



Research article

Conformance control in oil reservoir based on magnetorheological behavior of nanoparticle suspension

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ABSTRACT

Water shut off and performance control in oil reservoirs involve many techniques both for reducing the water cut and for enhancing oil production with the aim of making it economical and environmental friendly. Therefore, suitable nanoparticles for injection in an oil reservoir regarding nano size, spherical morphology, and better dispersibility were synthesized by one step, facile, and inexpensive method and then characterized in this work. In addition, new magnetorheological (MR) fluids based on the crude oil and the nanoparticles were developed, and the analysis of their rheological properties carried out by rotational and oscillation tests showed their ability of forming gel-like structure. Furthermore, from the core flooding experiment investigated, values of both resistance factor and residual resistance factor showed that the MR fluids exhibit a solid-like form with the magnetical field applied in oil reservoirs, thereby reducing the water cut.

1. Introduction

Water shut-off techniques that are used for reducing water cuts in crude-oil fields (Liu et al., 2010; Sharifpour et al., 2015) have drawn considerable attention recently because surplus water production, generally in matured oil reservoirs, is a severe problem: (Arnold et al., 2004), for producing one barrel of oil, three barrels of water are generated worldwide (Veil et al., 2004) and even worse, seven barrels are generated in the United States (Bailey et al., 2000). Incremental water production in mature reservoirs can increase costs associated to corrosion, scaling, and water-oil separation, and shutting-in the well (Zhdanov et al., 1996), and can also affect environmental aspects such as degradation in soils and surface and ground water quality (Rahbari-Sisakht et al., 2017). Thereby, water shut-off/conformance control remains an important environmental and financial mission in oil reservoirs (Elsharafi and Bai, 2012).

There are two main techniques for conformance control. The first one is to enhance shear viscosity of a driving fluid during flooding

operation with a polymer solution (partially hydrolyzed polyacrylamide (HPAM) (Jung et al., 2013; Pei et al., 2017), xanthan gum (Alquraishi and Alsewailem, 2012; Jang et al., 2015; Olajire, 2014), and an associative polymer (Liu et al., 2017b) or foam (Liu et al., 2017a), and the second one is the permeability reduction treatment in which various materials such as silicate gel (Nasr-El-Din and Taylor, 2005), polymer gel (Bai et al., 2015), micro gels (Abdulbaki et al., 2014; Saghafi et al., 2016), and others (Dai et al., 2016) could be used. In the former technique, a huge volume of conformance control fluid is needed to affect almost all areas in the reservoir, but in the later one, an intermediate volume is enough for permeability modification. In addition to all these mentioned methods that need a considerable volume for operation, the use of a resin facilitates conformance-improvement fluid shut-off: the resin can usually only be located in a wellbore, perforation, and an extra near-wellbore highly permeable flow channel (Kabir, 2001), and therefore, a low volume, i.e., only 1–5 barrels, is sufficient (Sydansk and Romero-Zern, 2011). For all of those materials, ideally, the viscosity should at first be low so that they can be pumped

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and injected in the reservoir easily, and then, the viscosity should increase or a solid structure such as a gel should be formed when the material reaches the final target location (Goudarzi et al., 2015). This is because injecting a viscous fluid involves significant energy consumption, and the process is difficult and costly (Lee, 2011). In addition, during injection, the original viscosity can be lost due to a high shear stress (Rezaei et al., 2016). Therefore, in situ viscosity enhancement (Imqam and Bai, 2015; Karimi et al., 2014) or the use of a viscosity-adjustable fluid (Yahya et al., 2014) is preferred. Magnetorheological (MR) smart fluids can be adjustable under applied magnetic fields and show rapid and reversible liquid-to-solid-like transformation (Dong et al., 2015; Sedláček et al., 2010); thus, they are ideal for conformance control and water shut-off at oil reservoirs. These smart fluids basically consist of magnetic particles (Wang et al., 2016; Yang et al., 2016) in a medium of oil (Iglesias et al., 2015; Kim and Park, 2016). In an absence of magnetic field applied, the magnetic particles are disorderly suspended in a medium fluid and the suspension becomes a Newtonian fluid (Wang et al., 2017). However, as soon as a magnetic field strength is applied, those magnetic particles form the columnar structure in the direction of magnetic field induced (Hajalilou et al., 2016), and the shear stress, yield stress, and storage moduli increase dramatically (Esmailnezhad et al., 2017b; Fang et al., 2009b). The solid-like structure of the MR fluid can act as a gel in an oil reservoir and block the pores and channels through a thief zone in the reservoir; therefore, water cannot flow easily and water cut will decrease. Although both the magnetic particles and carrier fluid significantly determine the characteristics of the MR fluid (Kim et al., 2012), and the magnetic particles should possess some special characteristics to be applicable in oil reservoirs (Ehtesabi et al., 2013; ShamsiJazeyi et al., 2014; Sun et al., 2017). There are some studies both about magnetic nanoparticles like as Fe_2O_3 (Joonaki and Ghanaatian, 2014) and Fe_3O_4 (magnetite) (Kazemzadeh et al., 2015) for the enhanced oil recovery (EOR), and also some reports about employing an external magnetic field onto oil reservoirs (Bera and Babadagli, 2015, 2017; Esmailnezhad et al., 2017a). It has been proved that magnetite nanoparticles are ideal for experimental purposes at the oil and gas industry as they exhibit a higher magnetic permeability and susceptibility at 373 K and 300 K, respectively (Avendano et al., 2012), and also can be injected in reservoirs even at high concentration without substantial retention (Yu et al., 2010). Our goal in this study is to introduce a conformance control fluid based on a MR fluid that can exhibit a solid-like form in the reservoir after a magnetic field is applied. As a good alternative to other techniques that have many drawbacks (Sydansk and Romero-Zern, 2011), we propose a method in which magnetite will be dispersed in the produced oil from the reservoir and then re-injected in a low volume to the thief zone of the reservoir to result in a solid structure after the application of a magnetic field, to prevent water production from the highly permeable zone. Using this method just nanoparticles will be added to the already-existing system in reservoir and changes with respect to the wettability, surface tension, scale, etc., will be kept to a minimum. The two main advantages of the proposed method are as follows: First, this solid-like structure is independent of temperature and can be created even in a high-temperature reservoir. Second, it can rapidly revert to the liquid form without a magnetic field, while in other methods, this reversible behavior from the gel-like structure to a liquid form is impossible (Goudarzi et al., 2014) or at least difficult (Choi et al., 2010) and time consuming (Vernáez et al., 2016). In the first step, magnetite nanoparticles were synthesized on a large scale by a simple one-step process, and then, the size and magnetic properties that are the main requirements for production and application of nano-particles in an oil reservoir, were measured. As a second step, the tenability of solid-like behavior through a rheological experiment was studied and finally this ability was confirmed in a core flooding experiment.

2. Experimental

2.1. Materials

For preparing the magnetic conformance control fluid, magnetite as a dispersed phase was fabricated following the procedure formerly reported (Hui et al., 2008) with slight modifications: briefly, NaOH (4 mmol) (Merck, Germany), citric acid trisodium salt dehydrate (1 mmol) ($\text{C}_6\text{H}_5\text{Na}_3\text{O}_7 \cdot 2\text{H}_2\text{O}$ Sigma-Aldrich, USA), and NaNO_3 (2 mmol) (Daejung Chem., Korea) were well dissolved in deionized water (19 mL) and heated to around 100 °C. 2 M $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (1 mL) (Yukuri Pure Chem., Japan) solution was then inserted quickly while keeping its temperature constant for 1 h. After naturally cooling, the precipitated magnetite was separated using a magnet and then cleaned using water. This synthesis method is facile and inexpensive, and it has real scale performance capability (Ryoo et al., 2012). Two different kinds of light and heavy oils respectively, from Kuwait (gravity [API] and viscosity at 71 °C [cP] were 30.6 and 16.24, respectively) and Van Gogh in Australia (gravity [API] and viscosity at 71 °C [cP] were 17.1 and 355.5, respectively), were selected as a continuous phase for proposed magnetic conformance control fluid. NaCl (Daejung Chem., Korea) and CaCl_2 (Duksan Chem., Korea) were adopted to prepare brine solutions for connate water and also water flooding via the core test.

2.2. Characterizations

Morphology of the magnetite was considered using both scanning electron microscope (SEM) (S-4300, Hitachi, Japan) and transmission electron microscopy (TEM) (Philips CM200, USA), while its particle size distribution was inspected with particle size analyzer (ELS-Z, Otsuka, Japan). Magnetic property of the magnetic particles was measured in their powder sample at the ambient temperature via vibrating sample magnetometry (VSM) (7407, Lake Shore, USA). More complete characterizations have been reported elsewhere (Esmailnezhad et al., 2018a). MR properties of two magnetic conformance control fluids were tested by rotation rheometer (parallel-plate type with a diameter of 25 mm) (MCR 300, Anton Paar, Austria) furnished with a magnetic field generator. Gas permeability of core samples was examined by steady-state gas permeameter (Gasperm, Vinci Technologies, France).

2.3. Preparation of core and fluid

A same procedure was followed for four cores of Berea sandstone in which their gas permeabilities were measured by the gas perimeter and saturation was then performed with brine (4.2 wt% NaCl and 1.8 wt% CaCl_2) by using a vacuum unit for at least 6 h with soaking for over two nights to attain ion equilibrium between the brine and rock surface. While their saturation process was being performed, their porosity was also investigated based on their wet and dry weights. Each core was input horizontally into the core holder while a confining pressure of around 700 psi was input on the core; then, the liquid permeability of each core was studied by injecting samples with several pore volumes (pv) with brine at different flow rates. Thereafter, light and heavy crude oil samples were injected in the cores according to the selected cores (Table 1) at different rates to reach irreducible water saturation and drain formation water as much as possible. Following this, the core was aged in crude oils for at least two weeks to make the wettability of the rock stable. Finally, the cores were ready for the core flooding experiment. As mentioned before, two types of crude oil from Kuwait and Van Gogh in Australia were injected in cores and used for making conformance control fluids.

Table 1
Properties of Berea sandstone cores.

Parameter	Core name			
	Core G	Core H	Core F	Core I
Length [mm]	92.45	93.30	93.50	91.75
Diameter [mm]	38.01	38.00	37.95	38.00
Porosity [%]	18.85	19.23	21.91	18.87
Gas Permeability [mD]	127	143	227	128
Liquid Permeability [mD]	57	66	109	71
Irreducible Water Saturation [%]	34.22	35.11	30.91	38.86

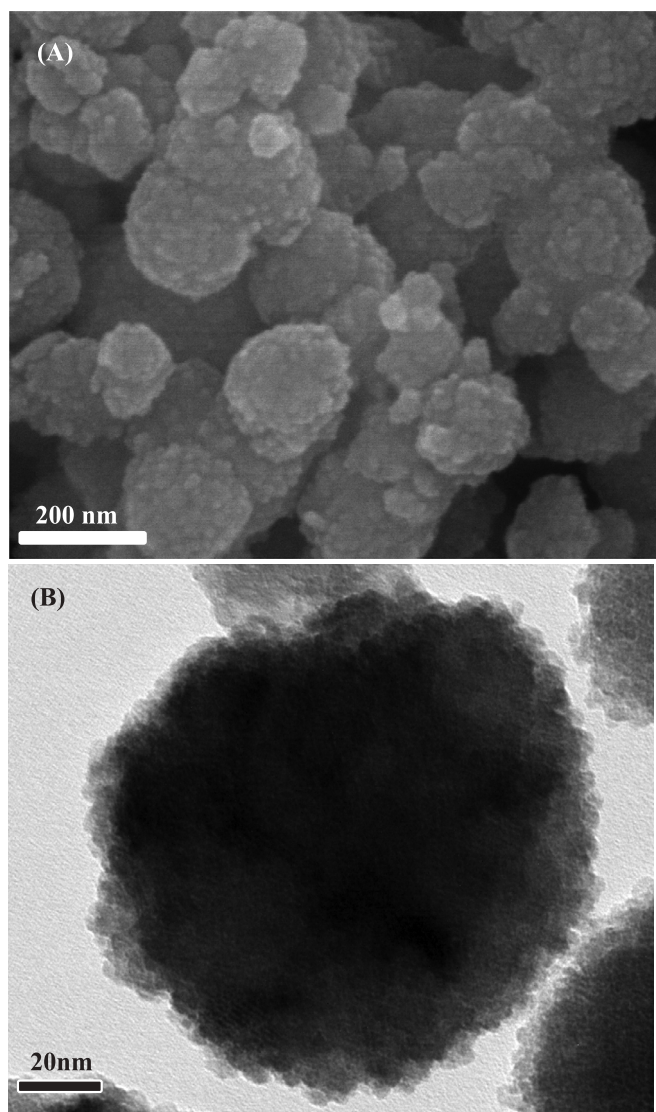


Fig. 1. (A) SEM and (B) TEM images of magnetite particles.

3. Results and discussion

3.1. Magnetites and their MR behavior

Fig. 1 shows both SEM and TEM images of synthesized NPs that indicate morphological image of synthesized magnetite in which spherical nano-sized magnetite particles were used as this morphology makes facile transport through reservoir rocks (Moradi et al., 2015; Mousavi et al., 2013) due to the high mobility and easy detachment (Xu et al., 2016). Furthermore, a saturation magnetization is an important parameter affecting the MR efficiency Fang et al. (2009a), and this

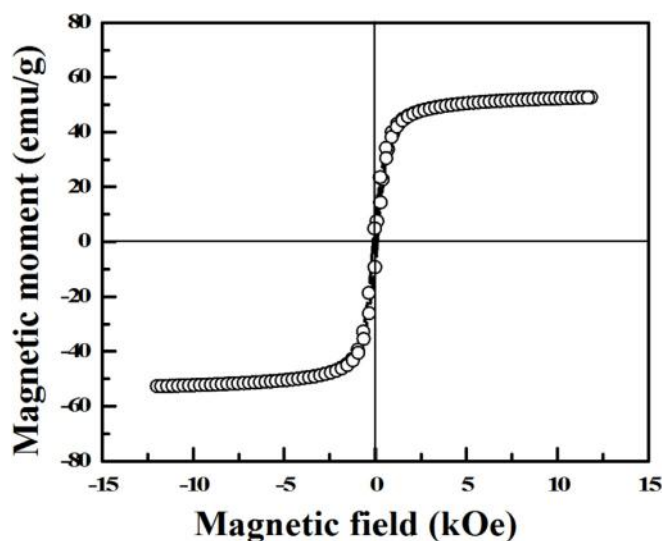


Fig. 2. VSM data of magnetite particles.

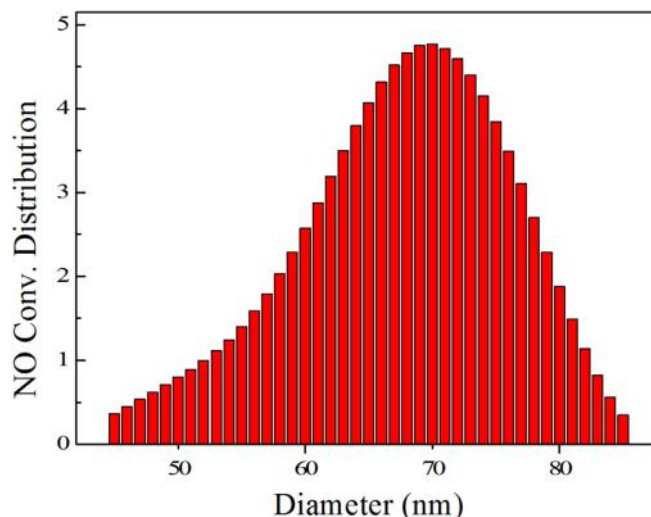


Fig. 3. Particle size distribution of the magnetite.

value according to Fig. 2, showing a rather small magnetic hysteresis loop of the magnetite powder from -12 kOe to 12 kOe (from -955 to 955 kA/m), is around 60 emu/g. Fig. 3 displays the magnetite particle size distribution with an averaged particle size of 70 nm which is appropriate for oil reservoir applications because in the oil reservoir with its permeability higher than 0.1 mD, the average radius of the pore throats could be larger than $1 \mu\text{m}$ (Xi et al., 2016) or even $2.0 \mu\text{m}$ in the shale reservoirs (Li et al., 2016).

On the other hand, although the proposed magnetic conformance control fluid will be finally used in the porous media, the MR behavior has to be considered using a rheometer first. The main factor is a weight percent of the dispersed phase in a continuous state because a low concentration of magnetite might not show MR properties, and a high concentration cannot be injectable through core samples. To find an optimum concentration, we performed a steady shear rate study using several magnetite weight percent values at different magnetic field strengths. For preparing MR fluids, the calculated amount (based on the desired weight percent concentration) of magnetite is well dispersed in two kinds of crude oil as a carrier fluid by using a vortex. A magnetic field strength sweep test was performed in a fixed shear rate (7.3 1/s), which is a dominant shear rate in the porous media in oil reservoirs, and this shear rate is proposed for laboratory studies in the literature

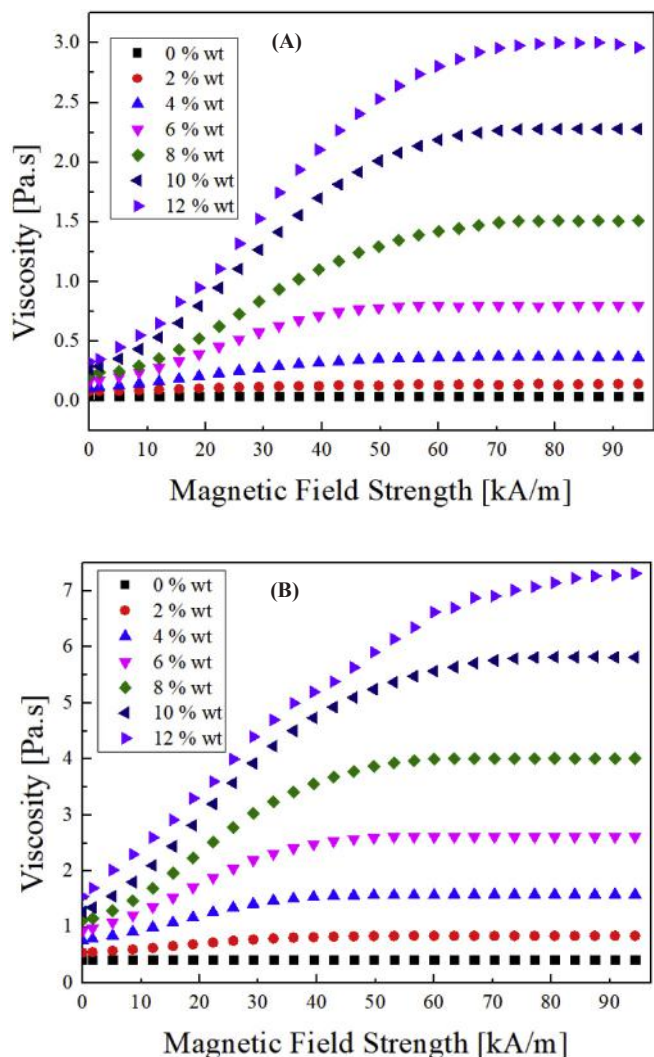


Fig. 4. Magnetic field strength sweep test in several weight percent of magnetite (A) in light and (B) in heavy crude oil.

(Kamal et al., 2015). Fig. 4 shows the magnetic field strength sweep test results for several weight percent values of magnetite (A) in light and (B) in heavy crude oil in a fixed shear rate (7.3 1/s). For all suspensions, saturation occurs at a given magnetic intensity, and thereafter, shear viscosity keeps a fixed value even though the magnetic intensity is increased. This means that after a threshold value, increasing the magnetic field strength does not affect the viscosity. This is very important especially for real scale operation as it confirms that increasing the magnetic field strength is effective only up to a certain limit and can just affect the distance that magnetic field goes far from the source point.

Another point is that magnetic saturation for different suspensions occurs at different magnetic field strength values, and these values increase with the concentration. Since the viscosity of the suspension during the flooding process is very crucial for real-scale application (Bai et al., 2017), the fact that the difference in the viscosity between the pure crude oil (0 wt% nanoparticles) and other suspensions in the absence of magnetic field strength induced is not large was a key advantage of our proposed method. The magnetic field can be applied in reservoirs after injection is completed to prevent excess pressure during the injection process. The viscosity of the continuous phase is a crucial parameter that influences the sedimentation rate of nanoparticles (Cvek et al., 2016); therefore, a higher concentration for heavy oils and lower concentration for light oils can be selected. Here, we select 2 wt% for

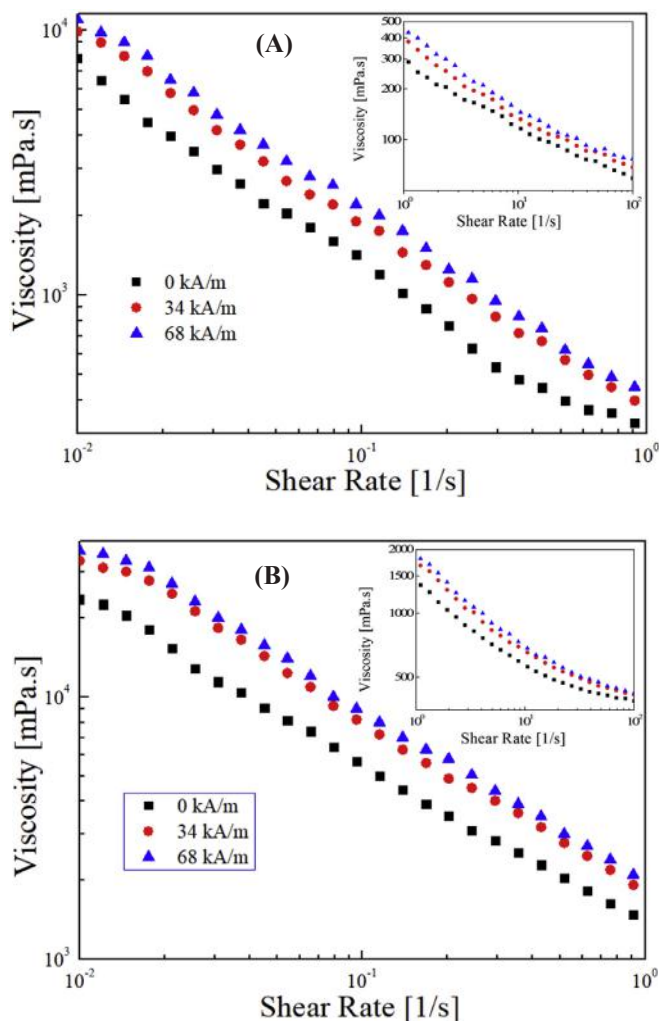


Fig. 5. Flow curve test of conformance control fluids (A) magnetite in light and (B) magnetite in heavy crude oil at different magnetic field strength.

both of heavy and light oils to perform MR studies and also core flooding tests because the MR effect can be seen for this suspension even at a low concentration. Thereafter, 2 wt% synthesized nanoparticles were dispersed in each of the crude oil samples, and the flow curve in the range of 0.01–100 s⁻¹ were plotted for three different magnetic field intensities. As given in Fig. 5, the shear viscosities of both conformance control fluids increased by applying magnetic fields that can affect mobility ratio in the reservoir and decrease the water cut; this is because viscosity is one of the main parameters in the Darcy equation following that govern the fluid flows through porous media (Karambeigi et al., 2016):

$$Q = \frac{KA dp}{\mu dx} \tag{1}$$

Here Q is a flow rate (cm³/s), A is a cross-section area (cm²), K is a permeability (D), dp/dx is a pressure gradient in core direction (atm/cm), and μ is liquid viscosity (cP); for a fixed geometry, any increase in the viscosity will also cause the pressure to increase, so water needs extra pressure to bypass conformance control fluid and thereby the water production decreases (Bai et al., 2007).

Two of important parameters for evaluating performance of the conformance control fluid are elastic modulus (G'), representing an elastic response of the fluid to store elastic energy which could be recovered afterwards and loss modulus (G''), representing irreversible energy portion that has been consumed to start the flow (Goudarzi

et al., 2015; Al-Muntasheri et al., 2007; Durán-Valencia et al., 2014). The ratio of G''/G' is a tangent of loss angle or phase angle (δ) between the strain and stress through the oscillatory shear test (Hatzignatiou et al., 2016), and the total resistance of conformance control fluid against ultimate applied strain is shown by a complex modulus (G^*) (Vargas-Vasquez and Romero-Zerón, 2008) which is expressed as follows (Koohi et al., 2010):

$$G^* = G' + i G'' \quad (2)$$

In which:

$$G' = G^* \cos \delta \quad (3)$$

$$G'' = G^* \sin \delta \quad (4)$$

When the viscous part still governs the elastic portion, $\tan \delta$ is bigger than one and the system is considered to have a liquid-like behavior (viscoelastic liquid); further, when the elastic part is above the viscous contribution, $\tan \delta$ is smaller than one and it shows gel-like characteristics (viscoelastic solid) (Mezger, 2015). Therefore, for the performance control purpose, a fluidic system with a higher G' is desirable. The results of the stress sweep test are given in Fig. 6 in which for the conformance control fluid in the light oil (Fig. 6 (A)), G' and G'' are dependent on the stress in zero magnetic field, but with an applied magnetic field intensity, the complex moduli are independent of shear stress and has a plateau state even it is increasing with increased magnetic field intensity. For a conformance control fluid in the heavy

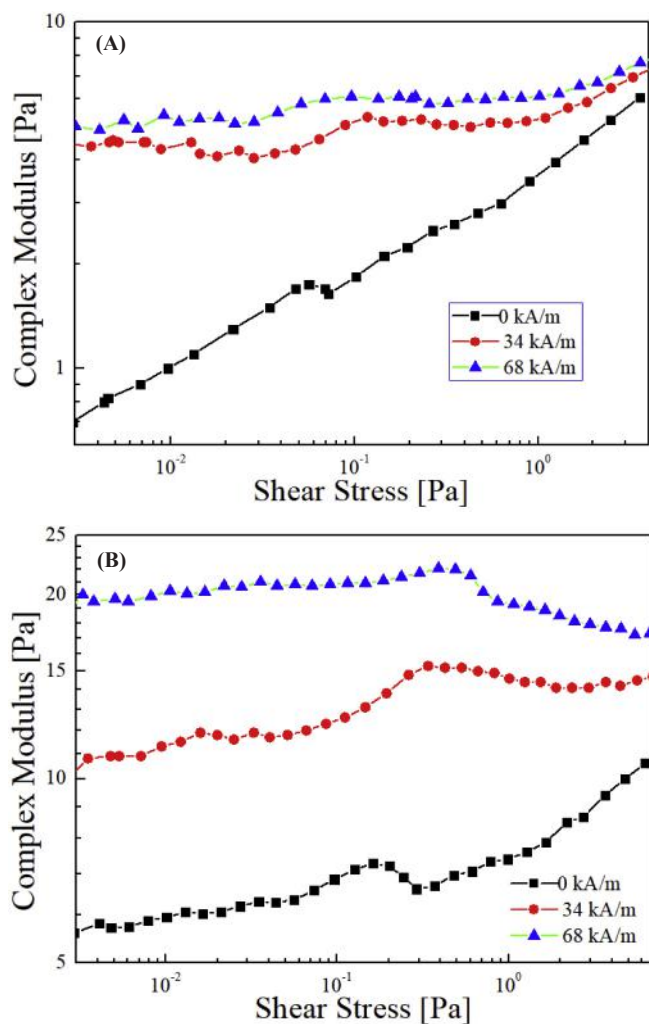


Fig. 6. Complex modulus as a function of stress for conformance control fluids (A) magnetite in light and (B) magnetite in heavy crude oil at different magnetic field strength, $f = 1$ Hz.

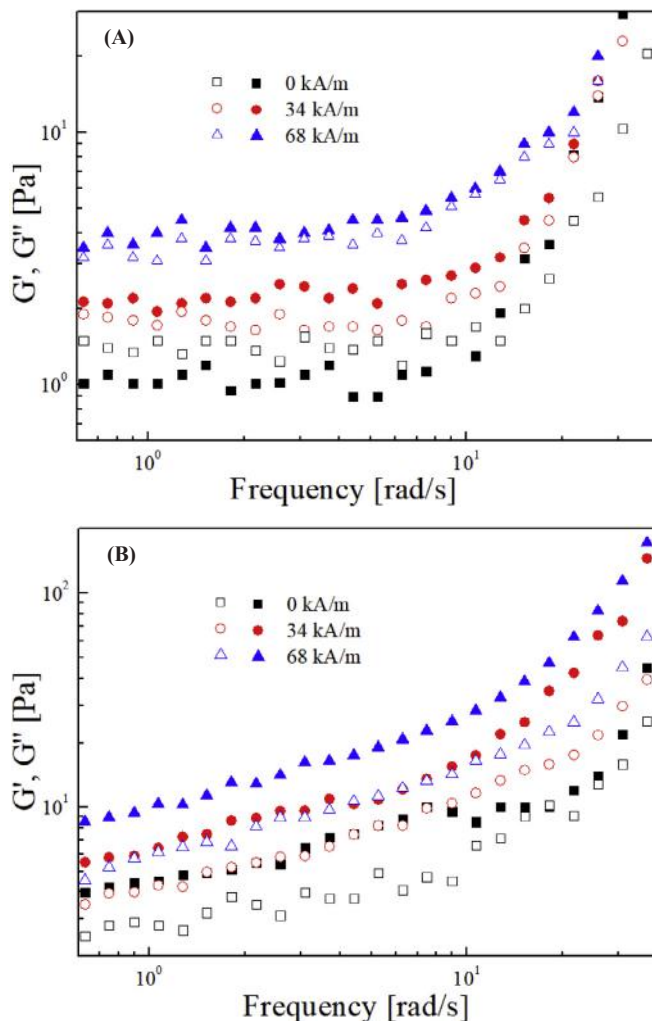


Fig. 7. G' (closed) and G'' (open) as a function of the frequency for conformance control fluids (A) magnetite in light and (B) magnetite in heavy crude oil at different magnetic field strength, strain = 0.02%.

oil (Fig. 6 (B)), the complex moduli increased dramatically after the magnetic field strength was adopted due to the tight column structure through strong dipole-dipole interaction among neighboring magnetic particles (Fang et al., 2011). Based on the independence of the complex modulus to shear stress and the significant increase with an applied magnetic field, it is obvious that proposed conformance control fluids can become solid-state structure like a gel, possessing the ability for water shut off. In general, it is usually confirmed that the systems are in a linear viscoelastic (LVE) area before the dynamic oscillation test is carried out.

For both conformance control fluids, G' and G'' were investigated through a frequency sweep test. For the conformance control fluid in the light oil (Fig. 7(A)), G' is lower than G'' in the zero magnetic field, showing the classical characteristic of the disordered structure at a low range of frequencies; however, in a high range of frequencies, the suspension shows a solid-like character. After the magnetic field is on, the G' became higher than the G'' and the solid-like nature can be seen in all frequency ranges. For the conformance control fluid in the heavy oil, G' is higher than G'' (Fig. 7(B)) in all ranges of frequencies tested especially after the application of the magnetic field intensity, and it could be considered that a solid-like structure was formed after the magnetic field was applied.

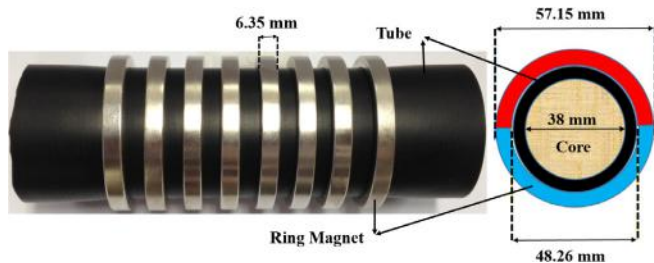


Fig. 8. Set up of magnet for applying magnetic field on core samples during flooding.

3.2. Core flood experiment

A very common set up for the core flooding experiment (Pu et al., 2016; Suman, 2014; Esmailnezhad et al., 2018b; Varjani and Upasani, 2016) was adopted to investigate the ability of the conformance control fluids for reducing the water cut. For applying the magnetic field, eight permanent ring magnets (R2250, Super Magnet Man, Alabama, USA) were installed on the tube of the core holder, as shown in Fig. 8. We used a permanent magnet instead of the other types such as an electromagnet, to increase accuracy and precision. This is because while using a permanent magnet the temperature can remain constant, but using an electromagnet especially for a long time can increase the temperature and affect the results due to viscosity reduction. Core (G) and core (H) for the light oil and core (F) and core (I) for the heavy oil were selected. To examine effect of magnetic field strength clearly, a following experiment was performed for core (H) and core (I) without and core (G) and core (F) with the applied magnetic field. Each aged core was horizontally located in the core holder and an extra-burden pressure was enforced. The first step of the test was started with the previously mentioned brine flooding. By considering the conventional front rate in a reservoir (1 ft/day), an injection flow rate was chosen for all steps. This step continued until the residual oil saturation was realized and oil cut was less than 1%. In the second step, the conformance control fluid in the light or heavy oil (depending on the selected core) was injected, and consequently, in the post-flush water flooding steps, brine was injected again to reach the primary situation.

The pressure drops of cores in both light and heavy oils were compared with or without an induced magnetic field to investigate performance of the conformance control fluids. Fig. 9 shows the pressure drop during the first step of the test agreed well with values previously reported (Almansour et al., 2017; Cardoso and Balaban, 2015; Kazempour et al., 2012; McMillan et al., 2016; Shojaei et al., 2015): the pressure drop is almost the same regardless to the magnetic field because in this step, there are no magnetic nanoparticles in cores and therefore no reaction due to magnetic field occurs. In the second step, when the injection of the conformance control fluids was started, the differential pressure became higher under the applied magnetic field because magnetite nanoparticles form a column through the pore throat; hence, a solid-like structure was formed and the pressure-drop increased. Based on this step, we suggest that in field scale, it is better that the magnetic field application start after this step is completed to facilitate the injection process. Also, in the third step, the pressure drop is higher, while applying magnetic field due to same reason about making column in pore through and decrease to a threshold higher than first step, as a result of some permeability reduction due to nanoparticles absorption or trapping through the core that is normal during nano flooding (Parvazdavani et al., 2014; Zheng et al., 2017). These results agree well with rheological properties discussed in section 3.2.

Conformance control fluids can decrease the effective permeability of water, and to study this ability, the resistance factor (RF) can be used. RF is a proportion of a differential pressure for the conformance control fluid injection to that for the primary water injection (Shedid, 2006):

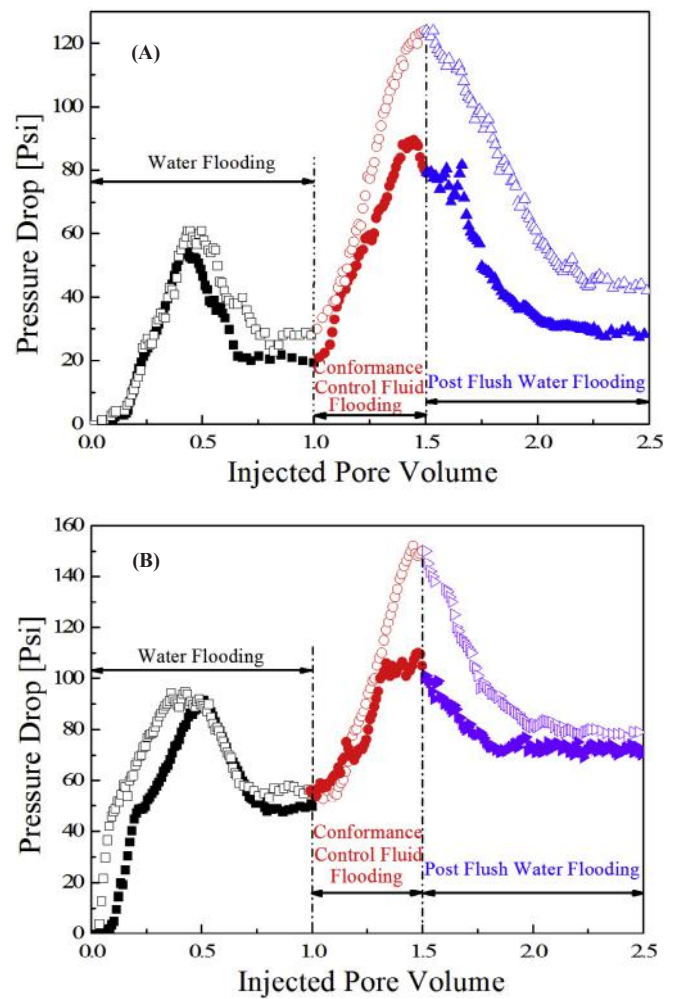


Fig. 9. Pressure drop during core flood experiment. (A) core G in magnetic field (open) and core H without magnetic field (closed) in light oil, (B) core F in magnetic field (open) and core I without magnetic field (closed) in heavy oil.

$$RF = \frac{(k_{water}/\mu_{water})_{Base\ water}}{(k_{conformance\ control\ fluid}/\mu_{conformance\ control\ fluid})} = \frac{\Delta P_{conformance\ control\ fluid}}{\Delta P_{Base\ Water}} \quad (5)$$

in which K and μ are permeability and viscosity, respectively.

In order to ascertain that the reduction in the permeability will remain in the post-flush water flooding, a residual resistance factor (RRF) represents a proportion of differential pressure for post-flush water flooding to that for an initial water flooding as follows (Shi et al., 2010):

$$RRF = \frac{(k_w/\mu_w)_{Base\ Water}}{(k_w/\mu_w)_{Post\ Flush\ Water}} = \frac{\Delta P_{Post\ Flush\ Water}}{\Delta P_{Base\ Water}} \quad (6)$$

Fig. 10 shows RF and RRF of the conformance control fluids in which the ability of the conformance control fluid for forming solid-like structures and maintaining this characteristic has been proved. The proposed conformance control fluid based on the MR effect of nanoparticles in the crude oil can be injected at a low volume and can be a good alternative for other conformance control fluids especially for resin. The solid-like structure can be made whenever or wherever needed throughout the reservoir with a locally applied magnetic field and could be removed easily by stopping the magnetic field strength applied. Another point is that crude oil can be produced again and magnetic nanoparticles can be easily adsorbed on the magnet for recirculation (Ko et al., 2016; Liu et al., 2014).

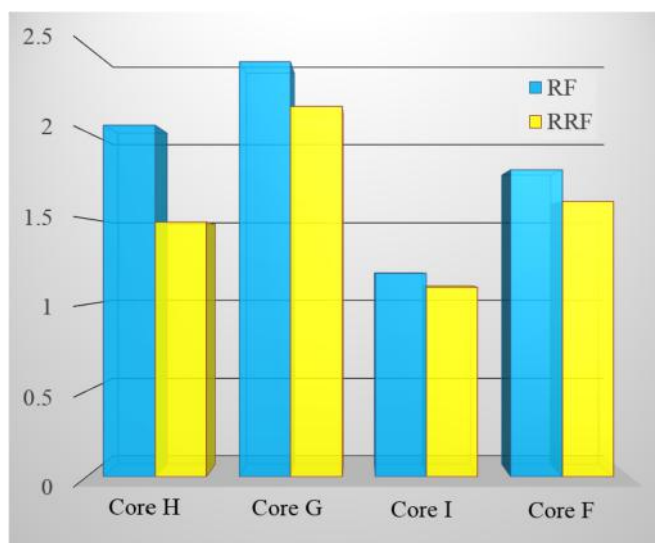


Fig. 10. Resistance factor (RF) and residual resistance factor (RRF).

4. Conclusion

Due to high-volume water production and also the inverse environmental effect of the produced water, the water shut off process is an important target in oil-field engineering. However, there are many limitations and drawbacks of each technique. In this study, the performance evaluation of a new conformance control fluid based on the MR effect is investigated. For this purpose, magnetite nanoparticles were fabricated in a facile, one step and inexpensive procedure to make the nanoparticle synthesis on a large scale feasible. Magnetite nanoparticles showed appropriate characteristics for injection to the oil reservoir in terms of the size and morphology and magnetic properties for preparing MR fluids. Two types of crude oils with a wide range of API that can be called light oil and heavy oil were selected as the carrier fluid for the MR fluids to be used as the conformance control fluids. A series of rheological tests, including magnetic field strength sweep test and rotational and oscillation tests were performed to select the nanoparticle concentration in the carrier fluid and evaluate the MR effect. MR studies using the rheometer showed that both proposed conformance control fluids in light oil and heavy oil have this ability to form solid-like structures by applying a magnetic field because its elastic modulus is higher than its loss modulus. To verify the former ability in porous media, four Berea sandstone cores (two for the light oil and two for the heavy oil) were studied by performing the core flooding test with and without applied magnetic field intensities. To compare effect of applied magnetic field, one core of each crude oil category without the magnetic field and another one with the magnetic field were investigated. The pressure drop increased even without the magnetic field due to the reduction of permeability due to nanoflooding, and it increased dramatically after the magnetic field was applied. This shows the ability of the magnetite nanoparticles for forming columns in pores as is expected for MR fluids. RF and RRF also showed the permeability reduction not only during the injection of the conformance control fluid but also during the post-flush water flooding. Overall, it is concluded by performing rheological studies and the core flooding experiment that the conformance control fluids based on both the light and heavy oils can form the solid-like state required for reducing water production.

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