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Experimental Investigation about Oil Recovery by Using Low-Salinity Nanofluids Solutions in Sandstone Reservoirs

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Abstract: Production of crude oil from matured oil reservoirs has major issues due to decreased oil recovery with water channeling; however, the low-salinity water flooding technique is more commonly used to maximize recovery of the remaining oil. In this study, we demonstrated a new hybridization technique of combining low-salinity water and nanofluids; this was achieved by using experiments such as contact angle measurement with water of different salinity levels and nanofluid concentrations, core displacement, and NMR (nuclear magnetic resonance) between low-/high-permeability rock. The trial results demonstrated that the test with KCl-1+NF outperformed those with other compositions by changing the original contact angle from 112.50° to 53.3° and increasing formation production up to 15 cc. In addition, we saw that when 2 PV of KCl-1+NF was injected at a rate of 5 mL/min, the middle pores' water saturation dropped quickly to 73% and then steadily stabilized in the middle and late stages. Regarding the novel application of the hybridization technique, the insights presented in this paper serve as a helpful resource for future studies in this field.

Keywords: low-salinity water; silica nanofluids; EOR; wettability alteration; NMR

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1. Introduction

According to forecasts that global energy consumption will go up by roughly 50% over the next 20 years, the world's energy demand is likely to continue rising in the future. The oil and gas industries are required to find a cost-effective strategy to recover remaining oil from reservoirs to fulfill the increasing global energy demand [1]. To extract the oil that primary or secondary recovery methods were unable to produce, EOR (enhanced oil recovery) techniques are utilized. The largest oil reserves in the world are found in sandstone reservoirs. A sandstone reservoir is a layer of sandstone that contains petroleum that may be extracted using existing technology. Sandstone reservoirs often contain fluid-filled pores, stable minerals, and supplemental minerals. Grains of sand must range in size from 1/8 to 2 mm for a rock to be referred to as sandstone [2–7]. Compared to oil-wet reservoirs, recovering oil from water-wet reservoirs is far simpler [8]. EOR is attracting increasing interest, since oil is in greater demand than ever before, hence the need to increase oil production [9].

Nanofluids, which are nanotechnology-based fluids containing nanoparticles, have emerged as a promising solution for enhanced oil recovery. The advancement of nanotechnology has led to the introduction of nanofluids as an economical, effective, and environmentally friendly replacement for existing chemicals [10]. Nanofluids might interact with the rock surface by chemical and physical mechanisms when they are injected into an oil reservoir. The rock's wettability can be altered from oil-wet to water-wet or intermediate-wet by the nanoparticles adhering to the surface and forming a thin layer. Better oil recovery is encouraged by this change in wettability because it strengthens the capillary forces between the fluid and the rock. A nanoparticle is one with a diameter of 1–100 nanometers (nm) [11]. A technique called "nanofluid flooding" is used in EOR operations to increase the efficiency of oil recovery by dispersing nanoparticles in a fluid and injecting them into oil reservoirs [12]. When creating a nanofluid, selecting the right nanoparticles is essential. For example, silica nanoparticles have demonstrated encouraging outcomes in modifying wettability and enhancing oil recovery. Because of their large surface area and ability to absorb the rock surface, these nanoparticles can facilitate oil displacement by lowering the interfacial tension between water and oil. Through several mechanisms, such as decreased interfacial tension and altered wettability, nanofluid flooding can enhance oil recovery. During the reduction of interfacial tension, the tension that exists between the oil and the injected nanofluid can be lessened by the presence of nanoparticles dispersed throughout the fluid. By lowering the interfacial tension, the oil can be more efficiently displaced by the nanofluid, increasing oil recovery. When oil and water come into contact, the nanoparticles group together to produce a stable layer that helps release trapped oil. The fluid's contact angle with the reservoir rock surface can be altered by nanoparticles. Wetness conditions may shift from oil-wet to water-wet or intermediate-wet as a result of this shift [13].

For decades, it was considered that injecting anything other than saline water into a reservoir could permanently destroy it, but we noticed that by reducing the salinity of injected water, trapped oil molecules can be more easily released from the surface of rock [14]. At the moment, the primary problem associated with enhanced oil recovery is wettability. The goal of the suggested EOR technique was to lower the oil's affinity and increase oil recovery by turning the reservoir from an oil-wet state to a water-wet. Wettability is important because the amount of oil recovered from water-wet reservoirs is higher than that from oil-wet reservoirs. Low-salinity water (LSW) flooding is a novel approach that has been effectively applied in several industries. It can convert an oil-wet sandstone reservoir to a water-wet one by changing the wetting characteristics of the reservoir rock. One of the most crucial factors to take into account is the ionic concentration and composition of low-salinity water. In our research, we demonstrated that oil molecules are bonded to clay particles on the surface of rock with the help of divalent cations. The electrical force of highly ionically concentrated saline water will force divalent cations to the clay surface [15]. If the salinity of water is reduced, divalent cations can expand, and mono-valent ions can access and replace their places [16]. LSW flooding is a new technology that has been successfully employed in various areas and affects the reservoir rock's wetting properties, thus increasing oil recovery; it can turn an oil-wet sandstone reservoir into a water-wet one [17]. The ionic composition of injected water is one of the main parameters to be considered. A study revealed deionized water has more potential to be used as an EOR technique than regular seawater. By injecting the right concentration of LSW, we can change the wettability of the reservoir [18].

To address the limitations of low-salinity water flooding, we propose a hybrid technique that combines low-salinity water flooding with nanofluids. This approach in EOR can improve the disadvantages of LSW flooding such as water channeling and improve sweep efficiency during flooding operations [19]. The nanofluids can modify fluid–rock interactions, while LSW can alter the wettability and promote spontaneous imbibition, leading to higher oil recovery [20]. LSW flooding can change the wettability of the rock surface by making it more favorable for displacement [21]. Nanofluids, on the other hand, can further enhance wettability alteration by providing additional surface activity through nanoparticle adsorption and interaction with the rock surface [22]. This combined effect can lead to improved oil mobilization and displacement [23].

In this study, we used sandstone rocks as the research object, and the treated thin slices were immersed in formation crude oil to form an oil film. The wettability angle of the thin slices' surface in both LSW solution and pure water at different times was measured. The formation of an oil film on the sandstone rock surface and the mechanism of wettability alternation were explained. We concluded that LSW-based NF flooding has many advantages for use as an EOR technique. Below, we present all the needed data and the method of our investigation.

2. Experiments

2.1. Experimental Materials

We used Berea sandstone core samples provided by Shengli Oilfield (Dongying, China) during these experiments. The specifications of core samples are shown in Table 1.

Table 1. Core sample properties.

Formation	Average Permeability (mD)	Average Porosity (%)	Dimensions	
			Diameter (mm)	Thickness (mm)
Sandstone	560	26	25	2~4

X-ray diffractometer (XRD) and core analyses were provided by Shengli Oilfield (Dongying, China) specialists; details are summarized in Table 2.

Table 2. XRD mineral composition test results.

SiO_2	Al_2O_3	FeO/Fe_2O_3	TiO_2	CaO	MgO	Alkalis	H_2O	N/D	Total
85.38	6.47	1.23	0.75	1.35	0.20	2.33	1.53	0.76	100

The density of light crude oil is 0.85 g/cm^3 with a viscosity of 65 cp; our core samples were acquired from a heavy oil reservoir (15 MPa, 80 °C) in Shengli Oilfield, Dongying, Shandong, China. Detailed specifications are listed in Table 3.

Table 3. Crude oil properties.

Gravity (API)	TAN (mg/gm)	Kinematic Viscosity (cSt)	Initial Oil Saturation (%)
20.6	0.8	325	57

The different salinity levels of KCl were compounded, and named KCl-1 (TDS = 500 mg/L) and KCl-2 (TDS = 6500 mg/L). Low-salinity formulations are listed in Table 4.

Table 4. Brine formulations.

Brine	K^+	Cl^-	TDS, mg/L
KCL-1	262.2	237.8	500
KCL-2	3408.6	3091.4	6500

The nanoparticles that we used are mono-dispersed commercial hydrophilic silica nanoparticles (SiO_2) of 99% purity. The surface area of SiO_2 is 180–600 m^2/g, and average particle size ranges from 20 nm to 30 nm.

2.2. Experimental Apparatus and Main Procedures

We divided our experiment into two main steps. The first step is to obtain the wettability measurements of rock core samples by using the contact angle method with different solutions including saline solutions, nanofluids, and a mixture solution of salt and nanofluids. The second step is to observe the effects of the hybridization on EOR, based on the core displacement results from nuclear magnetic resonance. The core displacement experiments provide data on breakthrough times, flow rates, and the displacement efficiency of various compositions. The fluid distribution and saturation changes that occurred within the sandstone cores during the displacement process could be seen and characterized thanks to the NMR analysis. This study verified the quick decrease in water saturation and the stabilization that followed, which showed that the oil had been successfully displaced. The details on each step can be found below.

2.2.1. Contact Angle Measurement

A commonly used method for determining the wettability characteristics of a solid surface by dripping a liquid droplet is contact angle measurement. It offers important insights regarding the interaction between liquid and solid at the interface, and is especially pertinent when researching phenomena associated with rock wetting. Rock wettability also can be measured by using the contact angle value. Angle (θ) created at the contact line of the liquid–fluid interface and the solid substrate is typically used to calculate the contact angle [24]. In applications involving oil recovery, the contact angle of a droplet of water or oil is measured for a rock surface that has already been treated with the substance anticipated to have an impact on the wettability [25–27]. The experimental steps were as follows and shown in Figure 1:

1. We polished the sandstone rock slices and washed and dried them with deionized water, immersed the slices in light crude oil, and then placed them inside a drying oven at 60 °C for 10 days. After taking out pieces from the oil, we rinsed them quickly with N-Heptane (C_7H_{16}). We then left them to air dry at room temperature for 1 day. We used contact angle measurement equipment to measure the wetting angle;
2. At room temperature, we prepared KCl LSW by adding 500 mg/6500 mg KCl to 1 L of deionized water. The solution is stirred for 1 h by using a magnetic stirrer to make sure that all KCl was fully dissolved in the deionized water. For the SiO_2 solution, we added 4 g of SiO_2 nanoparticles to 1 L of deionized water. The solution was stirred for 1 h using a magnetic stirrer, and after that, we used an ultrasonic cleanser to fully break up and dissolve all the formed SiO_2 clumps. Then, the rock slices were immersed in the solutions, and we then took pictures of the wetting angle at 6 h, 12 h, 24 h, 36 h, 48 h, 60 h, and 72 h timepoints;
3. Using contact angle measuring equipment, we measured the wetting angle for each solution at different times;
4. After obtaining the best result from the LSW and NF solutions, we mixed them to form a mixed hybrid solution. We placed the oil-wet rock slices in the solutions, and then took pictures of the wetting angle at 6 h, 12 h, 24 h, 36 h, 48 h, 60 h, 72 h timepoints;
5. To summarize, oil-wet rock slices were soaked in different solutions. By measuring the wetting angle and recording the difference in the change in the angles, we analyzed and compared them. Using, Excel v.2021 and Origin v.2022 we obtained post-processing results.

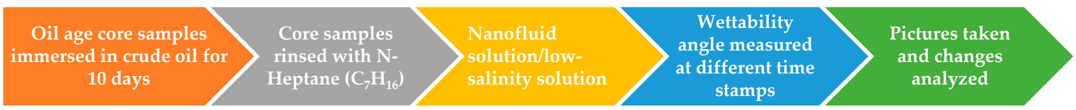

Figure 1. Wettability measurement experiment flow chart.

2.2.2. Core Displacement

A core displacement experiment is a laboratory method that we used to model and examine fluid flow and displacement processes in a porous medium. It is sometimes referred to as a displacement test or core flood test. To investigate the displacement efficiency, fluid flow behavior, and several factors influencing fluid–fluid and fluid–rock interactions, one fluid (LSW, NF, and a mixed hybrid solution) must be injected into a prepared core sample saturated with another fluid (light crude oil). The detailed core flooding experimental steps are as follows and shown in Figures 2 and 3:

6. Core Preparation: Cylindrical rock samples, known as core plugs, are extracted from reservoir rocks and prepared for experimentation. The cores are cleaned, dried, and

saturated with light crude oil. Saturation is carried out by using kerosene. First, we vacuum-cleaned rock cores to be sure that any other foreign matter was not left in the rock pores. After that, we saturated them with kerosene at 5 MPa for 1 day.

7. Saturation and Injection: Core plugs were placed in a core holder, and the desired fluid (low-salinity water, nanofluid, and mixed hybrid solution) was injected into the core with a certain pressure and flow rate. The injection was performed using a displacement pump, also known as an ISCO pump.
8. Fluid Flow Monitoring: To evaluate fluid flow behavior, saturation profiles, and recovery efficiency during the injection process, injected fluid volumes and the pressure drop of the core were tracked. These data aid in assessing how well flooding with nanofluid and low-salinity water works to remove oil from the core.
9. Recovery and Analysis: After the displacement process, the core was extracted, and the residual oil saturation was measured. Additional analysis, such as fluid composition analysis or imaging techniques, can be performed to further evaluate the fluid–rock interactions and recovery mechanisms.

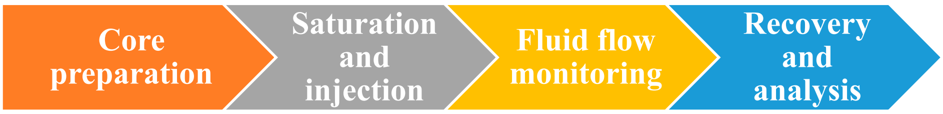

Figure 2. Core displacement experiment flow chart.

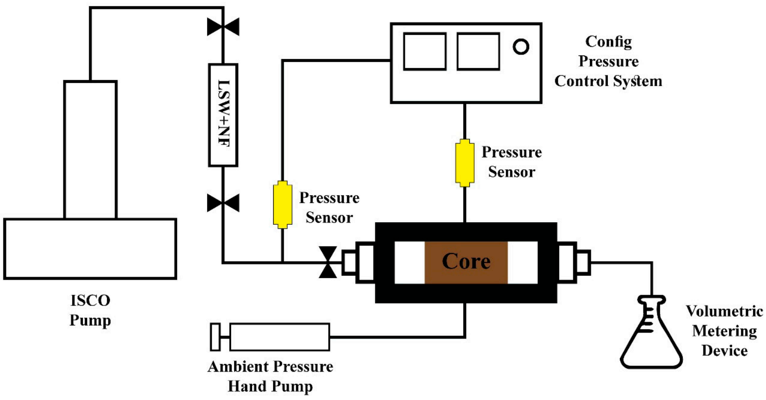

Figure 3. Core displacement experiment scheme.

2.2.3. NMR (Nuclear Magnetic Resonance)

Reservoir rocks and other porous media can be studied using NMR, an effective method for studying fluid behavior and interactions [28]. NMR provides insights into fluid distribution, pore size distribution, fluid-phase characterization, and wettability [29]. We used $MnCl_2$ solution for NMR spectroscopy instead of distilled water, because manganese chloride solution is a frequent paramagnetic shift reagent in NMR spectroscopy. Paramagnetic shift reagents are chemicals that interact with the NMR instrument's magnetic field, causing a change in the chemical shift of adjacent nuclei, resulting in a shift in the NMR [30]. The $Mn_2{}^+$ ion in manganese chloride has an unpaired electron, making it paramagnetic. When $Mn_2{}^+$ ions are given to a sample, they interact with the magnetic field of the NMR instrument, causing a paramagnetic shift in adjacent nuclei such as proton (1H) and carbon-13 (13C). This shift can help to improve the resolution and sensitivity of the NMR spectra by enhancing the NMR signals of these nuclei. Manganese chloride is a popular paramagnetic shift reagent in NMR spectroscopy because it is widely available, reasonably priced, and gives strong signal enhancement for a wide variety of nuclei [31]. The detailed experimental steps of NMR are as follows and shown in Figure 4:

10. Sample Preparation: Similar to the core displacement method, rock core samples were extracted and prepared for experimentation. The cores were cleaned, dried, and saturated with kerosene. For NMR, we used unsaturated dry rocks and rocks

saturated with kerosene and deionized water, first to observe all rock information, and then to observe the difference they make.

11. NMR Measurements: The saturated cores were placed within an NMR instrument, and NMR measurements were performed. NMR signals were generated by the hydrogen nuclei (protons) present in the fluids within the rock pores.
12. Fluid Characterization: Information on fluid-phase characteristics, including fluid composition, saturation, and distribution, was provided by NMR. It helps in understanding how low-salinity water, nanofluids, and mixed hybrid solutions interact with the reservoir rock, and how they affect the wettability and displacement of oil.
13. Time-Lapse Monitoring: NMR can also be used for time-lapse monitoring to observe fluid displacement and flow processes within rock cores. This helps in tracking changes in fluid saturation, flow patterns, and recovery efficiency during low-salinity water, nanofluid, and mixed hybrid solution flooding experiments.

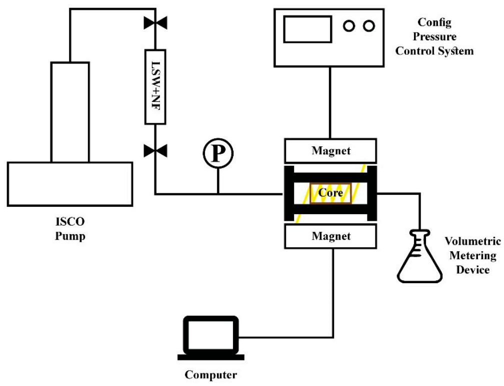

Figure 4. Schematic diagram of NMR experimental setup.

3. Results

3.1. Wettability Changes

The contact angle of the rock sample progressively rose, and its development rate was rapid, as seen in Figure 5. The wetting angle increment was mild and stabilized after the seventh day. As the wetting angle shifted from the original 22.5° to 118°, the rock sample's surface changed from water-wet to oil-wet.

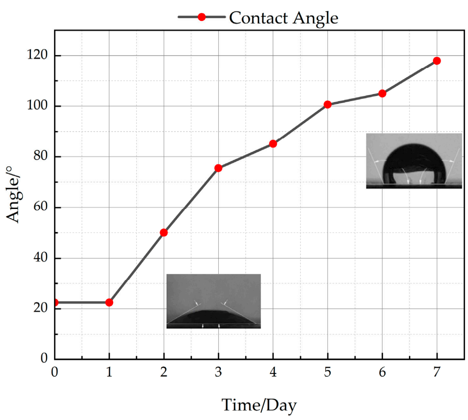

Figure 5. Progression of the static contact angle of rock sample over time (days).

After that, we treated rock samples with pure water, KCL-1, and KCL-1+NF solutions. As shown in Figure 6, it is possible to see how low salinity affects the change in wettability. Figure 6a shows the effect of pure water, Figure 6b the effect of KCL-2 solution, Figure 6c that of KCL-1 solution, and Figure 6d that of the mixed KCL-1 and NF solution. The experiment's findings showed that as opposed to pure water, KCl changed the contact angle by making it more water-wet. The combination solution including KCL-1 and NF showed the biggest alterations in wettability, followed by KCL-1 solution. The KCL-1+NF's contact angle showed a notable change in wettability, changing the initial angle from 112.50° to 53.3°.

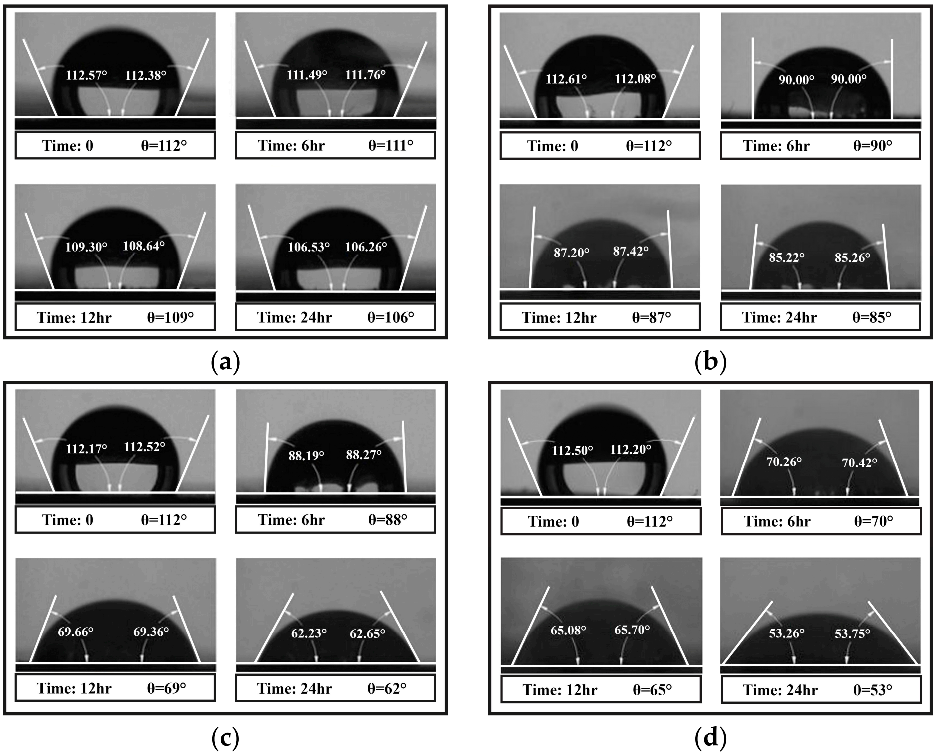

Figure 6. Contact angle variation for different solutions: (**a**) pure water; (**b**) KCl-2 solution; (**c**) KCl-1 solution; (**d**) KCl-1+NF solution.

3.2. Core Flooding

To observe the effects of brines during core flooding, we used pure water, KCl-1, KCl-2, and KCL-1+NF with SAS concentration set at 1/0 wt%. The KCl-1+NF injection pattern is depicted by the red curve in Figure 7. The injection pattern of KCl-2 is represented by the yellow curve. Finally, the pure water injection pattern is represented by the green curve. As demonstrated in Figure 7, in the case of KCl-1+NF injection, the residual oil recovery rate increases up to 0.5 PV directly after the injection. This is the key indicator of the favorable conditions generated by LSW's wettability-altering features. Starting from 0.5 PV to 2 PV, the recovery rate is not visible, passing the barrier when a minimum of 2 PV was produced. Our observation showed during this interval, the maximum recovery rate was achieved by the KCl-1+NF injection pattern. This is because of the significant wettability changes. Oil recovery can be increased in addition to the wettability adjustment by lowering the IFT between the displacing fluid and oil. This was primarily the function of the mixed composition's injected NF, which also allows for improved mobility control and sweep efficiency.

3.3. Fluid-Flow Characteristics Based on NMR

Matching MR images are displayed in Figure 8, showing the situation after the injection rate is changed from 1 mL/min to 5 mL/min. The displacement areas are visible in the high-permeability and low-permeability layers at 5 PV. As the injection continues, the high-permeability layer's displacement front moves clearly, and the low-permeability layer's displacement front's position barely changes between 10 and 15 PV. Conversely, we discovered that in both the high and low-permeability layers, the displacement front moves at the same pace.

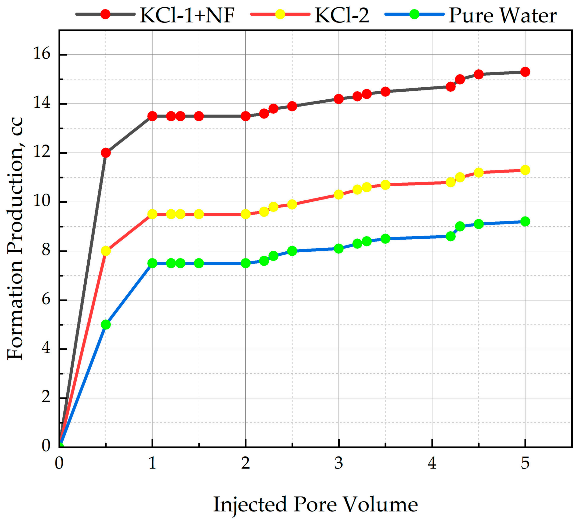

Figure 7. The total amount of water generated over time as a result of oil injection.

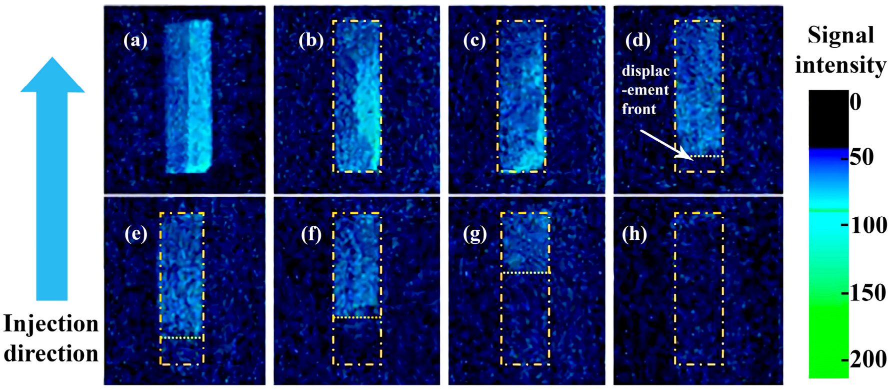

Figure 8. Images taken by MR: (**a**) 0.0 PV; (**b**) 0.6 PV; (**c**) 1.1 PV; (**d**) 4.0 PV; (**e**) 5.5 PV; (**f**) 7.5 PV; (**g**) 10.5 PV (**h**) 15.0 PV.

When 2 PV of KCl-1+NF is injected at a rate of 5 mL/min, Figure 9 shows how the water saturation in the middle pores quickly declines to 73%, and then gradually flattens in the middle and late stages. The water saturation in the medium pores decreases to 35.9% at 15 PV, which is a significantly higher percentage than what KCl-2 produced. Raising the injection rate reduces the impact of fluid displacement in the medium and small holes, as shown by the observation that the water saturation in small pores only decreases to 63.6%. This may be the result of a high fluid input rate decreasing jamming capacity. The fast injection rate quickly causes a shear-thinning effect, which drastically lowers the fluid's apparent viscosity. Increasing the injected solution sweep efficiency in the small and medium holes becomes more difficult when the blocking resistance in the large pores decreases.

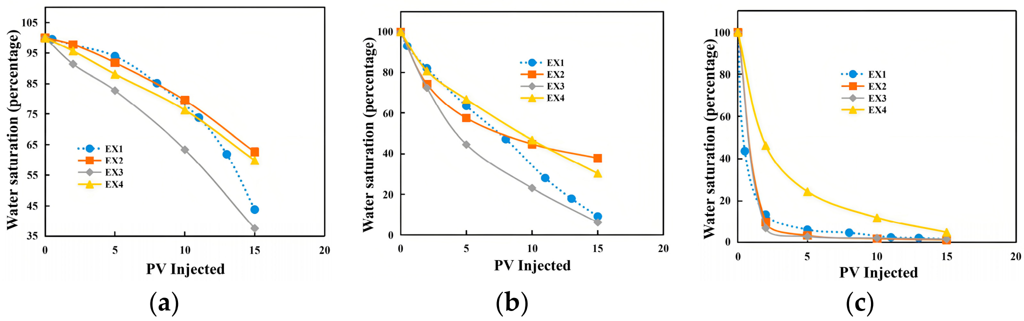

Figure 9. Water saturation during injection in various kinds of pores: (**a**) small (φ24.7%); (**b**) medium (φ25.5%); (**c**) large (φ27.3%).

4. Discussion

This study took into account the benefits of combining LSW with NF as well as the problems associated with NF flooding and LSW, such as sweep efficiency and the influence of wettability alteration. Then, to increase the effectiveness of low-salinity flooding, a novel hybrid EOR technique of combining LSW with NF to alter the wettability of the rock surface was presented. This technique is known as LSWNF (low-salinity water + nanofluid) flooding. Throughout this research, various experimental methods were employed to analyze the effects of different concentrations of low-salinity water and nanofluids on wettability alteration and enhanced oil recovery. The results demonstrated that the composition with KCl-1+NF outperformed other compositions in terms of wettability alteration and enhanced oil recovery. This finding indicates that a specific combination of LSW and NF can have a significant impact on improving oil recovery efficiency. The most significant findings and accomplishments from this research include the following.

First, nanofluids can alter the wettability of the reservoir rock further, enhancing the displacement of oil by water. The nanoparticles in the nanofluids interact with the rock surface, modifying the surface properties and improving the spreading and penetration of the injected fluid. This leads to improved oil recovery by reducing the residual oil saturation and increasing the sweep efficiency.

Second, the presence of nanoparticles in the nanofluids can also exhibit unique fluid behavior, such as reduced viscosity and increased thermal conductivity. These properties can enhance the fluid flow within the reservoir, allowing for better fluid mobility and improved displacement of oil. The reduced viscosity of the nanofluids can mitigate the challenges of a high mobility ratio between injected fluids and the reservoir oil, resulting in more efficient oil displacement.

Furthermore, the hybridization of nanofluids and low-salinity water can potentially address the issue of water channeling. The nanoparticles in the nanofluids can act as solid diverting agents, plugging the high-permeability zones and redirecting the injected fluids into the low-permeability areas. This helps in achieving a more uniform sweep of the reservoir, preventing the bypassing of oil and improving overall oil recovery efficiency.

Both NF and LSW have a positive impact on the wettability alteration of the rock surface. However, oil binds to the rock surface as KCl and SiO_2 concentrations rise. As a result, choosing compositions with lower LSW and NF concentrations is a smart practice.

The KCl-1+NF injection pattern from the core flooding trials produced the maximum residual oil recovery, which was followed by the KCl-2 and pure water injection patterns. The presence of NF has been shown to increase the amount of oil that can be collected, since it increases the mobility and sweep efficiency of the residual oil. Additionally, it was discovered that the KCl injection pattern produced the maximum recovery for the same interval. Its high wettability variations are the cause of this.

The magnetic resonance images and the T2 spectrum test results obtained from the NMR investigations may demonstrate the migration properties of LSW + NF in heterogeneous cores, and the displacement of the solution in different porosities. The results showed that due to the initial injection stage's low flow resistance, the saturation in big pores increased quickly. At 15.0 PV, the saturation increased slightly to 95.8%. At 15 PV, the medium pores' occupation climbed gradually to 91.5%.

5. Conclusions

In conclusion, a viable strategy for improved oil recovery from mature oil reservoirs is the hybridization of nanofluids with low-salinity water floods. The synergistic effects of combining these two approaches improve fluid movement, sweep efficiency, and wettability change. This study's findings add to our knowledge of the possible advantages of this hybrid strategy, and offer insightful information for future research as well as possible applications in the realm of oil reservoir production. The increasing need for energy worldwide makes it imperative to investigate new and economical ways to maximize oil recovery from current reserves. Combining low-salinity water floods with nanofluid hybridization is an achievable approach to ensure sustainable and effective use of oil resources, while also helping to fulfill the world's growing energy demand.

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Data Availability Statement: The data presented in this study are available on request from the corresponding author. The data are not publicly available due to links' access being geoblocked outside of China.

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

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