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# Investigating the effect of hybrid silica nanoparticles-copolymer on increasing oil recovery in a three dimensional porous media

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## KEYWORDS

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3D-micro-model;  
Sulfonated copolymer;  
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Hybrid;  
Inherent viscosity;  
Interfacial tension;  
Contact angle.

**Abstract.** In this work, hybrid of silica nanoparticles (NPs) with sulfonated copolymer has been prepared in order to improve the copolymer properties for Enhanced Oil Recovery (EOR). Some tests are done to find the effectiveness of injecting fluid on Recovery Factor (RF), such as Contact Angle (CA), interfacial tension (IFT), inherent viscosity, and eventually Micro-Model (MM) flooding. In CA test, wettability alteration from Oil-Wet (OW) to Water-Wet (WW) is reached by sedimentation and adsorption of NPs on the rock slice. In addition, IFT reduction is obtained by increasing the NaCl concentration. The viscosity change is investigated for the hybrid and copolymer under simulated high-temperature oil reservoir conditions. It is found that silica NPs-copolymer hybrid exhibits better inherent viscosity and thermal stability than copolymer alone does. At MM flooding, more RF and produced oil in Water (O/W) emulsion are obtained from hybrid than from polymer alone. Hybrid injection results in IFT reduction, increment in viscosity, and O/W emulsion formation that cause enhancement in capillary number ( $N_{Ca}$ ) and RF, and, subsequently, reduction in residual oil saturation. Therefore, NPs-copolymer hybrid could be a much better candidate than even high-performance polymer solutions for EOR process.

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## 1. Introduction

By production of hydrocarbons from oil reservoirs, the reservoir pressure decreases until oil cannot be produced spontaneously on the surface. This stage is called “the late second age” or “beginning of the third age” of the oil reservoir that needs application of

Enhanced Oil Recovery (EOR) methods to produce the remained oil. Chemical flooding is one of the efficient EOR methods as a tertiary oil recovery method. In this straight, new materials and substances must be used to face complex oil reservoir. Complexity in the reservoir porous media should be considered to predict the real reservoir behaviour. Recently, Golshokooh et al. [1] presented a patent using a new idea for fabrication of a novel porous medium with controllable characteristics. This idea could also be used for fabrication of Micro-Model (MM) with a controllable characteristic that is applicable in many areas such as petroleum and chemical engineering, and water and pollution studies.

MM is a visual porous medium that signifies the

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Recovery Factor (RF) and mechanism of EOR, especially for investigating effectiveness of new materials such as nanoparticles (NPs) and polymers. Usually two common types of MMs are used to flood and investigate the fluid flow into the porous media: laser and chemical methods. Laser MM with a CO<sub>2</sub> laser machine was used to construct flow patterns on a glass surface, comprehensively, by Mohammadi et al. [2]. Also, Hekmatzadeh et al. [3,4] studied this kind of MM for water-drive gas reservoirs. Laser MM is easily fabricated with low price but without uniform geometry of pores and throats. Chemical MM is difficult to fabricate with using hazardous acid (sulfuric) and needs trial and error to reach the desired pattern. In addition, both methods produce high porosity and permeability that are not similar to actual reservoir media. In this work, from a novel viewpoint, more actual MM is designed.

EOR methods are categorized into two main groups: water and gas base injection methods. Reservoir conditions dictate the suitable method based on the effectiveness of the injecting fluid and economic aspect. Possibility of asphaltene precipitation and severe fingering in fractured reservoirs are the main disadvantages of gas injection methods. Among various water base injection methods, polymer flooding is the most widely used method, which is relatively simple and has a long history of successful field applications [5].

Most of the complex oil fields face harsh conditions, such as high temperature and salinity, that destroy common polymers. There are two ways to dominate this dilemma; one way is synthesizing novel polymers. Recently, Shaban et al. [6], Tamsilian et al. [7], and Tamsilian and Ramazani [8] invented new sensitive polymers to salinity and temperature that overcame the reservoir harsh conditions. The other way is modification of polymers with NPs additives to avoid time consumption or high cost of new polymer synthesizing. Zhu et al. [9] reported application of NPs to increase the optical, thermal, magnetic, and electric properties of polymers. Attempts to find additives for EOR purposes have been made by some researchers. Hendraningrat and Torsaeter [10] used polyvinylpyrrolidone (PVP) as stabilizer to disperse and stabilize the NPs and decrease the existence of aggregates. Maghzi et al. [11,12] investigated dispersed silica NPs into polyacrylamide (PAM) in two types of laser and chemical MMs. Zhu et al. [9] used core flooding test and checked hybridization of silica NPs and Hydrophobically Associating Hydrolyzed Polyacrylamide (HAPAM) in high-temperature and high-salinity reservoirs. In addition, Yousefvand and Jafari [13] showed that by adsorbing of SiO<sub>2</sub> NPs on pore walls, the wettability of MM changes to more Water-Wet (WW), so HPAM results in increasing RF.

In this study, we examine the fumed silica NPs with Sulfonated Polyacrylamide (SPAM) with viscos-

ity, CA, and IFT tests. After that, SiO<sub>2</sub> NPs-copolymer hybrid is flooded in a novel MM to find the amount of remaining oil in-place.

The appropriate criterion to have a sense of the remaining oil after EOR methods is capillary number ( $N_{Ca}$ ).  $N_{Ca}$  is the relation between viscous and capillary forces [14]. Hence, by controlling these forces, the maximum and optimized recovery with minimum residual oil could be reached. Measurement of viscosity, IFT, emulsion formation of oil-injected fluids, and CA represents the  $N_{Ca}$  and, subsequently, residual oils changes, qualitatively.

Therefore, in this research, we first describe a suitable method to introduce sulfonated copolymer into silica NPs suspension to form a hybrid in aqueous solution. Reported here are the viscosity, IFT, and CA behaviour of these substances at different temperatures and concentrations. Afterwards, the fabrication method of the novel MM will be illustrated. Then, the materials that are injected into the MM in three scenarios of saline water, copolymer, and hybrid are presented. Finally, we discuss the results and outline the conclusions.

## 2. Materials

This section discusses the used materials in this research. First, the applied fluids will be illustrated. After that, construction method and structure of the novel MM applied in this research will be presented.

### 2.1. Fluids

The fluids which are used in this study include silica NPs, sulfonated copolymer, hybrid silica NPs-copolymer, crude oil, and brine solution.

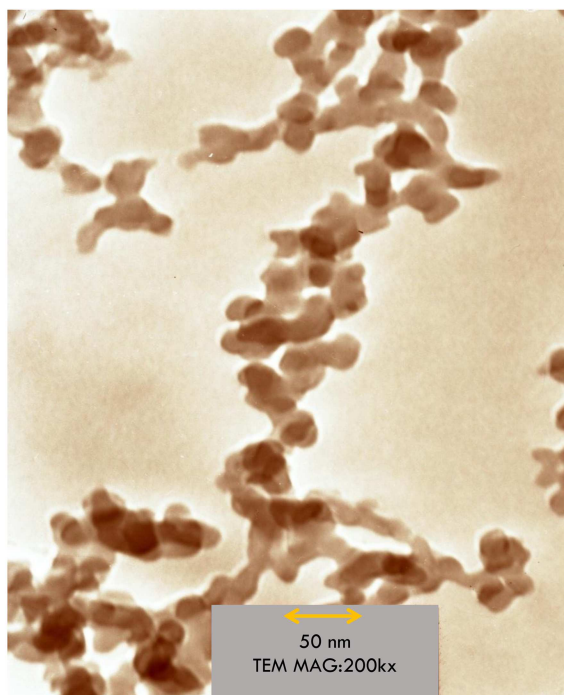
#### 2.1.1. Silica NPs

Hydrophilic silica NPs with average single particle size of 12 nm are used. The NPs are provided from Evonik Industry and consist of silicon dioxide (SiO<sub>2</sub>)  $\geq 99.8\%$ , aluminum oxide (Al<sub>2</sub>O<sub>3</sub>)  $\geq 0.05\%$ , titanium dioxide (TiO<sub>2</sub>)  $\leq 0.03\%$ , hydrogen chloride (HCl)  $\leq 0.025\%$ , and ferric oxide (Fe<sub>2</sub>O<sub>3</sub>)  $\leq 0.003\%$ . The more detailed information is provided in Table 1.

The hydrophilic silica NPs have been characterized under Transmission Electron Microscopy (TEM) that demonstrates the primary particle size of 12 nm (Figure 1). It must be mentioned that the synthesized production of the fumed silica NPs is chain-like and the only type of silica NPs with primary particle size is colloidal silica.

#### 2.1.2. Sulfonated copolymer

The sulfonated copolymer is provided from SNF FLO-ERGER. Table 2 represents some of the technical properties of the used sulfonated copolymer in this study.



**Figure 1.** Fumed silica NPs that are characterized under TEM with magnification of 200 Kx.

**Table 1.** Silica NPs characteristics data.

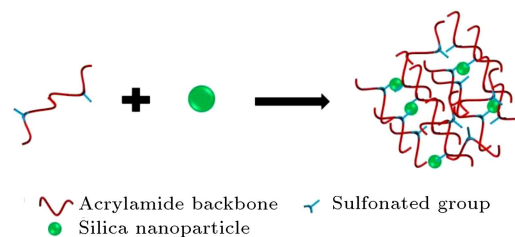
Fumed Silica NPs		
Properties	Unit	Typical value
Average particle size	Nm	12
Specific surface area (BET)	m <sup>2</sup> /g	200
pH	–	3.7–4.7
SiO <sub>2</sub>	Wt.%	≥ 99.8
Bulk density	g/cc	0.05
Appearance	–	White powder
Behavior in the presence of water	–	Hydrophilic

**Table 2.** Characteristics of the copolymer.

Polymer	Description	Molecular	
		weight (Dalton)	degree
AN132	acrylamide/AMPS based copolymer	$8 \times 10^6$	32

### 2.1.3. Hybrid silica NPs-copolymer

Mixing of polymer with silica NPs can generate “composite” or “hybrid” compounds based on the polymer type. Common polymer can result in composite mixture only, while copolymers with reactive side chain can lead to a hybrid of organic (polymer)/inorganic (NPs) mixture. In the case of a composite mixing, the final



**Figure 2.** Hybridization of sulfonated copolymer with silica NPs.

product has the segregated advantages of organic and inorganic materials, but hybrid mixtures can exhibit new properties not necessarily found in the individual components [15].

In addition to polymerization of the monomer in a suspension of inorganic particles (silica NPs), hybridization can also be obtained by swelling and gentle mixing of copolymer with side chain into silica NPs suspension [9]. In this way, we compare two different types of mixing of sulfonated copolymer and silica NPs to find out which one is more effective and results in a more powerful network to bear harsh conditions like high temperature. Two samples with the same concentration of 200 ppm sulfonated copolymer and 200 ppm SiO<sub>2</sub> NPs are prepared. In the first sample, powdery polymer is added to nanosuspension and in the second one, solved polymer is added to the nanosuspension. For preparation of nanosuspension with various salt concentrations, silica NPs are dispersed in brine solution by an ultrasonic probe (amplitude of 60% and cycle of 0.5) for 15 minutes. In the case of powdery polymer, brine nanosuspension is poured into the dishes containing polymer samples. After a day, the polymer chain swells and constructs a network with silica NPs (Figure 2). To have a homogenous hybrid, nano silica and polymer are gently stirred for one day more.

Based on the final products, viscosity of the hybrid that is provided by powdery polymer is more than that of the solved polymer. The result shows that the kinematic viscosity of the sample prepared by powdery polymer is 3.03 CentiStock while the viscosity of the sample containing solved polymer is 2.76 CentiStock.

### 2.1.4. Crude oil

Crude oil from one of the Iranian southwest oil fields is used for all tests. Properties of the crude oil are presented in Table 3.

### 2.1.5. Brine solution

Brine solution is prepared synthetically by dissolving equivalent salinity (NaCl) in deionized water from the Persian Gulf (50,000 ppm) and connate water (200,000 ppm) salinity. Other salinities between the aforementioned components are made to trace the results.

**Table 3.** Crude oil properties.

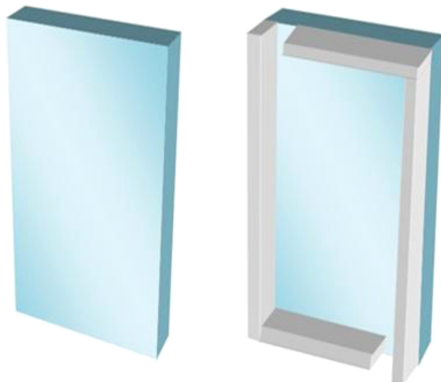
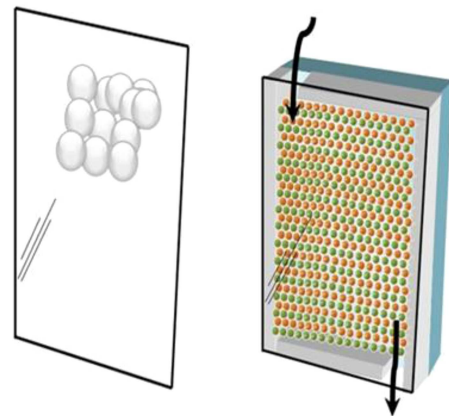
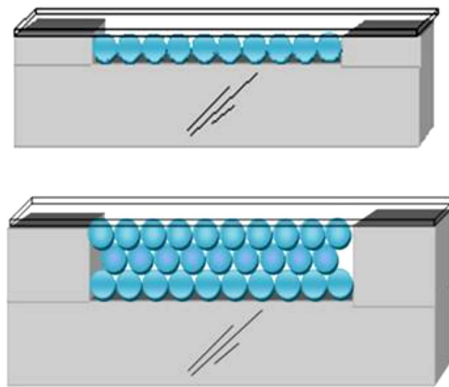
Properties at standard condition (15°C and 1 atm)	Unit	Value
Methane	Mol.%	0
Ethane	Mol.%	0.14
Propane	Mol.%	1.54
Iso-butane	Mol.%	1.03
Normal-butane	Mol.%	3.66
Iso pentane	Mol.%	2.66
Normal pentane	Mol.%	3.6
Hexanes	Mol.%	6.38
Heptanes & higher	Mol.%	80.99
H <sub>2</sub> S	Wt.%	0.5404
Density of stock tank oil	g/cm <sup>3</sup>	0.8451
°API	—	35.9

## 2.2. Micro-model

To fabricate new MM, glass beads or any reservoir rock minerals as porous media grains are used. First, a flat glass with 4 mm thickness is selected for the bottom of the MM. Then, four films of glass (with arbitrary thickness) are glued on the edge of the flat glass to create a container to set glass beads and arrange an inlet and outlet to inject and produce fluids (Figure 3). After that, another flat glass with 2 mm thickness is placed on the glass films to create a closed porous medium as it is shown in Figure 4. Next, the MM is heated at about 700°C in a furnace for 30 min (retention time) to fuse and give a closed space for flooding.

Some advantages of the novel MM in comparison with the previous ones are as follows:

- Capability of utilizing reservoir grains to fabricate porous media;
- Easy fabrication procedure (no need for a laser machine or any chemical substances);

**Figure 3.** Flat glass and four films for MM fabrication.**Figure 4.** Glass beads and 3D MM.**Figure 5.** 2D and 3D MM configurations.

- Capability of creation of 3D MMs, as it is shown in Figure 5, like 2D ones;
- Fabricating porous media with desired porosity and permeability;
- Easy cleaning with no fine migration;
- Ability to create a flat core flooding to visualize and study flow mechanisms;
- Capability of heterogeneous reservoir construction with two different grains

The aforementioned novel MM also requires new holder design for fluid injection under high-pressure conditions. In new designed holders for novel MM set-up, the restrictions of previous ones are omitted. In addition, many advantages including flexible design; no limit for MM thickness, length, and width; and ability to use several ports in different locations as inlets or outlets are added (Figure 6).

Characteristics of the 3D MM used in this study for various flooding are tabulated in Table 4.

## 3. Results and discussion

In this section, first, the inherent viscosity of hybrid silica NPs and copolymer in comparison with copoly-

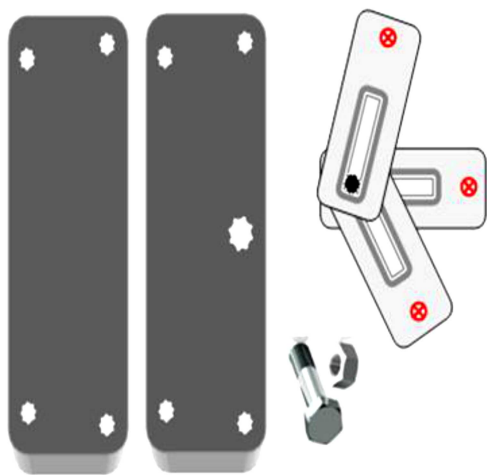


Figure 6. MM's holder and accessories.

Table 4. 3D MM characteristics.

Variable	Value	Unit
Porosity	45.44	%
Permeability	2	Darcy
Length	5	cm
Width	5	cm
Thickness	0.1	cm
Grains	250	Micron
Grain geometry	Spherical	—
Pattern	One quarter of 5 spot	—
Wettability	WW	—

Table 5. Inherent viscosity at 35 and 60°C.

Samples	Inherent viscosity (dL/g)	
	35°C	60°C
DI Water	1	1
Silica NPs	1	1
Sulfonated copolymer	76.9	58.66
Hybrid	102.33	100.96

mer alone is discussed for two different temperatures. Then, IFT of silica NPs in various brine salinity is investigated. After that, CA of this hybrid with oil is tested in the maximum salinity. Finally, the results of MM flooding in three scenarios of saline water, copolymer, and hybrid injection are illustrated.

### 3.1. Inherent viscosity measurement

The inherent viscosity of copolymer and silica NPs, and their hybrids is measured by an Ubbelohde capillary viscometer. Inherent viscosity is evaluated as the intercept on the ordinate axis of the viscosity plot versus concentration ( $C$ ) as  $C$  trends to zero. The results at 35°C and 60°C are summarized in Table 5.

In Figures 7 to 10, vertical axis is inherent viscosity and horizontal axis is four concentrations (100, 200, 300, and 400 ppm) of copolymer and silica NPs. For hybrid solutions, 500 ppm of silica NPs is added to these concentrations.

Inherent viscosity of hybrid solutions in Figure 7 increases with the addition of 500 ppm silica NPs to copolymer concentrations (100, 200, 300, and 400 ppm) and silica NPs-copolymer hybrids exhibit better inherent viscosity. It is found that the inherent viscosity increases from 76.9 dL/g to 102.33 dL/g at 35°C. Also, by increasing the silica NPs concentration, there is no viscosity improvement and SiO<sub>2</sub> NPs play no role in viscosity treatment without copolymer.

Figure 8 shows the inherent viscosities of copolymer and its hybrids with silica NPs at 60°C. It is found that in higher temperatures, the hybrid is stable; however, copolymer shows a significant decrease in viscosity from 102.33 dL/g to 100.96 dL/g.

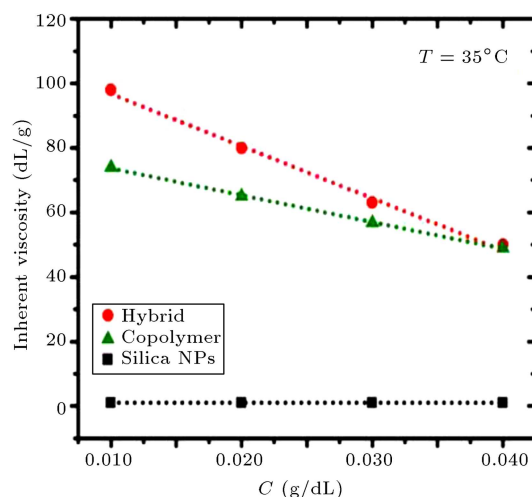


Figure 7. Inherent viscosity of SiO<sub>2</sub> NPs, copolymer, and hybrid at 35°C.

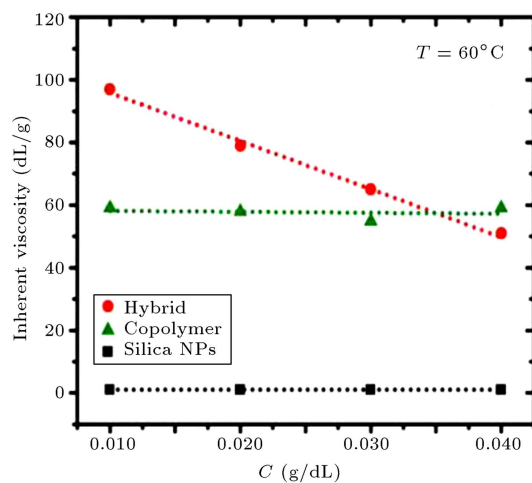


Figure 8. Inherent viscosity of SiO<sub>2</sub> NPs, copolymer, and hybrid at 60°C.

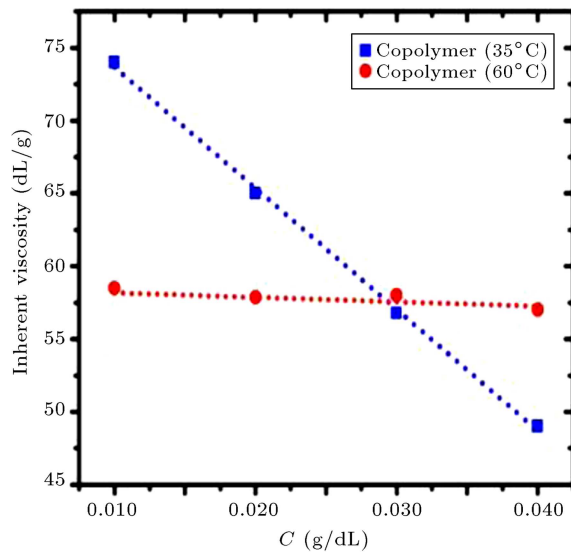


Figure 9. Inherent viscosity of copolymer at 35 and 60°C.

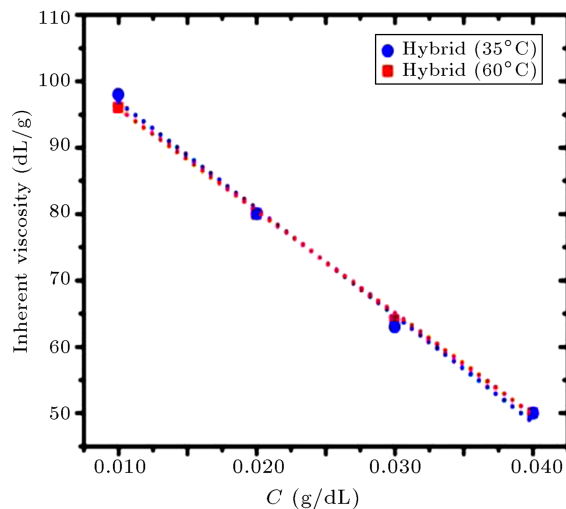


Figure 10. Inherent viscosity of hybrid at 35 and 60°C.

Figures 9 and 10 compare the thermal stability of silica NPs-copolymer hybrid with that of copolymer. As illustrated in these figures, hybrid of copolymer and silica has a good thermal stability due to formation of network between silica NPs and copolymer. Adding SiO<sub>2</sub> NPs to copolymer reinforces the viscosity so that the inherent viscosities are almost similar to each other for hybrids at two different temperatures of 35°C and 60°C.

Hence, adding NPs to copolymer not only results in thermal stability, but also causes viscosity improvement, thus higher  $N_{Ca}$ , leading to more RF and decreasing residual oil saturation.

### 3.2. IFT measurement

Torsæter and Abtahi [16] reported that pendant drop is a widely used and accurate method to determine the IFT between two liquids. The method was intended for

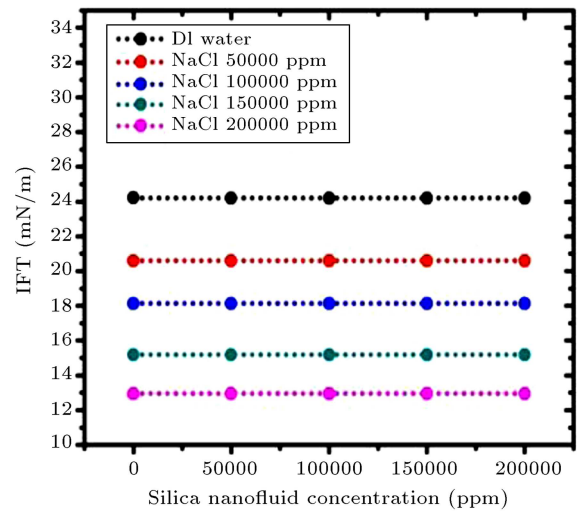


Figure 11. IFT between crude oil and silica nanosuspension at different salinities.

application to liquid pairs with not too low or too high IFT. Small drops tend to be spherical because surface forces depend on area. Principally, one can determine the IFT from measurements of the drop shape.

The IFT of crude oil in a reservoir is a complex function of a wide variety of unknown factors, including temperature, pressure, salinity, amount, and type of hetero-atoms present; acid number, base number, and pH of the aqueous phase; and viscosity, amount of asphaltenes, and amount of dissolved gases [17].

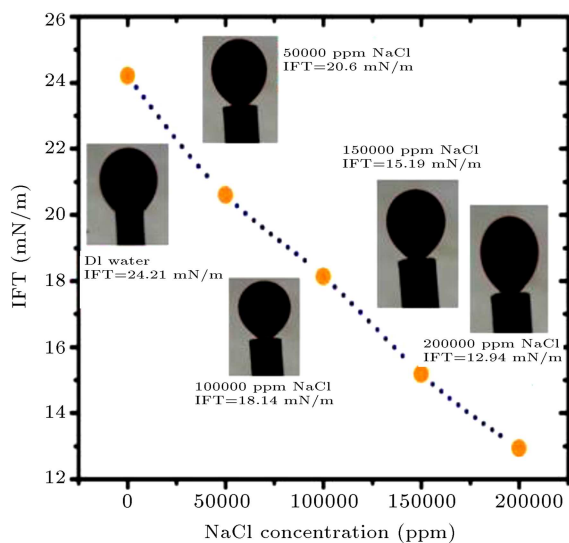
Kumar [18] performed a general study of the effect of salinity on IFT; he showed that the data were sparse with antithetical trends and an increase or decrease in IFT with salt addition might occur. We measure IFT at room condition between crude oil and silica NPs with different concentrations in Persian Gulf and connate water equivalent salinity and other salinities among them.

As shown in Figures 11 and 12, IFT between oil and SiO<sub>2</sub> NPs is constant for all silica NPs concentrations. However, inversely, IFT decreases from 24.21 mN/m to 12.94 mN/m with increase in salinity. It must be mentioned that silica NPs have no effects on IFT reduction in static IFT test and the only factor to create lower IFT is brine water for this special oil. Therefore, the high salinity of Persian Gulf or reservoir connate water is an appropriate candidate to apply for injection fluid.

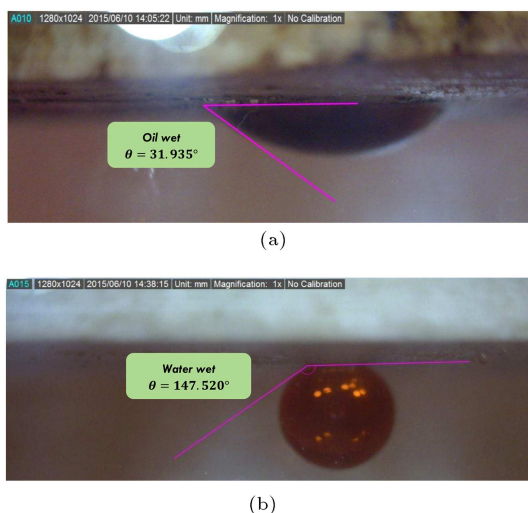
Therefore, in static IFT test (without flooding), it is shown that saline water can decrease IFT and increase  $N_{Ca}$ , so more oil recovery is obtained.

### 3.3. CA measurement

Torsæter and Abtahi [16] described wettability of a reservoir rock-fluid system as the ability of one fluid to spread on the surface of the rock in the presence of another fluid. Wettability plays an important role



**Figure 12.** IFT between crude oil and different saline waters.



**Figure 13.** CA of reservoir rock slice: (a) Between crude oil and saline water, and (b) between crude oil and hybrid.

in oil production. The degree of wetting of solid by liquids is usually measured by the CA that a liquid-liquid interface makes with a solid.

We use a thin section of the studied reservoir rock that is sliced from a reservoir core sample by a cutter machine. To measure the CA of oil drop against injection fluid as bulk phase on rock slice at ambient condition, the oil drop volume in range of 10-12  $\mu\text{L}$  is injected to the reservoir rock slice. CA is measured two times, before and after reservoir rock treatment with hybrid.

Figure 13(a) illustrates that the oil drop spreads on the thin section that expresses oil wetness with angle of 31.935 degrees in the presence of connate saline water.

To investigate the wettability alteration after hybrid injection, the reservoir rock slice is soaked in

the hybrid of NP-copolymer in connate water salinity for 30 minutes. It is observed that silica NPs sit on the rock slice and the color of slice converts from darkness to blankness. This phenomena occur because salinity destroy the hybridization network between NPs and copolymer. Hence, NPs are released in the saline aqueous that result in NPs aggregating.

Afterwards, the CA for the slice with covered silica NPs is measured. The wettability of the reservoir rock is altered from OW to WW (147.52 degrees) as it is shown in Figure 13(b).

Wettability alteration of the reservoir rock from OW to WW can result in higher RF and lower residual oil saturation.

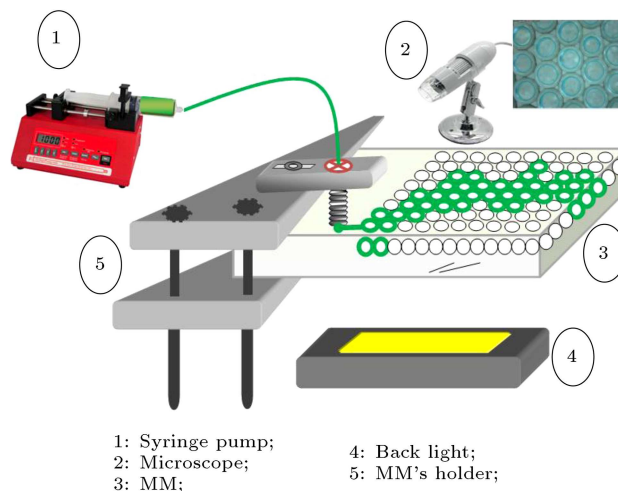
### 3.4. MM flooding

First, 3D MM should be saturated by 100% oil. Then, pore volume of 3D MM is computed ( $PV = 1.136 \text{ cc}$ ) by measuring the weight difference between saturated and unsaturated MMs and known oil density ( $0.8451 \text{ gr/cm}^3$ ). Finally, the injection rate is calculated to be 0.5 cc/hr by considering average reservoir fluid velocity equal to 1 ft/day. As a result, the injection time for one pore volume injection is obtained to be 147 min (2.27 hrs).

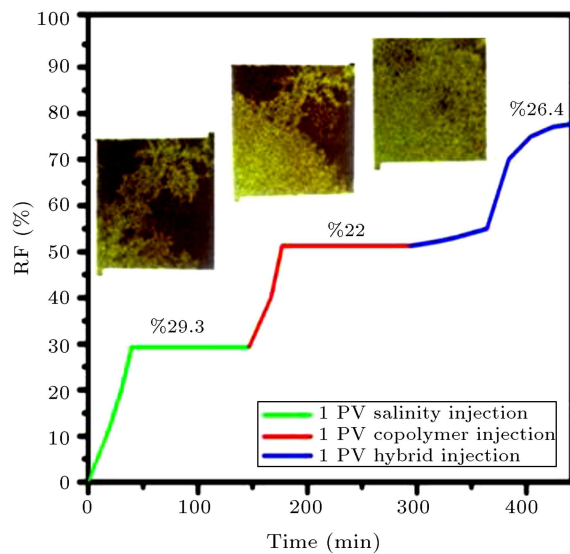
Figure 14 shows the MM flooding set-up and requirements that consist of a syringe pump, microscope, MM, backlight, and MM's holder.

In this research, one PV of three injecting fluids (as described below) is injected into the MM defined in the previous section:

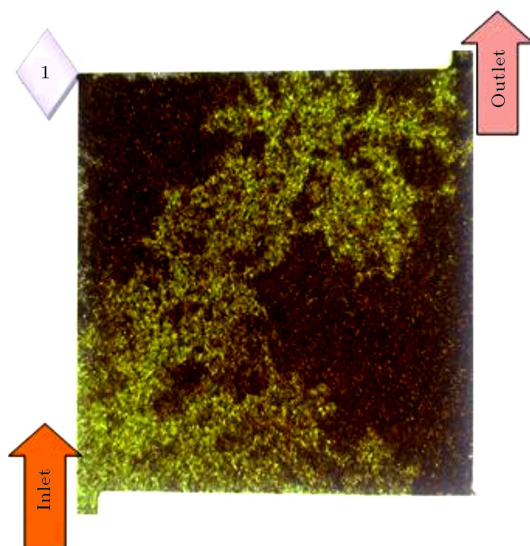
- First, saline water with 50,000 ppm NaCl is injected;
- Next, sulfonated copolymer with 1000 ppm is injected;
- Finally, hybrid of silica NPs (2000 ppm) and sulfonated copolymer (1000 ppm) is injected.



**Figure 14.** MM flooding set-up.



**Figure 15.** RF for saline water, copolymer, and hybrid injection scenarios.

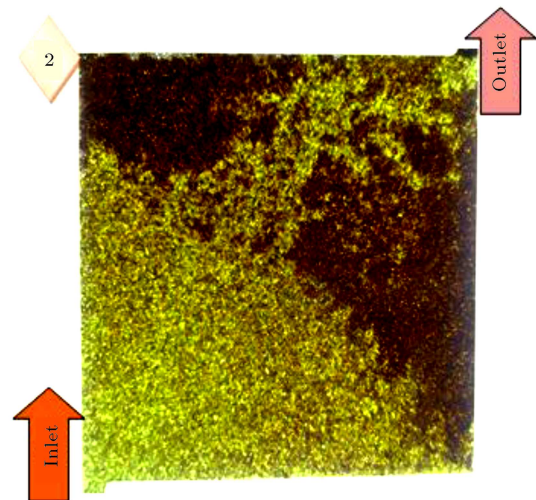


**Figure 16.** Saline water injection into 3D MM.

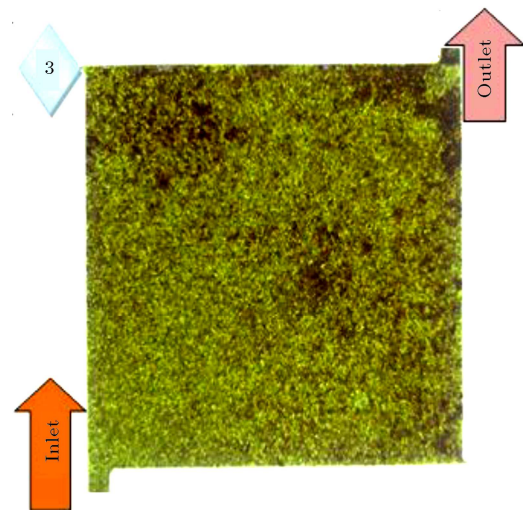
It must be noticed that copolymer and hybrid silica NPs-copolymer solution is stable during flooding and four days after flooding.

RF for saline water, sulfonated copolymer, and hybrid of silica NPs-copolymer injection are calculated to be 29.3%, 22%, and 26.4%, respectively. Total RF after all fluid injections is obtained to be 77.7% as it is shown in Figure 15.

In the first scenario, namely, saline water injection, severe fingering is observed because of high mobility ratio (Figure 16). In the second scenario, namely, polymer flooding, higher viscosity results in better mobility control and higher  $N_{Ca}$  and RF than that in saline water injection scenario (Figure 17). While, in hybrid of silica NPs and sulfonated copolymer, i.e. the third scenario, not only viscosity enhancement, but



**Figure 17.** Copolymer flooding injection into 3D MM.



**Figure 18.** Silica NPs-copolymer injection into 3D MM.

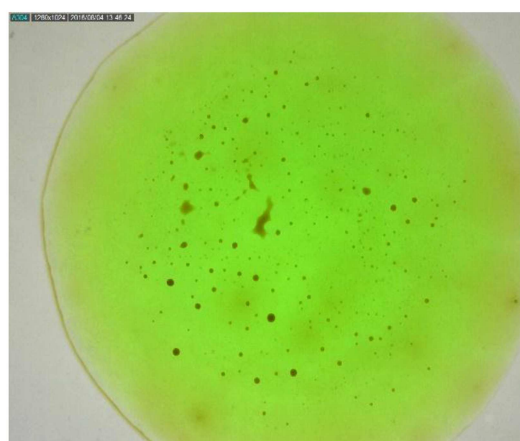
also IFT reduction and emulsion formation affect  $N_{Ca}$  and RF [19]. Polymers lead NPs to virgin zones such as the trapped area or the edge of the displacing and displaced fluids to exert the effectiveness of silica NPs (Figure 18).

After hybrid injection, produced fluids are observed under the microscope. The observation shows O/W emulsion drops formation and, then, less residual oil saturation than that in polymer flooding (Figure 19).

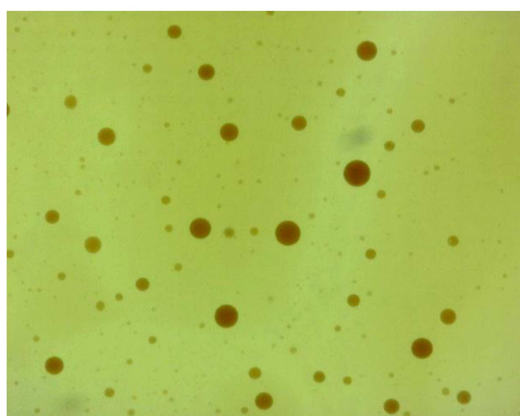
#### 4. Conclusions

In this study, the capability of hybrid silica NPs complexes with sulfonated copolymer for decreasing residual oil saturation is examined. To achieve this purpose, viscosity, IFT, emulsion formation, CA tests, and MM flooding are done. Based on the observed results, silica NPs-copolymer hybrid is a promising





(a)



(b)

**Figure 19.** O/W emulsion formation of production fluid by hybrid flooding: (a) 50× and (b) 200× magnifications.

candidate for performing EOR process, especially in fractured reservoirs. The main results are summarized as follows:

- The preparation method of silica NPs-copolymer hybrid has a significant effect on the viscosity. The best approach for hybrid preparation is adding powdery copolymer to silica nanosuspension.
- Hybridization of SiO<sub>2</sub> NPs-copolymer not only increases injection fluid viscosity in lower polymer concentrations, but also leads to thermal and salinity stability of the copolymer.
- NPs have a great potential to apply wettability alteration and exert a positive influence on low and very low permeability. However, it should be noted that NPs suspensions must be stable enough to perform their real impact. Using hybrid of polymers and NPs enhances the NPs stability.
- Hybrid injection in MM test results in high RF in comparison with water and copolymer injection. After hybrid breakthrough, oil and hybrid are produced simultaneously the copolymer helps

SiO<sub>2</sub> NPs to penetrate into the virgin zone and increase microscopic efficiency so remained oils are decreased. In addition, O/W emulsion formation in MM flooding is observed due to decrease in IFT.

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### Nomenclature

N <sub>Ca</sub>	Capillary Number
IFT	Interfacial Tension
CA	Contact Angle
RF	Recovery Factor
EOR	Enhanced Oil Recovery
MM	Micro-Model
OW	Oil Wet
WW	Water Wet
NPs	Nanoparticles
O/W	Oil in Water emulsion

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