Hydrophobic Silica with Potential for Water-Injection Augmentation of a Low-Permeability Reservoir: Drag Reduction and Self-Cleaning Ability in Relation to Interfacial Interactions

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ABSTRACT: An aqueous nanofluid containing superhydrophobic silica nanoparticles with a high surface activity and an average size of 7 nm was used to enhance the water injection of a low-permeability well. The mechanism for the aqueous nanofluid to enhance water injection was discussed. Findings indicate that the silica aqueous nanofluid can greatly increase the effective water permeability even after injecting water for 2100 pore volumes. This is because the hydrophobic silica nanoparticles can be well adsorbed onto the surface of the porous channels to cause hydrophilic to hydrophobic transformation. Both the hydrophobic capillary force and adhesion work contribute to increasing water injection; and in particular, there is a critical point in the pressure-permeability curves for the rock cores with different wettabilities. Only above the critical point, the hydrophobic rock core exhibits a higher effective water permeability than that of the hydrophilic one, which is imperative for drag reduction. Moreover, the hydrophobic rock core surface has a remarkable self-cleaning ability and can reduce the expansion ratio of clay and inhibit the formation of scale in association with the increase of effective porosity via decreasing the hydration film amount. This approach, highlighting the important role of wettability alteration in increasing water injection, could potentially promote the application of a silica aqueous nanofluid in enhanced oil recovery.

1. INTRODUCTION

Low permeability reservoirs are widely distributed among the world, and Changqing Oilfield (Xi’an, China) and Yanchang Oilfield (Yan’an, China) are the representative ones in China. Usually, the low permeability reservoirs are characterized by low waterflood injectivity, poor sweep, and low productivity, because of their internal microporous or even nanoporous structure.1 The average oil recovery from low permeability reservoirs after the primary production period (produced by natural reservoir energy) is only about 13%, which means that an enormous amount of petroleum is left underground thereafter. In recent years, most of the low permeability oil fields in the world have entered the stage of water flooding exploitation, with which crude oil cannot be displaced from the formation unless enough water is injected into the macro and nanopores.2 However, the macro and nanopores are often easily blocked owing to the gradual formation of scale and the hydration layer on the surface of the porous rock core walls, which makes it infeasible for water to be injected into the reservoir after a long period of operation.

Usually, waterflooding injectivity is greatly affected by the injection pressure, the wettability of the pore surface, and the size of the pore.3 It often requires the injection of a large volume of water into the reservoirs, which could cause a very high injection pressure of the water injection well. This is why it still remains a challenge to develop more cost-effective high-pressure equipment and technology for the extraction of low permeability reservoirs.4 As a traditional pathway for enhancing water injection of low permeability reservoirs, acidizing can help to open the pores, but it has a short validity and can cause irreversible damage to the stratum.

Wettability may mediate water resistance to increase injection without acting alone to add pressure, and it could control the flood and spatial distribution of fluids in the porous medium, thereby greatly affecting water and oil permeability.5,6 When the rock is hydrophilic, there is a tendency for water to occupy the small pores and the majority of the rock surface. Pure water does not penetrate spontaneously into a hydrophobic porous capillary unless being given an external force.7 In recent years, surfactants and nanoparticles have been adopted to alter the wettability of the pore surface so as to increase water injection and enhance oil recovery.8 To date, it has been found that surfactants with ultralow interfacial tension (IFT) (0.001 mN/m) can reduce the capillary pressure, thereby potentially increasing the amount of water injected into reservoirs.9 For example, Ju et al.10 used one kind...
of polysilicon with a size of 10–500 nm to change the wettability of porous media and enhance water injection of an oil field. Zhang et al.11 and Yan et al.12 separately investigated the water injection behavior of hydrophobic silica nanoparticle; and they suggested that the enhanced water injection could be related to the drag reduction of the superhydrophobic surface via a slip effect. Mousavi et al.13 Sephrinia and Mohammadi,14 and Feng et al.15 prepared hydrophobic/oleophobic fluorinated silica nanoparticles for removing liquid banking near the wellbore region. They found that the nanosilica particulates can be diffused into the layers of low-permeability rocks to form low drag surfaces with macro-nanochannels, therein effectively removing the liquid film of the pore surface and reducing the flood resistance.

We previously reported that a series of superhydrophobic silica nanoparticles can alter the hydrophilic well bore to a hydrophobic one and enhance water injection. Nevertheless, currently available superhydrophobic silica nanoparticles are actually cost ineffective and environmentally unacceptable in oil recovery engineering, because they rely on organic solvents such as diesel and benzene for dispersion and transfer and exist in a state of serious aggregation with a size from 40 to 1000 nm (the aggregates in organic solvents can form a thick adsorption layer to block the microscopic or nanoscale pore throats of the reservoir rock). Recently, we found that the aqueous silica dispersion with a size of about 7 nm as well as good dispersibility in water may have promising potential for increasing water injection of low permeability reservoirs.16 The mechanism for the silica aqueous nano-fluid to increase water injection, however, still remains unknown at this stage, especially with a view to the different physical and chemical properties of the hydrophobic surface and the hydrophilic one.

In the present research, therefore, we focus on the effect of the silica nanofluid on the surface wettability, water permeability, and water injection of low permeability reservoirs. Based on the validity evaluation by the clay expansion test and scale formation examination as well as the core flooding test and porosity measurement of the porous channels, this article deals with the long-term enhanced water-injection mechanism of the silica nanofluid in relation to the interfacial reactions between the injected water and the porous channel surface, including capillary pressure, adhesion work, and injection pressure.

2. RESULTS AND DISCUSSION

2.1. Interface Behavior of the Silica Nanofluid. The silica aqueous nanofluid containing hydrophobic silica nanoparticle and sodium dodecyl benzene sulfonate (SDBS) is used as an agent to reduce the injection pressure so as to enhance the water injection efficiency in low permeability reservoirs. The silica aqueous nanofluid with a size distribution from 4 to 12 nm has extremely stability, and there is no phase separation even after being placed in the natural environment for more than 3 months.16 It can be reckoned as a microemulsion dispersion system, because the superhydrophobic nanoparticle can be thought of as the dispersed oil droplets. The IFT between the silica aqueous nanofluid and kerosene is about 0.2 mN/m (see Figure 1a); and such a low IFT could be meaningful for promoting the injection of the silica aqueous nanofluid into the reservoirs. Because the SDBS-stabilized emulsions can be easily destroyed by salt water, the superhydrophobic silica nanoparticle can be induced to be separated from the aqueous dispersion by NH₄Cl. The as-released superhydrophobic nanosilica can transfer from the water phase into the oil phase, as illustrated in Figure 1b. This means that the as-prepared superhydrophobic silica nanofluid is simultaneously hydrophobic and lipophilic.

The rock in the water-injection well is commonly water-wet due to the long-term water injection. Water contact angle (WCA) measurements were conducted to evaluate the wettability of the rock core before and after the introduction of the silica aqueous nanofluid. As shown in Figure 2a, the water droplet can rapidly permeate into the untreated rock core, and the WCA is only about 32°. However, the WCA rises to 134° after the core is treated with the silica aqueous nanofluid for 36 h (Figure 2b). In the absence of the silica aqueous nanofluid, a large amount of clay sheets with clear edges and corners is present on the rock core surface (Figure 2c). After treatment with acid liquor, a large amount of hydroxyl is exposed on the rock core surface; and the exposed hydroxyl can react with the hydrophobic silica nanoparticle released from the silica aqueous nanofluid to form a continuous and dense adsorption layer with a low surface energy as well as macro and nanostructure on the porous channel surface (see Figure 2d,e). The silica layer adsorbed on the rock core surface is very thin and would not obviously occupy the micro-nanochannels; and it could alter the hydrophilic rock core surface to a hydrophobic one, thereby enhancing water injection.
2.2. Water Injection Behavior. Injection pressure and effective water permeability are two important parameters to describe the water injection capacity of a rock core. The fluid permeability ($K$) is estimated based on the Darcy–Weisbach formula: $K = (Q / L / \mu) / (A \times \Delta P)$, where $Q$ is the permeation flow rate, $L$ and $A$ are the length and sectional area of the core, $\mu$ is the viscosity of the injected liquid, and $\Delta P$ is the injection pressure. Figure 3a shows the effect of the silica aqueous nanofluid on the injection capacity (the injection pressure and effective water permeability) of no. 1 rock core under different injection rates before and after treatment with the silica aqueous nanofluid (a) as well as injection validity of no. 2 rock core after the injection of SDBS and silica aqueous nanofluid (b).

The injection pressure and water permeability of no. 1 rock core are about 1.5 MPa and 1.8 mD, respectively. After SDBS injection, the injection pressure decreases to 1.1 MPa and the effective permeability declines to 2.4 mD. After adsorbing for 36 h, the injection pressure is further reduced to 0.69 MPa and the effective permeability of the rock core is increased to 4.0 mD, which indicates that the silica aqueous nanofluid contributes to significantly reducing the resistance of the core upon water injection. After SDBS is injected into no. 2 rock core with a water permeability of 4 mD, the water permeability rises to 5 mD because of the enhanced dispersion of the silica aqueous nanofluid in water therewith. However, the water permeability declines from 5 to 4 mD after the water injection of 1400 PV (Figure 3b). This implies that SDBS is adsorbed on the surface of the rock core channel via a weak force. Besides, the introduction of the silica aqueous nanofluid increases the water permeability of no. 2 rock core from 4 to 8.3 mD (Figure 3b), which demonstrates that the silica aqueous nanofluid is more efficient in enhancing water injection than the SDBS, possibly because of the adsorption of superhydrophobic silica nanoparticles on the pore surface. Moreover, the silica aqueous nanofluid is able to retain a stable water permeability of about 8.3 mD during a significantly prolonged injection of more than 2200 PV. This could be because the highly active silica nanoparticles can be combined promptly with the pore surface.

2.3. Drag Reduction Mechanism for Hydrophobic Pores. When the rock is water-wet, there is a tendency for water to occupy the small pores and to contact the majority of the rock surface. Pure water does not penetrate spontaneously into a hydrophobic porous capillary, because if the contact angle is greater than $\pi/2$, it can only be forced to penetrate through the stranded layer. The hydrophobic silica nanoparticles on the pore surface can prevent water from flowing into the pores, thereby effectively reducing the resistance of the water flow. However, the water permeability of the pore surface is greatly increased in association with the change of wettability upon the adsorption of hydrophobic silica nanoparticles thereon. Figure 4a shows the effect of the silica aqueous nanofluid on the injection pressure and water permeability of no. 3 rock core with different wettability (the silica aqueous nanofluid is injected for different cycles). The WCAs of no. 3 rock core treated once and twice are about 134° and 142°, respectively, which indicates that an increasing injection cycle leads to an increase in the hydrophobicity of the rock core. In the meantime, the same rock core with different wettability exhibits a much different phase permeability under different injection pressures. Namely, the hydrophilic rock core exhibits a larger effective water permeability than that of the hydrophobic one under a low injection pressure; and the hydrophobic rock core exhibits a larger effective water permeability than that of the hydrophilic one under a high injection pressure. More importantly, three critical points are observed in the three phase permeability curves under an injection pressure of 0.223, 0.181, and 0.16 MPa, respectively, which could help to provide some clues to explaining the mechanism responsible for water-injection augmentation of the silica aqueous nanofluid and to controlling the wettability of the reservoir for enhanced oil recovery.

As shown in Figure 4b, three kinds of forces have an impact on the injected water: the pressure of the to be injected water ($P_{iw}$), the adhesion force between water and the porous channel surface ($W_{ad}$), and the capillary force ($P_c$). $P_{iw}$ is a power for water flooding. The capillary force, according to the Laplace theory, is described as $P_c = -2(\sigma \cos \theta)/r_c$ (where $\gamma$ is the surface tension, $\theta$ is the contact angle of the liquid on the rock core surface, and $r_c$ is the radius of curvature). Therefore, the injected water would tend to occupy the small pores and the majority of the rock surface when the rock is hydrophilic, because in this case $P_c$ is larger than zero. Moreover, based on the Young Dupré’s equation, $W_{ad}$ can be calculated as $W_{ad} = \gamma_{lv}(1 + \cos \theta_h)$, where $\gamma_{lv}$ and $\theta_h$ refer to the surface tension of the liquid/air interface and relevant contact angle of the liquid in air, respectively. Because the hydrophilic rock core exhibits a larger $W_{ad}$ than the hydrophobic one, $P_c$ and $P_{iw}$ should act as the dominant powers for water flooding of the hydrophobic rock core under a low injection pressure (in this case, $W_{ad}$ would act as the secondary power for water flooding). Under a high $P_{net}$, however, the stranded layer would be thickened, because of the increase in the flowing rate of the injected water; and the effective seepage porosity would tend to decrease while the water permeability remains nearly unchanged owing to adequate permeation of the injected water in most pores.
Under a low injection pressure, the hydrophobic rock core surface exhibits a considerably lower water permeability than the hydrophilic one, because in this case $P_c$ and $W_{ad}$ would play key roles to prevent water flooding. However, under a high injection pressure, the $W_{ad}$ between water and the hydrophobic channel surface is much smaller than that of the hydrophilic surface, which, in association with the decrease of the water retention layer, plays a key role in changing the water permeability. As a result, the water permeability of the pore surface under a large injection pressure is greatly increased in association with the change of wettability upon the adsorption of hydrophobic silica nanoparticles. For achieving desired water-injection augmentation, the equilibrium point, that is, the critical point, among $P_{crit}$, $P_c$, and $W_{ad}$ would be of special significance.

2.4. Self-Cleaning Performance. The reduction of $W_{ad}$ is a key factor for increasing water injection in association with decreasing injection pressure, but not the only one. Therefore, the self-cleaning performance of the silica aqueous nanofluid is investigated in this study. The pore size directly affects water flow efficiency; and the wettability of the untreated porous channel surfaces are hydrophilic and liable to wetting by water. In other words, a small effective porosity refers to a thick hydrated layer; and the effective porosity corresponds to the effective channels for water flooding. To further study the water injection behavior, we measured the effective porosity and the amount of the hydration layer (i.e., the amount of the adsorbed silica nanoparticle) of the sand-filled tube. As shown in Figure 5a, when the sand-filled tube undergoes hydrophilic to hydrophobic transformation upon the introduction of the silica aqueous nanofluid, the thickness of the hydration layer tends to decrease obviously; and its effective porosity tends to significantly increase from 18.3 to 23.1%. As a result, the pore radius is effectively expanded to the decrease water injection pressure and increase effective permeability.

The swelling of clay is a key factor responsible for permeability decrease of the low permeability reservoir. Figure 5b shows the swelling rate of the clay in the water and silica aqueous nanofluid. After 12 days of immersion in SDS solution, the clay has a swelling volume of 20 mL. However, the swelling volume is only about 15 mL in the silica aqueous nanofluid. This means that the silica aqueous nanofluid contributes to significantly improving the swelling resistance of the clay. At the same time, the clay particles are more likely to accumulate in silica aqueous nanofluids, thereby greatly inhibiting particle migration and reducing the possibility for the particles to block microporous channels.

The commonly used injection water in oil fields is brine, and it tends to form a lot of scale on the rock core channel surfaces. Simulated rock core displacement tests were conducted to investigate the effect of the silica aqueous nanofluid on the formation of scale on the porous channel surface. The energy dispersive spectrometry (EDS) mappings of the hydrophilic and hydrophobic core sections after immersion in the brine solution (2 wt % CaCl₂ + 1 wt % MgCl₂) for 48 h are shown in Figure 6a. After immersion in the brine solution, the untreated hydrophilic rock core contains a large amount of Ca and Mg, corresponding to the formation of a large amount of scale on the pore surfaces thereafter. The as-formed scale can greatly decrease the pore size, leading to a very small effective porosity of water permeating. However, the rock core treated with the silica aqueous nanofluid has a strong hydrophobicity, and the hydrophobic surface can keep water away from the pore surface. As a result, a very small amount of Ca and Mg is detected by EDS on the treated rock core surface, which is favorable for maintaining a high effective porosity for water permeability and a stable effective water permeability for a long period.

Another core flooding experiment conducted to evaluate the effect of surface wettability on scale formation is shown in Figure 6b. It is seen that no. 4 rock core untreated with the silica aqueous nanofluid exhibits an injection pressure of about 0.17 MPa and an effective permeability of about 4.8 mD. When a certain volume of brine (2 wt % CaCl₂ + 1 wt % MgCl₂) is injected into the rock core at a low velocity, the injection pressure increases to 0.21 MPa and the effective permeability declines to 3.7 mD. This is due to the formation of scale on the hydrophilic channel surface. When the same rock core is treated with HCl to remove the as-formed scale and then injected with the same volume of the silica aqueous nanofluid to alter the wettability, the resultant hydrophobic rock core has quite a low injection pressure of about 0.09 MPa and a high effective permeability of about 7.9 mD. This hydrophobic rock core surface is nearly free of scale formation after the injection of the same brine solution, and its injection pressure and...
effective water permeability vary slightly therewith. This demonstrates that the introduction of the silica aqueous nanofluid results in hydrophilic to hydrophobic transformation of the rock core surface and helps to prevent the formation of scale in its porous channel, thereby adding to the structural stability of the porous channel and to water-injection augmentation in the long run as well.

3. CONCLUSIONS

Silica aqueous nanofluid with an average particle size of about 7 nm is used to alter the hydrophilic rock core surface to a hydrophobic one via forming a dense nanosilica layer thereon. Thanks to the small size and high activity of the silica nanoparticle released from the silica aqueous nanofluid, the resultant hydrophobic porous channels of the rock core exhibit greatly increased water permeability and significantly reduced the injection pressure as well as drastically prolonged injection validity for the water-injection well of the low-permeability reservoir. This is highly dependent on the hydrophilic to hydrophobic transformation of the porous rock cores in association with the increase in the water injection via the hydrophobic capillary mechanism and the decrease in the surface energy of the rock cores upon the introduction of the silica aqueous nanofluid. Moreover, a critical point is observed in the pressure-permeability curves for the rock cores with different wettability; and the hydrophobic rock core exhibits a higher effective water permeability than the hydrophilic one only above the critical point, which could be significant for better understanding the mechanisms of drag reduction at the hydrophobic core channels. In addition, the self-cleaning performance of the hydrophobic surface treated with the silica nanofluid could provide a clue to regulating the oil–water phase flow for enhanced oil recovery. In summary, the present approach, highlighting the interface behaviors between water and pore channels, could be promising for water-injection augmentation of low permeability reservoirs and enhancing oil recovery as well.

4. EXPERIMENTAL SECTION

4.1. Materials. The silica aqueous nanofluid with good dispersibility in water was fabricated at our laboratory; and its structure characterizations are available in our previous work. Industrial reagents calcium chloride (CaCl2) and magnesium chloride hexahydrate (MgCl2·6H2O) as well as analytical reagents SDSBS were purchased from Sinopharm Chemical Reagent Company Limited (Shanghai, China). Distilled water prepared in our laboratory was used as the solvent and/or for rinsing.

4.2. Rock Core Flooding Test and Effective Porosity Measurement. A series of rock core flooding tests were conducted with four artificial rock cores. The parameters of the rock cores are listed in Table 1. The water phase permeability before and after acidification was tested at a constant water injection rate; and the clay swelling behavior was also measured. Furthermore, a sand-filled tube was used to measure the effective porosity and the amount of hydrated layer adsorbed on the sand. The sand-filled tube has a fixed volume denoted as V. The amount of the dried sand filled into the tube is denoted as Wd; and the amount of water filled into the pores of the sand-filled tube is denoted as Ww. After being filtrated until there is no water dripping, the amount of the wet sand with a hydrated layer is obtained as Wh. The amount of the hydrated layer (Wd referring to the amount of the adsorbed silica nanoparticle) is calculated as W = Wh − Ww. The effective porosity (P) of the sand-filled tube is calculated as P = W/Ww × 100%.

4.3. Characterizations. The IFT between kerosene and the silica aqueous nanofluid was measured with a Texas-500C spinning drop tension meter at a rotation speed of 5000 rpm and a temperature of 40 °C; and the IFT was calculated based on the Young–Laplace formula. The morphology and element composition (determined by EDS) of the rock cores were analyzed with a FEI Nova Nano SEM 450 field emission scanning electron microscope (FESEM) at an accelerating voltage of 5 kV. Prior to FESEM imaging, the to be tested sample was coated with a thin layer of Au (1 min sputtering; Hitachi Corporation, Japan). The water contact angles were recorded with a DM300 liquid/solid interface analyzer (Kyowa Interface Science Co., Ltd., Japan), and at least three repeat measurements were conducted for each to-be-tested sample.

Table 1. Rock Core Parameters

| No. | rock cores | length (mm) | diameter (mm) | pore volume (cm^3) | porosity (%) |
|-----|------------|-------------|---------------|-------------------|-------------|
| no. 1 | 44.3 | 25.4 | 3.65 | 16.3 |
| no. 2 | 88.2 | 25.1 | 5.83 | 13.4 |
| no. 3 | 98.0 | 25.0 | 7.19 | 16.2 |
| no. 4 | 45.9 | 25.2 | 3.42 | 15.0 |

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Notes
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