Conformance Control in Oil Reservoirs by Citric Acid-Coated Magnetite Nanoparticles

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ABSTRACT: Reservoir conformance control methods may significantly improve enhanced oil recovery technologies through reduced water production and profile correction. Excessive water production in oil and gas reservoirs leads to severe problems. Water shutoff and conformance control are, therefore, financially and environmentally advantageous for the petroleum industry. In this paper, water shutoff performance of citric acid-coated magnetite (CACM) and hematite nanoparticles (NPs) as well as polyacrylamide polymer solution in a heterogeneous and homogeneous two-dimensional micromodel is compared. A facile one-step technique is used to synthesize the CACM NPs. The NPs, which are reusable, easily prepared, and environmentally friendly, are characterized using Fourier-transform infrared spectroscopy, field emission scanning electron microscopy, dynamic light scattering, and X-ray diffraction. The results confirm uniform spherical Fe₃O₄ NPs of an average diameter of 40 nm, well coated with citric acid. CACM NPs provide a high pressure drop coupled with an acceptable resistance factor and residual resistance factor owing to NP arrangement into a solid-/gel-like structure in the presence of a magnetic field. A resistance factor and a residual resistance factor of 3.5 and 2.14, respectively, were achieved for heavy oil and the heterogeneous micromodel. This structure contributed to an appreciable plugging efficiency. CACM NPs respond to magnetic field intensities between 4500 and 6000 G. CACM NPs act as a gel, forming a solid-/gel-like structure, which moves toward the magnetic field and thereby shuts off the produced water and increases the oil fraction. The findings of this study suggest the ability to shut off water production using specially designed magnetic field-responsive smart fluids. The application would require innovative design of field equipment.

1. INTRODUCTION

The global cumulative oil production from 1970 to 2014 was 115.6 billion barrels, which captures the importance of oil as a global energy source. Excessive production of water is a serious problem widely encountered during oil production. Production of water and water coning can restrict the economic life of oil and gas wells and cause severe corrosion, fine migration, and hydrostatic loading and leads to decreased sweep efficiency and oil recovery. In 2012, produced water was estimated to cost the oilfield industry approximately $50 billion. Produced water contains different organic and inorganic components, which can contaminate surface and underground water and soil. Therefore, water shutoff and conformance control represents an attractive financial and environmental alternative for the petroleum industry. Water production can be reduced by recombining the well or by placing mechanical devices to isolate formation water zones. These remedies are, however, expensive. Water shutting-off techniques are divided into three main categories: namely, mechanical, chemical, and microbiological. However, heterogeneity of reservoirs seriously influences the flow of gas, oil, and water and leads to small oil recovery and early excessive water production. Several investigations explored chemical methods to homogenize reservoirs and control excessive water production.

Polymers have been used as conformance control additives since 1960s. Adding a polymer can resolve fingering problems since 1960s. Adding a polymer can resolve fingering problems and improve the sweep efficiency of water flooding by increasing water viscosity and subsequently reducing water mobility. Polymer flooding coupled with conformance control was achieved using hydrolyzed polyacrylamides (PAMs), and biopolymers such as polysaccharides (e.g., xanthan gum), and hydrophobically associated polymers. These are water-soluble polymers with short hydrophobic branches attached to

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their backbone. Conformance control consists of injecting an aqueous solution of an activator and a polymer into high-permeability pores to seal off pore throats via polymer cross-linking and gelling. Microgels and polymer gels have been utilized for conformance control in reservoirs with appreciable disparity in permeability. On the other hand, cross-linking agents added to polymers achieve more plugging and better permeability reduction. Nevertheless, polymer injection is challenged by undesirable gel-phase separation, the requirement of effective mixing of the polymer and cross-linker, and gelation kinetics control in heterogeneous reservoirs. Although a wide variety of conformance chemicals have been proposed, most are not demonstrated in environmentally sensitive zones such as the North Sea. There is a growing need for environmentally benign conformance products that are biodegradable, nontoxic, and economical. Moreover, an enormous capacity of conformance chemicals have been proposed, most are for shutting-off water or gas-producing zones (e.g., thief zones) to reduce water production. Kalgaonkar et al. reported that a low volume (1−5 barrels) of nanofluids is sufficient to shut off a zone in a reservoir.

Many recent investigations have reported advantageous applications of nanotechnology in the petroleum industry, including nanoparticle (NP) applications for enhanced oil recovery (EOR). Relatively small amounts of NPs significantly enhanced specific properties of the base well fluid. The characteristic dimension of the pores in oil reservoirs is typically in the order of 1000 nm, whereas the size of stable NPs is lower than 100 nm. Hence, the transport of NPs in porous media is unlikely to be hindered by size exclusion. Consequently, NPs may smoothly run across porous media without altering the reservoir permeability. Furthermore, NPs have shown ability to alter wettability, reduce interfacial tension (IFT), and increase viscosity. In particular, magnetite Fe₃O₄ NPs have been used as nanosensors, with the ability to record certain reservoir characteristics, as well as EOR agents. Nevertheless, few studies reported the application of NPs for shutting-off purposes. When a magnetic field was applied, magnetorehydrological smart fluids demonstrated reversible and swift liquid-to-solid-like transfiguration. Accordingly, magnetic NP-based well fluids are ideal for water shutoff and conformance control, especially since external magnetic field applications to oil reservoirs have been demonstrated. A solid-like structure can be targeted through effective mixing of the polymer and cross-linker, and gelation of the micromodels is measured, and the resistance factor (RF) are counted up for different systems in the presence and absence of a magnetic field. The general sketch of the problem is shown in Figure 1.

### 2. EXPERIMENTAL SECTION

#### 2.1. Materials

Two types of Iranian crude oils were employed in order to widen the scope of the current study. Properties of the oils are listed in Table 1. The PAM polymer was purchased from SNF Company (an average M₀ of 5 × 10⁶ Da), and commercial Fe₂O₃ NPs with an average size distribution of 50 nm (US Research Nanomaterials, Inc., USA) were used for comparison. Deionized water was used in order to prepare the polymeric solution. Brine solutions for pre- and post-flush water flooding via the micromodels were prepared using NaCl and CaCl₂ (Merck Co., Ltd., Germany). In order to disperse the commercial Fe₂O₃ NPs in oil, sodium dodecyl sulfate (Kian Kaveh Azma Co., Ltd., Iran) was used.

#### 2.2. Synthesis of CACM NPs

Figure 2 is a schematic illustrating the in-house synthesis of the Fe₃O₄ NPs, which was adopted from Hui et al. with minor changes. In brief, 4 mmol of NaOH (Merck Co., Ltd., Germany), 1 mmol of citric acid trisodium salt dehydrate (C₆H₅Na₃O₇·H₂O Merck Co., Ltd., Germany), and 20 mmol of NaNO₃ (Merck Co., Ltd., Germany) were dissolved in 19 mL of deionized water. Then, 1 mL of a 2 M FeSO₄·7H₂O (Merck Co., Ltd., Germany) solution was surcharged rapidly to the admixture which was heated to ∼100 °C under mixing in a capped vessel for 120 min. The black precipitate of citric acid-capped Fe₃O₄ was collected at the end of heating, which was washed with deionized water and left to dry under ambient conditions for 60 min. More details can be found in our previous study.
2.3. Characterization. The morphology and particle size distribution of the as-prepared Fe₃O₄ NPs were determined using transmission electron microscopy (TEM) (LEO912-AB, LEO, Germany), field emission scanning electron microscopy (FESEM) (TESCAN, MIRA 3, Czech Republic), and dynamic light scattering (DLS) (Nano ZS, Malvern instruments, Worcester, UK). The nature of interaction between citric acid and Fe₃O₄ NPs was determined using Fourier-transform infrared (FTIR) spectroscopy (Bruker, Tensor 27, Germany). X-ray diffraction (XRD) (PW-1730, Philips) was used to confirm the identity of the as-prepared NPs.

2.4. Micromodel Experimental Setup. In this study, glass micromodels (heterogeneous and homogeneous) were prepared for imaging heterogeneous and homogeneous reservoirs. The laser technology was used to design and manufacture the patterns of the 2D micromodel. Figure 3 depicts patterns of the two micromodels. Physical and hydraulic specifications of the micromodels are listed in Table 2. The same micromodels were used in our previous work.

Figure 2. Schematic of the synthesis procedure of Fe₃O₄ NPs.

Figure 3. Patterns of the heterogeneous and homogeneous micromodels employed in this study.

A uniform light source was used under the micromodels to create better resolution for the captured photographs. A high-precision differential pressure gauge and a high-accuracy syringe pump with low-rate injection (Fusion one, US) were used to control the injection rate through the micromodels. The experimental setup is shown in Figure 4. A box of magnetic field containing two strong fixed magnets was used to apply a magnetic field with the ability to alter the strength of the magnetic field. More details are included in our previous study. The orientation of the magnetic field is a key variable during the application of magnetic NPs for water shutoff treatment and EOR purposes. In fact, during EOR experiments, two magnets were located along the two sides of the micromodels, helping the magnetite nanofluid to move toward the sides of the micromodels to enhance volumetric sweep efficiency. During water shutoff experiments, on the other hand, the two magnets were located above and below the micromodel, helping the NPs to create a solid-like structure and shutoff pores with a high water cut. Given the fact that the models are 2D, it is anticipated that structures of the magnetic NPs would arise in the vertical direction, especially in larger pores hindering the flow in these pores and forcing the injected fluid into smaller pores, hence reducing the water cut.

2.5. Experimental Procedure. Washing and cleaning the micromodels with acetone, toluene, and hot water were the primary steps before starting the injection. Magnetic Fe₃O₄ NPs were dispersed in selected oils at 2 wt %. The choice of 2 wt % followed literature findings and led to organosols that can be stable for several days. Comparison with the nanofluid was tested using polymer flooding. The commercial superpusher PAM polymer in aqueous solution was used at 2000 ppm for all the experiments. It is noted that the polymer concentration ranging from 1000 to 5000 ppm has been utilized for polymer flooding. In order to simulate connate water saturation, brine (42,000 ppm NaCl + 18,000 ppm CaCl₂) was utilized to

Table 2. Physical and Hydraulic Properties of the Micromodels

| type of micromodel | absolute permeability (m²) | porosity (%) | average pore diameter (m) | average throat diameter (m) | dimensions (m × m) |
|-------------------|---------------------------|--------------|---------------------------|---------------------------|-------------------|
| heterogeneous²   | 19.62 × 10⁻¹¹             | 28.96        | 412 × 10⁻⁶                | 89 × 10⁻⁹                 | 0.14 × 0.04       |
| homogeneous      | 28.48 × 10⁻¹³             | 27.62        | 376 × 10⁻⁶                | 72 × 10⁻⁹                 | 0.14 × 0.04       |

²The heterogeneous micromodel has three sections with different permeabilities as follows: section 1 (bottom layer): the permeability of this layer is 1.472 × 10⁻¹² m²; section 2 (middle layer): the permeability of this layer is 1.977 × 10⁻¹² m²; section 3 (top layer): the permeability of this layer is 2.386 × 10⁻¹² m².
initially saturate micromodels and then selected oils, heavy and light, were used for further saturation. One pore volume (PV) of the 2 wt% NPs dispersed in the oils was injected into the micromodels, followed by post-flush water flooding with the brine solution. A 0.01 cc/min injection rate was set for following injection tests at room temperature. RF and RRF were calculated as follows:

\[
RF = \frac{\frac{K_{\text{Base water}}}{\mu_{\text{Base water}}}}{\frac{K_{\text{Conformance control fluid}}}{\mu_{\text{Conformance control fluid}}}} = \frac{\Delta P_{\text{Conformance control fluid}}}{\Delta P_{\text{Base water}}} \quad (E1)
\]

\[
RRF = \frac{\frac{K_{\text{Base water}}}{\mu_{\text{Base water}}}}{\frac{K_{\text{Post flush water}}}{\mu_{\text{Post flush water}}}} = \frac{\Delta P_{\text{Post flush water}}}{\Delta P_{\text{Base water}}} \quad (E2)
\]

where \(\mu\) is the viscosity; \(K_{\text{Base water}}\), \(K_{\text{Conformance control fluid}}\), and \(K_{\text{Post flush water}}\) reveal the permeabilities of the system before, during, and after injecting NPs and the polymer solution, respectively; and \(\Delta P_{\text{Base water}}\), \(\Delta P_{\text{Conformance control fluid}}\), and \(\Delta P_{\text{Post flush water}}\) are the pressure differences occurring over the porous media at each stage. During RF and RRF calculations, pertinent phase properties and physical model parameters and oil rate were assumed constant throughout.

3. RESULTS AND DISCUSSION

3.1. Nanoparticle Characterization. Figure 5A,B represents FESEM and TEM photographs of the as-synthesized Fe₃O₄ NPs. FESEM photographs show spherically shaped Fe₃O₄ well coated with citric acid, as confirmed by XRD and FTIR spectroscopy below. The spherical morphology and the relatively narrow size distribution promote effective particle transport through the porous media owing to the high mobility and ease of detachment of the NPs. Figure 5C shows the size distribution of the Fe₃O₄ NPs based on TEM images, which suggests a particle size <80 nm with a size peaking ∼40 nm. These values are consistent with the DLS test of Figure S1. It should be noted that small-size NPs are always desired in order to achieve proper peptization and ensure effective particle transport through the medium.

FTIR and XRD patterns of the as-prepared Fe₃O₄ NPs were obtained. The XRD pattern shown in Figure S2 displays a trace...
amount of the Fe$_3$O$_4$ phase. Pattern identification suggests that the as-prepared Fe$_3$O$_4$ model has an inverse spinel crystal structure. Scherrer’s equation at $2\theta = 30^\circ$ suggests a crystal size of 17 nm.

Figure S2 confirms that the peak is related to citric acid of CACM NPs, whereas noncapped magnetic NPs do not display this peak.

FTIR spectra of the citric acid-capped Fe$_3$O$_4$ NPs and the noncapped magnetic NPs are shown in Figure S3 in the range of 400–4000 cm$^{-1}$. The peaks at 3460, 578, 1788, 1586 cm$^{-1}$, and 833 cm$^{-1}$ belong to H$_2$O in air, the vibration of the Fe–O functional group, C=O,112,113 the stretching of carboxylate COO$^-$, and C–H and vibrations, respectively. In summary, both XRD and FTIR patterns confirm citric acid-capped Fe$_3$O$_4$ NPs.

### 3.2. Conformance Control Experiments

The ability of the Fe$_3$O$_4$ NP organosol to create a solid-/gel-like structure which helps reducing produced water flow was assessed. The performance of the Fe$_3$O$_4$ NP organosol is compared to that of the hematite (Fe$_2$O$_3$) nanoﬂuid and the PAM polymer solution. Magnetite or hematite NPs were dispersed into the two types of oils, heavy and light, and then injected into the micromodel in the presence and absence of a magnetic field. A solid-/gel-like structure is expected for the Fe$_3$O$_4$ NPs due to their nonmagnetic properties. The solid-/gel-like structure of the magnetorheological fluid, in turn, can block the pores and channels of high-permeability zones, and subsequently, water cut reduction happens. Both the magnetic NPs and the transporting fluid considerably define the features of the magnetorheological fluid.

The contact angle between a glass surface which was totally coated by the synthesized NPs and distilled water under atmospheric conditions was 21.4°. Moreover, the IFT between a nanofluid, containing the synthesized NPs and NaCl (40,000 ppm), and crude oil was 10 mN/m under atmospheric conditions. Generally, the amount of dispersed NPs needed for water control purposes is more than that used for EOR purposes. Using magnetic NPs, on the other hand, specific parts of the reservoir can be targeted for water shutoff purposes, while the rest of the reservoir is not impacted. The same water shutoff effect can be attained using conventional methods such as resin injection as the conformance control agent; however, much larger volumes, 1–5 bbl, are needed. It is advantageous to employ the reservoir’s native oil as the carrier fluid since other fluids may lead to asphaltene precipitation/deposition, scale deposition, etc. Moreover, crude oil has greater viscosity than water and hence is capable of transporting a higher fraction of the NPs if needed. Last, crude oil and the Fe$_3$O$_4$ NPs can be simultaneously produced at the producer well, whereby the NPs are separated and reused. Since heavy oil contains more asphaltenes, both its viscosity and its ability to interact with the CACM NPs are higher. Functional groups on asphaltenes, especially the basic ones, readily interact with the citric acid coating, leading to a solid-like/gel-like structure, which is more effectively protected at the higher viscosity of the heavy oil.

#### 3.2.1. Effect of Magnetic Field Strength on RF

Altering the space among two magnets changes the strength of the magnetic field. Consequently, the viscosity of the magnetic nanoﬂuid can be manipulated by selectively forcing the magnetic NPs to accumulate in certain zones, hence increasing the viscosity of the nanoﬂuid in regions with a high water flux. The viscosity, however, increases to a maximum value due to magnetic saturation. Beyond this maximum, increasing the magnetic field spreads the oil too thin over the magnetic field. Hence, finding the optimum severity of the magnetic field is important to minimize energy use. Furthermore, injection of the Fe$_3$O$_4$ nanoﬂuid into the porous medium could reduce water relative permeability. To this end, RF and RRF were used to evaluate the water shutoff treatment. RF serves as a measure of conformance control flooding, whereas RRF gauges the permeability impairment level due to retention of NPs or the polymer in the porous media. Figure 6 depicts the variation of RF for the Fe$_3$O$_4$ nanoﬂuid as a function of magnetic field strength. This variation helps identifying the optimum intensity of the magnetic field. It is concluded from Figure 6 that Fe$_3$O$_4$ NPs commenced responding to the magnetic field at approximately 1000 G of intensity. Depending on the nature of the formation, homogeneous versus heterogeneous, and the viscosity of the base oil, RF of the nanoﬂuid initially increases with the magnetic field strength and then levels off. A magnetic field strength of 6000 G was selected for the subsequent experiments. Figure 6 shows that RF of the heavy oil is higher than that of the light oil owing to its higher viscosity. Moreover, by applying the magnetic field, the shear viscosity of NP conformance control ﬂuids increases, which may alter the mobility ratio in the porous medium and reduce the water cut.

#### 3.2.2. Nanoﬂuids and the Polymer Fluid as Water Shutoff Agents

Figure 7 shows the pressure drop pertaining to the different agents injected through the heterogeneous micromodel. Different fluids were injected for each stage. The first stage involved brine ﬂooding. The pressure drop for this stage closely matched previous water ﬂooding studies and was not impacted by the magnetic field. The second stage involved the injection of a nanoﬂuid or a polymer solution and corresponded to high pressure drops, especially for the Fe$_3$O$_4$ NPs, for both the heavy and light oils. Given the orientation of the magnetic field, that is, in the same direction as the micromodel, columnar structures close to the magnetic field arose corresponding to the formation of a solid-/gel-like structure. The viscosity of the Fe$_3$O$_4$ nanoﬂuid increases in zones targeted by the magnetic field, and subsequently, water occupying these zones gets trapped, leading to a lower overall water cut. Figure 7 also shows that polymer ﬂooding displayed a higher pressure drop than the nonmagnetic and magnetic nanoﬂuids in the absence of the magnetic field. A high
pressure drop is typical for polymer flooding, which increases the viscosity of the base fluid.135 The third stage, that is, flush water flooding, the pressure drop decreased gradually to a level higher than the initial water flooding for all the fluids employed. The higher level of pressure drop in the post-flood flush is a result of retention of some NPs or the polymer in the porous media leading to permeability reduction.6,138,139 Furthermore, magnetic NPs are easily recoverable using magnets and can be reused several times. The CACM NPs can be prepared by a facile and inexpensive experimental protocol. The application of the magnetic field to a reservoir during production, on the other hand, may require an innovative technology.6,138,139
values of the Fe3O4 NP agent were higher than those of the retention tendencies of the agent within the porous media. RRF solid-/gel-like structure. Fe3O4 NPs dispersed in heavy oil presence of the magnetic micromodels. A general comparison between Figures 9 and 10 magnetic fi well as the polymer solution in the presence and absence of a mobility ratio and may lead to diminishing water cut.

According to the Darcy equation, viscosity is crucial factor in the viscosity of the heavy oil which also promoted NP peptization. corresponded to a higher RF than light oil owing to the higher RF pertains to reduced water relative permeability, which entails Fe3O4 NP potency as a conformance control agent. RRF values were calculated during post-injection of an agent. The RF value for Fe3O4 NPs in the presence of the magnetic field is the highest and suggests a solid-/gel-like structure. Fe3O4 NPs dispersed in heavy oil corresponded to a higher RF than light oil owing to the higher viscosity of the heavy oil which also promoted NP peptization. According to the Darcy equation, viscosity is crucial factor in the mobility ratio and may lead to diminishing water cut.

In addition, RF pertains to reduced water relative permeability, which entails Fe3O4 NP potency as a conformance control agent. RRF values were calculated during post-flush water flooding, where the RRF values slowly decreased and reached a constant for all agents. Different levels of RRF demonstrate different retention tendencies of the agent within the porous media. RRF values of the Fe3O4 NP agent were higher than those of the polymer solution in attendance of a magnetic field but lower in the nonexistence of a magnetic field. The lower RRF value of the Fe3O4 NPs in the absence of the magnetic field than the polymer solution suggests lower potency of the NPs toward formation damage. Consequently, the magnetic field must be deactivated in order to achieve a low RRF value during post-flooding flush. Furthermore, resuspension of the Fe3O4 NPs and the reversibility to a solid-/gel-like structure to a liquid-like structure in the presence and absence of a magnetic field is essential for water shutoff applications. Figure 11 confirms that...
magnetic Fe₃O₄ NPs via a facile, large-scale-applicable technique were tested as water shutoff regents. The particle size distribution, identity, and nature of interaction with citric acid were confirmed using FESEM, TEM, DLS, FTIR, and XRD techniques. Uniform, spherical, citric-acid-coated Fe₃O₄ particles ranging in size from 10 to 66 nm have been synthesized. The following conclusions can be drawn from the observations made in this study:

1. Fe₃O₄ NPs reacted to the magnetic field at ~1000 G of intensity. A constant trend for RF within the intensity range of 4500–6000 G has been observed. Accordingly, an excessive increase in magnetic field did not show enhanced plugging performance.

2. Light and heavy oils containing Fe₃O₄ NPs displayed a higher pressure drop in the presence of a magnetic field than Fe₂O₃ NPs and PAM polymer systems. This observation is attributed to a solid-like structure attained by concentrating the Fe₃O₄ NPs in certain zones upon the influence of the magnetic field.

3. Heavy oil as a carrier fluid induced a high pressure drop owing to its higher viscosity.

4. The Fe₃O₄ NPs displayed the highest RF and RRF values of 3.5 and 2.14 for heavy oil and the heterogeneous micromodel in the presence of a magnetic field, respectively. Furthermore, these values of RF and RRF reflect the ability of Fe₃O₄ NPs to reduce water relative permeability. This improvement is due to creation of a solid-/gel-like structure within the pores and the pore throats of the micromodels, which invites an effective water control strategy.

**ASSOCIATED CONTENT**

**Supporting Information**
The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.1c00026.

Characterizations of nanoparticles (PDF)

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**Notes**

The authors declare no competing financial interest.

**NOMENCLATURES2-COL**

API American Petroleum Institute

EOR enhanced oil recovery

IFT interfacial tension, mN/m

NPs nanoparticles

ppm parts per million

RF resistance factor

RRF residual resistance factor

CACM citric acid-coated magnetite

FTIR Fourier-transform infrared spectroscopy

FESEM field emission scanning electron microscopy

DLS dynamic light scattering

MW molecular weight

XRD X-ray diffraction

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