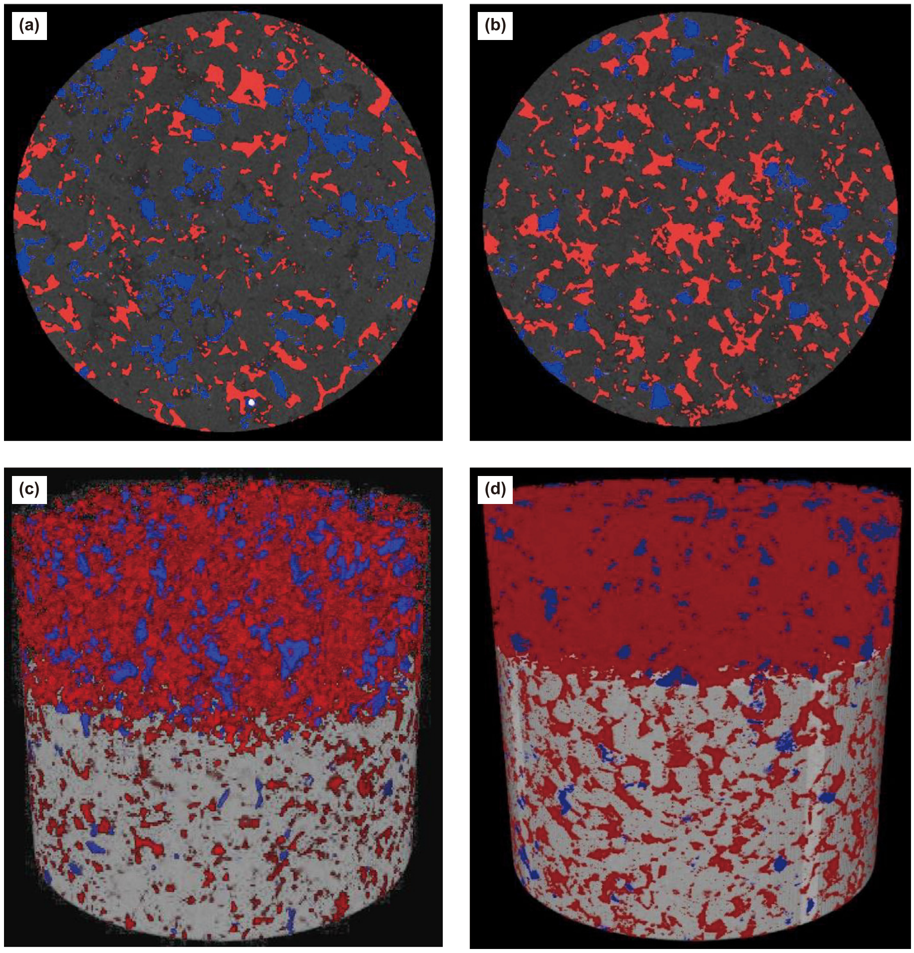

Fig. 12. Oil-water distribution: **(a)** injection end; **(b)** production end.

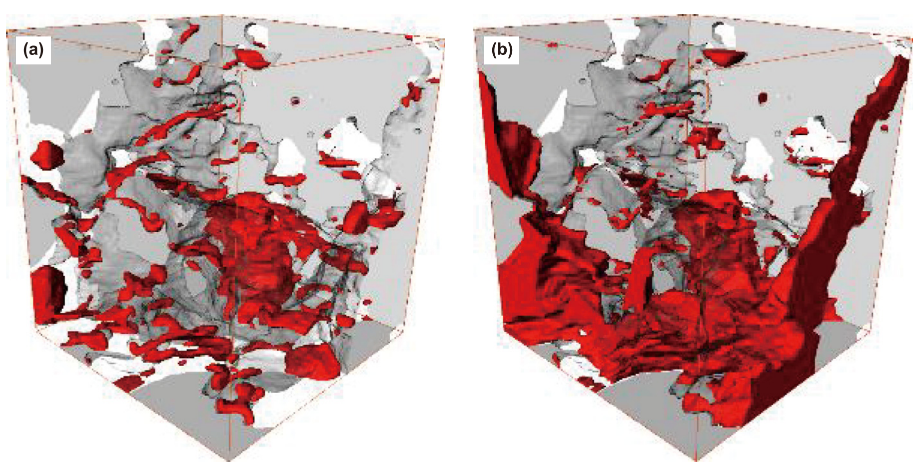

Fig. 13. 3D-grid of residual oil distribution: **(a)** injection end; **(b)** production end.

distribution of oil and water, followed by three-dimensional reconstruction. Fig. 12 displays the oil-water distribution from the CT scan results and the three-dimensional construction. At the injection end, the nanofluid's influence effectively displaced the oil phase within the region. The areas occupied by the oil phase (identified in red) and the water phase (identified in blue) were fairly balanced, indicating a high displacement efficiency. In contrast, at the production section, the injected nanofluid had minimal influence on this area, with only a small portion of the oil phase being displaced. In the three-dimensional construction results, the red-colored oil phase dominated majority of the area.

Next, a 10-μm-sided box was extracted from the three-dimensional construction results to zoom in on the distribution of the remaining oil. Fig. 13 displays the distribution of remaining

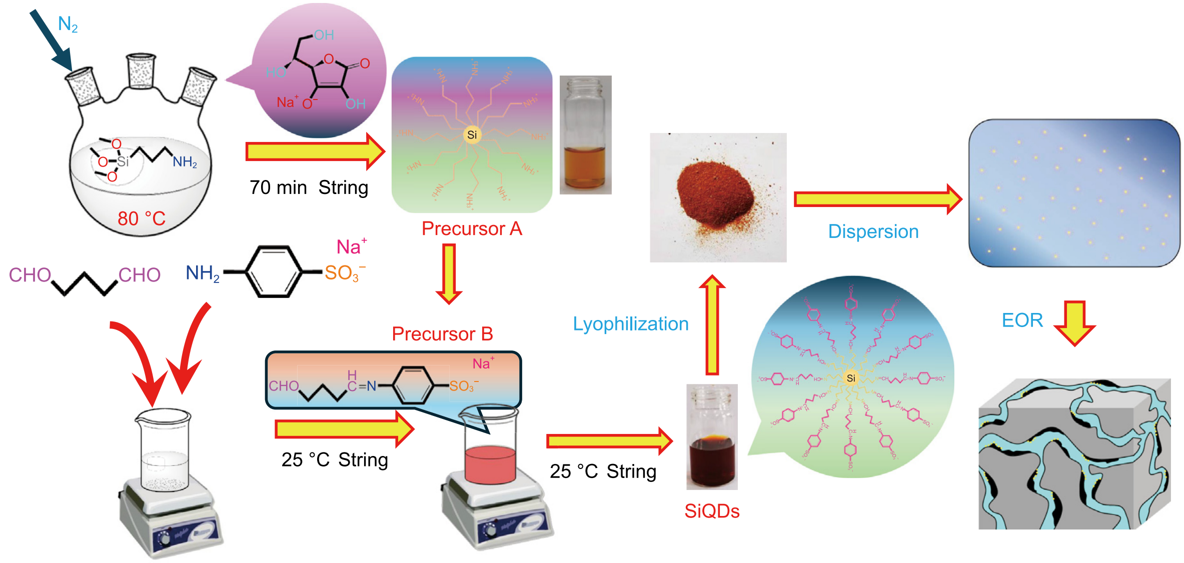

Fig. 14. Si-QDs based nanofluid for effective EOR in tight reservoir.

oil at the injection and production ends. At the production end, the nanofluid couldn't effectively displace or influence the crude oil in this area. After displacement, the oil formed large, clustered regions that were difficult to displace further, resulting in residual oil. At the injection end, the nanofluid's influence effectively displaced the crude oil within the region. Simultaneously, it improved the remaining oil's distribution, making it easier to displace in subsequent stages. The increased recovery rate in the third stage of the core flooding experiment confirmed this mechanism.

4. Conclusions

This study synthesizes Si-QDs using a two-step method and shows promising EOR potential. The main conclusions are listed as follows (Fig. 14):

1. Si-QDs exhibit ultra-small dimensions (~3 nm) and show excellent temperature and salinity resistance in high-temperature, high-salinity conditions.
2. Compared to conventional water flooding, nanofluids based on Si-QDs can improve oil recovery rates by 17%, surpassing the 8% improvement compared with commercial nanomaterials.
3. The adsorption of nanomaterials improves rock wettability, increasing the sweeping area of injected water. Simultaneously, Si-QDs effectively sweeping oil from rock surfaces, thereby enhancing oil displacement efficiency.
4. CT scan results indicate that nanofluids can disperse large chunks of crude oil within pore spaces, facilitating easier displacement in subsequent stages and further improves the EOR effectiveness of Si-QDs.

CRediT authorship contribution statement

Cheng Liu: Writing – review & editing, Writing – original draft, Resources, Investigation, Data curation. **Biao Zhou:** Resources, Methodology, Investigation. **Bing-Shan Wang:** Resources, Project administration, Methodology, Investigation. **Huan Wang:** Software. **Qing You:** Validation, Supervision, Funding acquisition. **Guang Zhao:** Project administration, Methodology. **Cai-Li Dai:** Visualization, Validation, Resources.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Qing You reports financial support was provided by National Natural Science Foundation of China. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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