

Research Article

INVESTIGATION OF THE INJECTION OF ALUMINUM OXIDE AND SILICA HYDROGELS ON OIL RECOVERY FROM HIGH-SALINITY HYDROCARBON RESERVOIRS

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ABSTRACT

Nowadays, laboratory studies on the use of nanoparticles and nanofluids to enhance oil production from oil reservoirs are among the hottest topics. Nanoparticles can reduce the water–oil interfacial tension and alter the wettability of reservoir rock. However, one challenge in using nanoparticles is their stability before and after injection. Nanoparticle suspensions alone are unstable, and within a few hours, all nanoparticles precipitate from the aqueous phase. In this paper, the effect of injecting aluminum oxide and silica hydrogels on oil recovery from high-salinity hydrocarbon reservoirs in glass-bead media, with and without xanthan gum as a stabilizer, at laboratory temperature (25 °C), has been investigated. The aforementioned nanoparticles were first tested individually without the presence of a stabilizer, and then the experiments were repeated in the presence of xanthan gum. The results showed that the best oil production results without the presence of xanthan gum were, respectively, related to silica (61.26), aluminum oxide (60.87), titanium dioxide (59.98), and copper oxide (57.76), while in the presence of xanthan gum they were, respectively, related to silica (74.00), aluminum oxide (68.86), copper oxide (65.69), and titanium dioxide (61.89).

INTRODUCTION

Water flooding, one of the oldest and most widely used enhanced oil recovery techniques, has long attracted the attention of petroleum engineers for producing from hydrocarbon reservoirs due to the easy availability of water in nature and its lower environmental impact. However, the lower viscosity of water compared to oil prevents the oil phase from being completely displaced by the water phase, leaving a considerable portion of oil in the reservoirs (Binshan et al., 2012). Therefore, the capillary number in the water injection process is significantly low, and for this reason, this method faces certain limitations. In recent decades, solutions have been proposed to improve the flooding mechanism. Among these solutions are the use of chemical agents such as surfactants, which release trapped oil by reducing interfacial tension and thus increase the capillary number and, in other words, improve microscopic sweep efficiency; polymers, which increase the viscosity of water and reduce the mobility ratio of injected water relative to oil, thereby improving macroscopic efficiency; and alkalis, which improve viscosity, interfacial tension, and wettability. Adding these chemicals to the water phase increases the capillary number, thereby increasing oil production from reservoirs. However, due to the high cost of chemical preparation, their degradation under reservoir salinity conditions, and their strong adsorption to reservoir rock surfaces, the use of chemical materials has encountered operational and economic limitations.

The main advantage of using nanoscale materials is their high surface area-to-volume ratio, which alters the physical properties and performance of materials. Silica (SiO_2), due to its abundance in nature and low cost, has been the most widely used nanoparticle in laboratory-enhanced oil recovery studies and has played a significant role in reducing interfacial tension and altering wettability (McElfresh et al., 201; Hendraningrat et al., 2013). Recently, with further advances in nanotechnology, researchers have also investigated the use of some metallic nanoparticles and their capability as enhanced oil recovery agents. For example, Soleimani et al. (2013) used aluminum oxide, titanium dioxide, and silica nanoparticles. Their experimental results showed that the mentioned nanoparticles significantly increased oil production by reducing the interfacial tension between oil and the aqueous phase and altering reservoir wettability toward water-wet conditions. They also noted that these metal oxide nanoparticles reduce the viscosity of the produced oil. Some other researchers have also used titanium dioxide and aluminum oxide nanoparticles for enhanced oil recovery in sandstone environments and have cited similar reasons for increased oil production (Ehtesabi et al., 2014; Zhang et al., 2014).

Mahmoudian (2013) conducted an experimental study of water and polymer injection in fractured porous media using micromodels. In this study, comparative experiments were performed to investigate the effect of water and polymer injection on oil recovery to reduce polymer mobility relative to water; the effect of polymer concentration in horizontal and inclined conditions; the effect of injection rate on oil recovery; the effect of fracture orientation on oil recovery in horizontal and 45-degree conditions; the effect of fracture configurations, one being horizontal and the other half its length; and the effect of fracture distribution. The following results were obtained: injection of a polymer solution at any concentration leads to higher oil recovery than water injection, and this recovery increases with increasing polymer concentration. With increasing injection rate, oil recovery decreases, which may be due to

reduced time for better flooding patterns. The geometric characteristics of fractures are important factors in determining oil recovery in fractured media. As the angle between the fracture and the injection point increases, oil recovery increases. In media with longer fractures, oil recovery is lower during polymer injection. Oil recovery in fractured media with more distributed fractures is greater than in media with less fracture distribution. Wu et al. (2013) showed that silica, aluminum oxide, and titanium dioxide improve oil production from a limestone sample at different temperatures. They stated that aluminum oxide has the lowest adsorption and is the best nanofluid in all temperatures within the porous limestone medium. Wang et al. (2013) conducted a study entitled “Enhanced recovery using nanoparticles: fundamental mechanisms and adsorption experiments.” After investigation, they concluded that this nanofluid displaced 50% of the crude oil, whereas a saline solution displaced only 17%. As a result, the IIT nanofluid is a suitable option for enhanced oil recovery from high-salinity reservoirs. Finally, they performed crude oil displacement in glass capillaries to observe the process at solid surfaces. Ogolo et al. (2012) performed several core flooding experiments using silica nanofluids (LHP) as well as aluminum oxide, titanium dioxide, hydrochloric acid, and iron oxide on water-wet sandstone samples with both high and low permeability. After analysis, they concluded that silica (LHP) reduces interfacial tension and also alters wettability. Based on mineralogical analysis, the core plug may contain illite, a clay mineral. Therefore, the interaction between salt and nanofluids was not the main cause of the reduction in permeability and porosity in this study.

Hashemi et al. (2013) investigated the effect of HSE on the transport and displacement of nanoparticles. In the first stage, brine was injected into the core at a constant rate of 1.25 mL/min. Then, in the second stage, enhanced oil recovery was performed using sequential smart nanoparticles through conventional flooding at a rate of 1.25 mL/min. The results showed that copper oxide produced better electrokinetic-nanoflooding results due to its higher density and thermal conductivity than nickel oxide. Also, due to nickel oxide's lower electrical conductivity, better results were obtained with sequential electrokinetic-nanoflooding.

In this paper, the effect of four nanoparticles—aluminum oxide (Al_2O_3), titanium dioxide (TiO_2), copper oxide (CuO), and silica (SiO_2)—on enhanced oil recovery from high-salinity sandstone reservoirs has been investigated. These nanoparticles were first used individually without a stabilizer, and then xanthan gum was used as a stabilizer alongside the nanoparticles.

METHOD

In this study, four types of nanoparticles were used, namely silica (with a size of 20 nm, purity of 99.5%, and specific surface area of 160 m^2/g), titanium dioxide (with a size range of 10–30 nm, purity of 99.5%, and specific surface area of 5–100 m^2/g), aluminum oxide (with a size of 41 nm, purity of 99%, and specific surface area of 60 m^2/g), and copper oxide. The nanoparticle oxides were supplied by Skyspring Nanomaterials Inc. (Houston, TX, USA).

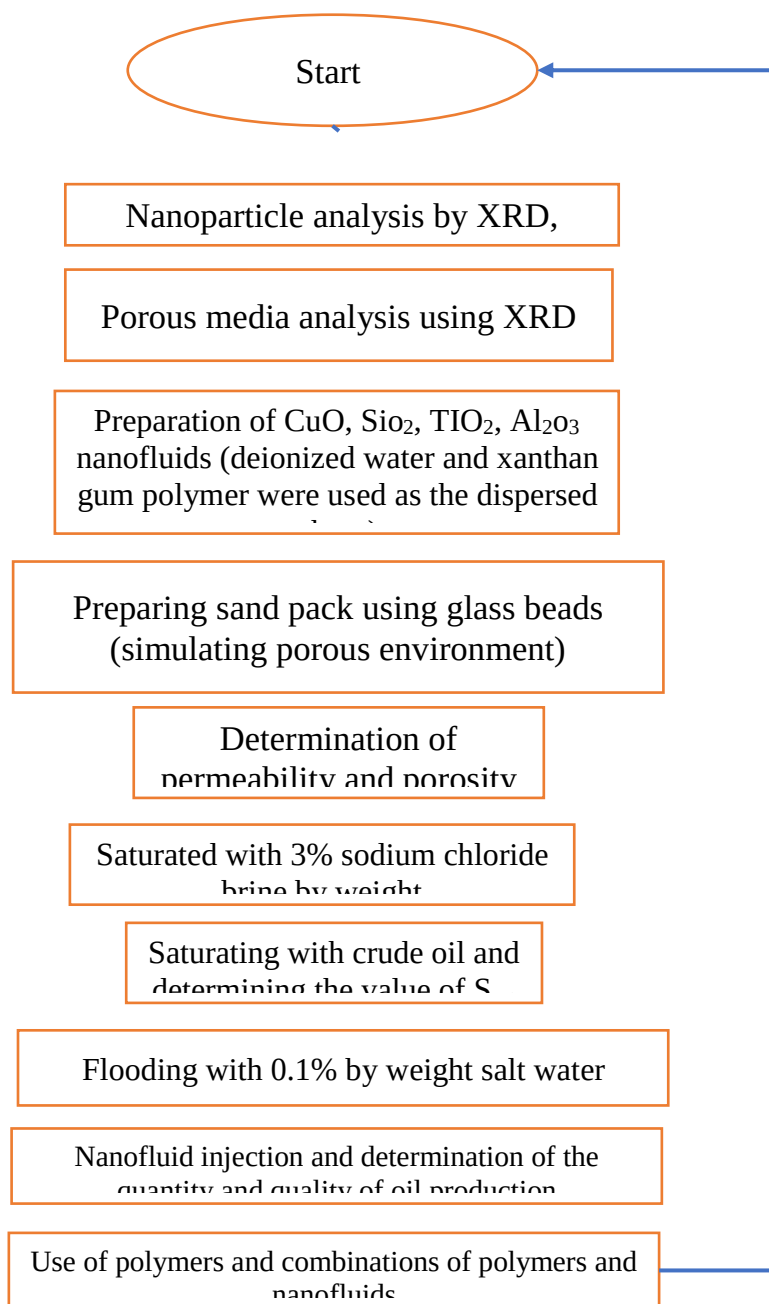


Figure 1: Description of the work procedure stages

The most important commercial polymers used are polyacrylamide biopolymers; however, in this study, xanthan polymer was used. Xanthan is a heteropolysaccharide with a very high molecular weight, ranging from 2 to 5 million Daltons, and consists of repeating units. It is produced through bacterial fermentation processes under ambient pressure from *Xanthomonas*, purified by ethanol or propanol recovery, then dried and milled. Its secondary structure depends on the arrangement of molecules, which may be ordered in a row or randomly distributed.

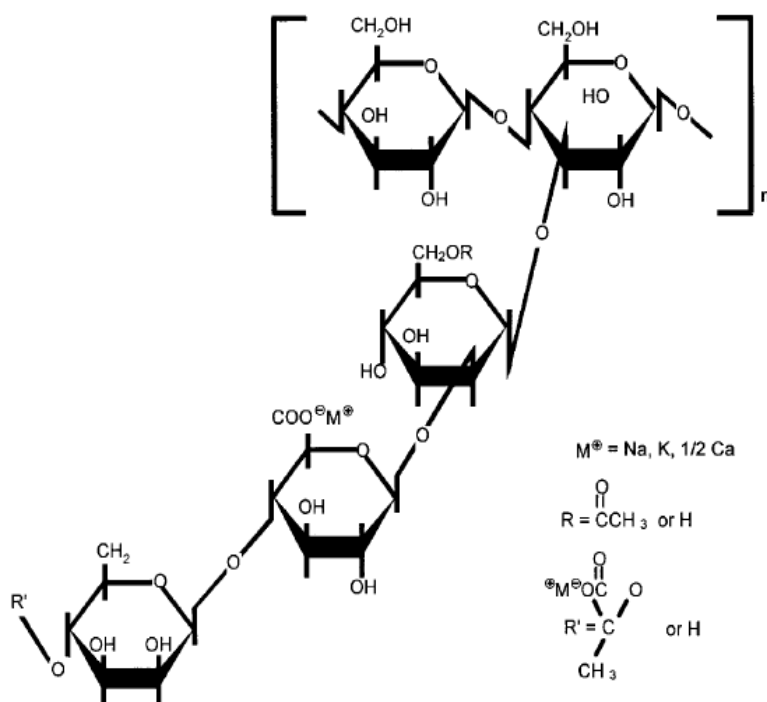


Figure 2: Structure of xanthan

Light scattering, hydrodynamic measurements, thermodynamic properties, and calorimetric measurements can determine the physical and chemical properties of xanthan in aqueous solutions. By linking this solution with crosslinking agents and nano-agents, better properties can be obtained when this polymer is placed in solution. Its amide groups are converted to carboxylate groups, and each group dissolves a negative charge. In this case, the xanthan polymer is hydrolyzed, making it ready for crosslinking.

Sodium chloride salt was used to prepare the designed solution and also to make the formation water sample of the oil reservoir. In this study, the glass beads sample, or in other words, glass beads, was prepared and purchased ready-made.

The properties of the glass beads are presented in Table 1.

Table 1: Properties of glass beads

Al ₂ O ₃ (%)	Mgo (%)	Cao (%)	Na ₂ O (%)	SiO ₂ (%)
0.4	3.3	9.8	13.7	72.5
Hardness (Moh)	Thermal conductivity , (cal/(sec)(cm ²)(c/cm)at500c)	Expansion coefficient, in/in/c*10 ⁻⁷	Softening point ,c	Specific gravity
6.0	0.0036	90	704	2.5

In this study, crude oil from one of the oil fields in southern Iran was used, with an API gravity

of 28.098, indicating it is light oil. The experimental results for this crude oil sample are presented in Table 2.

Table 2: Oil properties

API gravity	Viscosity (cP)	Density (g/cm ³)
28.098	13.46	0.8666

Ultrasonic baths and high-pressure homogenizers have been powerful tools for breaking aggregation, used by researchers more than other devices, such as magnetic stirrers and high-speed devices. However, sometimes after the optimal time for this process, new problems arise due to rapid aggregation and sedimentation. In this bath, samples mixed in an aqueous medium are homogenized and uniform using hot water. The sample remains in the bath for 15 minutes so that any fine particles suspended in the water will flow through the microchannels when the suspension contacts the inner walls of the interaction chamber. Therefore, the flow velocity of the stable suspension through the microchannels increases according to Bernoulli's theorem, and at the same time, extensive cavitation occurs. In this fast-flow region, the particle cluster must be broken by a combination of mechanisms, including strong, irregular shocks within the interaction chamber wall, microbubbles formed by cavitation due to the bursting energy, and the high shear rate of the flow. This results in a suspension with less particle aggregation at high pressure.

To measure interfacial tension using the tensiometer test, the IFT device available at the Science and Research Branch of Tehran University was used. In this study, an injection pump system was used to hold saturation. To determine the viscosities of water and oil solutions and to obtain interfacial tension values in the tensiometer test, the viscosities of the water and oil samples were measured using an SVM 3000 viscometer manufactured by Anton Paar.

For washing and cleaning the glass beads, appropriate solvents are used. Washing glass beads removes grease from porous solids. In this method, a suitable solvent is selected based on the requirements and knowledge of the compounds present in the sample. Washing continues until the grease present in the particles is minimized. In this study, the solvents used for washing include water, dishwashing liquid, and industrial alcohol. After washing, the glass beads are placed in an oven at 80 °C for 12 to 18 hours to dry and become ready for subsequent studies. Four stainless steel tubes with an inner diameter of 2.2 cm and a length of 31 cm were prepared as holders for a porous medium composed of 99% pure glass beads, used as sandstone in injection scenarios. In addition, nine sections with a thickness of 1 cm from this formation were prepared to investigate wettability alteration. All samples, after preparation, were sieved using a vibrating sieve shaker (Retsch AS 200) in the 125 to 180 micron size range to obtain an average glass bead size of 150 microns. Then, to prevent the effect of possible impurities, they were washed with water and placed in an oven at 80 °C for 10 hours to completely dry. Subsequently, the beads were poured into the tubes and compacted under pressure. To ensure homogeneity of the contents inside the tube, vibration and deionized water were simultaneously injected from the top of the tube during compaction. Finally, after preparation, the system was placed in an oven at 110 °C for 12 hours. A sandpack system was used to saturate the sample and to inject oil and fluid into the holder.

Considering that the total volume is a constant value, and by having the pore volume, porosity can be calculated at this stage:

$$V = \pi r^2 \times L \quad (1)$$

$$V = 114 \text{ cm}^3$$

Using the porosity formula, it is obtained as a percentage:

$$\Phi = \frac{vp}{vb} \quad (2)$$

In addition to the static oil storage property in reservoirs represented by porosity, another important property of a reservoir is permeability. Permeability is defined as a proportionality coefficient in Darcy's law:

$$q = \frac{k A}{\mu} \times \frac{\Delta P}{L} \quad (3)$$

In this equation, q is the volumetric flow rate, A is the cross-sectional area, μ is viscosity, ΔP is the pressure drop, L is the flow length, and k is the absolute permeability.

To obtain absolute permeability, flow rates of 1, 2, and 3 mL/min were selected, yielding three corresponding pressure drops. By substituting into the above equation, three permeability values were obtained, and the absolute permeability of the core was calculated using the arithmetic mean:

$$K_{abs} = \frac{k_1 + k_2 + k_3}{3} \quad (4)$$

Usually, oil in the reservoir is accompanied by some immobile water; this water, which is equivalent to the final residual oil by definition, is called covalent or non-reducible water. S_{wc} usually represents covalent water saturation. It should be noted that the initial saturation in the reservoir is not necessarily equal to the covalent water saturation and is denoted by S_{wi} . At this stage, S_{wi} was measured by mixing 3 wt% sodium chloride and injecting water until the glass-bead sample reached a specific density and viscosity at a limited temperature in this study. Oil injection was performed at a rate of 1 mL/min to about PV2 to ensure that the sample holder was saturated with oil. The core holder was held vertically with fluid flow from the bottom of the tube. Saltwater injection at 3 wt% concentration was performed at a rate of 1 mL/min. Then the S_{wc} value was reported for each test, followed by injecting PV2 of the available oil into the void space at twice the volume of the void space at a flow rate of 1 mL/min. On the other hand, the oil and brine produced were measured with very high accuracy.

Then:

Produced oil volume (cc) – injected oil volume (PV2) = S_o

Amount of oil saturation inside the core = S_o

Produced water during injection – brine inside the core = S_{wc}

Connate water saturation = S_{wc}

After saturating the core with oil, the water flooding stage was performed. In these experiments, brine flooding at a rate of 1 mL/min was carried out to two pore volumes (PV2), with the injected brine divided into six parts; in each part, the produced oil relative to the injected water was plotted.

Nanofluids at 50 mg/L were prepared as the base phase for wettability, interfacial tension, and injection scenario studies by dissolving metal oxide nanoparticles in deionized water using appropriate equipment. To enhance nanoparticle stability, xanthan gum polymer with concentrations of 10, 20, 30, 40, 50, and 60 wt% was added to the nanofluids. This study aimed to create a suspension with desirable stability under unfavorable conditions (high salinity, temperature, high pressure, etc.). To prevent sedimentation of nanofluids, a polymeric material

was added; xanthan gum was selected among various biopolymers due to its non-toxicity and high natural production. The suspension stability was evaluated visually based on polymer concentration.

The experiments were conducted in two stages. First, each of the nanofluids of silica, aluminum oxide, titanium dioxide, and copper oxide was mixed based on a certain concentration of nanoparticles in deionized water using a rotary shaker at a speed of 220 for 1 hour, and finally, for synchronization of the suspension, it was provided in an ultrasonic bath for 15 minutes at ambient temperature. The instability of the injected nanofluids can lead to leakage, accumulation of nanoparticles in the porous medium, and blockage of bottlenecks and flow paths. As a result, the second stage of the experiments involved combining the xanthan gum polymer at a concentration of 10 mg% by weight with each of the mentioned nanofluids using a rotary shaker at 220 rpm for 1 hour, followed by synchronization in an ultrasonic device for 15 minutes. Finally, the results of both stages of the experiments were examined. Also, to simulate formation water, deionized water and a 3% sodium chloride solution by weight were provided. Table 3 shows the properties of salt water and nanofluids at ambient temperature (26).

Table 3: Nanofluid properties

Fluid	Density	Viscosity	pH
Aluminum oxide	0.9831	0.90757	7.03
Silica	0.9844	1.0723	8.37
Titanium dioxide	0.9887	1.0236	6.32
Copper oxide	0.9890	1.1601	8.32
Xanthan	0.9897	1.4366	8.41
Xanthan + aluminum oxide	0.9779	1.3885	7.81
Xanthan + silica	0.9861	1.5583	8.05
Xanthan + titanium dioxide	0.9976	1.1797	7.55
Xanthan + copper oxide	0.9974	1.4081	7.85

During flooding, the injection processes were carried out at 26 °C. First, distilled water was used to measure porosity and permeability in accordance with international standards. To establish initial saturation conditions in the pipes to simulate the equilibrium state that naturally exists in the oil reservoir, salt water was injected at a flow rate of 0.2 ml/min until the pipes were saturated. Then, crude oil equivalent to the same flow rate was injected into them at twice the volume of the void space, until no more water was produced from the cores; in other words,

the water saturation in the cores reached the initial immobile state. The flooding scenario used to fill the pipes first injected 3% saltwater into each pipe to simulate formation water, at a rate twice the volume of the void space, to simulate the primary and secondary oil recovery processes. After that, the nanofluid was injected at a concentration of 50 mg/lit. This scenario was carried out for each of the pipes, which have the same characteristics. This injection rate was 0.2 ml/min in all injection processes. During fluid injection, the accumulation of saltwater, nanofluid, and produced oil was measured in the graduated cylinder. In this way, the amount of oil recovery from water and nanofluid injection can be calculated. Also, by measuring the pressure difference between the two ends of the pipe, the pressure drop trend during fluid injection was investigated. The amount of oil produced per injection cycle of salt water and nanoparticles was calculated, and the viscosity change is shown here. A centrifuge was used to separate the water and oil phases for 2 hours at 2000 rpm.

There are many methods to obtain the interfacial tension between nanofluids and oil. In this study, a tensiometer (Tensiometer Dataphysics/DCAT9) and the Du Noüy Ring method (equation (5)) were used to determine the IFT between the oil phase and the nanofluid at a limited temperature.

$$\gamma = \frac{F}{4\pi R \cos \theta} \left(0.725 + \sqrt{\frac{9.075 \times 10^{-4} F}{\pi^3 \Delta \rho \cdot g \cdot R^3} - \frac{1.679 r}{R} + 0.04534} \right) \quad (5)$$

γ = interfacial tension

F = force acting along the three-phase contact line equal to the weight of the liquid eniscus above the plane

R and r = dimensions of the ring

θ = surface wettability

$\Delta \rho$ = density difference of fluids

g = gravitational acceleration

FINDINGS

Figure 2 shows oil production rates across different sand packs.

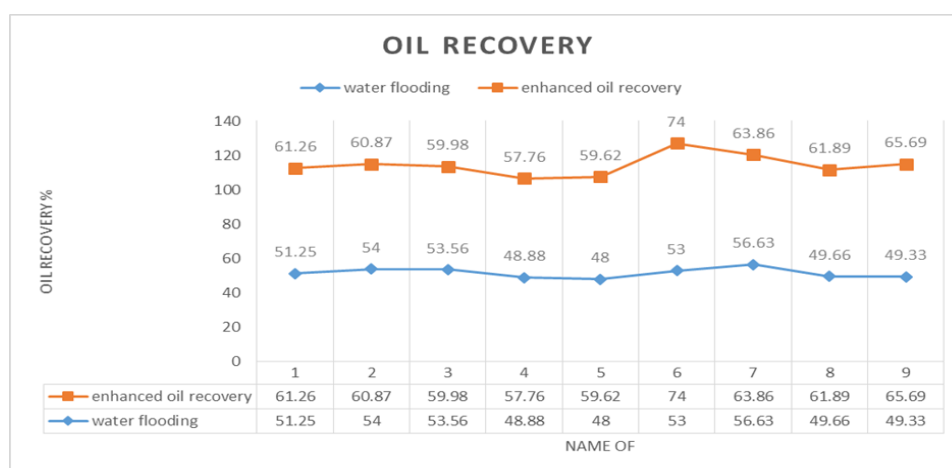


Figure 2: Oil recovery during the injection scenario

As observed, the experiment was conducted in 9 runs. Before the start of enhanced oil recovery, a 0.1 wt% salt solution was injected for reservoir simulation, and the average sodium chloride flooding result was 46.87%. Subsequently, the following results were obtained. Sand pack 1 is completely water-wet, and the production rate is approximately constant across all sand packs, which can be attributed to the uniform porous medium conditions.

Table 3: (Sand pack 1) Holder No. 1

Porous medium	Nanofluid	Porosity (%)	Permeability (Darcy)	Connate water saturation	Water flooding	EOR	Total recovery
Sand pack 1	Silica	42.96	2.27	18.37	51.25	10.00	61.26

As shown, a significant amount of oil was produced by this step, indicating the effect of using silica nanoparticles. It seems that the production resulting from replacing the initial solution with silica nanoparticles improves, albeit slightly, compared to water flooding, thereby enhancing the main mechanisms we will mention later (Table 3).

Table 4: (Sand pack 2) Holder No. 2

Porous medium	Nanofluid	Porosity (%)	Permeability (Darcy)	Connate water saturation	Water flooding	EO R	Total recovery
Sand pack 2	Copper oxide	42.53	2.93	14.18	48.88	8.88	57.76

The secondary solution recovered 8.88% of the remaining oil.

Table 5: (Sand pack 3) Holder No. 3

Porous medium	Nanofluid	Porosity (%)	Permeability (Darcy)	Connate water saturation	Water flooding	EO R	Total recovery
Sand pack 3	Titanium dioxide	40.60	2.82	19.43	53.56	5.98	59.98

According to Table 5 (Sand pack 3), only 53.56% of the original oil in place was produced, and capillary equilibrium was established. Nevertheless, the secondary solution recovered about 5.98% of the remaining oil, which clearly originates from various enhanced oil recovery mechanisms discussed later.

Table 6: (Sand pack 4) Holder No. 4

Porous	Nanofluid	Porosit	Permeabilit	Connate	Water	EO	Total
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medium		porosity (%)	permeability (Darcy)	connate water saturation	water flooding	R	total recovery
Sand pack 4	Aluminum oxide	41.09	2.37	14.29	54.00	7.31	60.87

Table 7: (Sand pack 5) Holder No. 5

Porous medium	Nanofluid	Porosity (%)	Permeability (Darcy)	Connate water saturation	Water flooding	EOR	Total recovery
Sand pack 5	Xanthan polymer	41.78	2.12	18.54	48.00	11.62	59.62

The EOR fluid in Sand pack 5, in addition to silica, contains xanthan gum polymer. The results from this core indicate that the presence of xanthan gum as a stabilizer significantly affects production.

Table 8: (Sand pack 6) Holder No. 6

Porous medium	Nanofluid	Porosity (%)	Permeability (Darcy)	Connate water saturation	Water flooding	EOR	Total recovery
Sand pack 6	Silica + xanthan gum	42.79	2.23	16.75	53.00	21.00	74.00

The EOR fluid in Sand pack 6, in addition to silica, contains xanthan gum polymer. The results from this core indicate that the presence of xanthan gum as a stabilizer significantly affects production. The production resulting from replacing the secondary solution is due to changes in several mechanisms.

Table 9: (Sand pack 7) Holder No. 7

Porous medium	Nanofluid	Porosity (%)	Permeability (Darcy)	Connate water saturation	Water flooding	EOR	Total recovery
Sand pack 7	Aluminum oxide + xanthan gum	42.02	2.33	15.25	53.63	13.33	68.86

Table 10: (Sand pack 8) Holder No. 8

Porous medium	Nanofluid	Porosity (%)	Permeability (Darcy)	Connate water saturation	Water flooding	EOR	Total recovery
Sand pack 8	Titanium dioxide + xanthan gum	42.96	2.00	19.10	49.66	12.23	61.89

Table 11: (Sand pack 9) Holder No. 9

Porous medium	Nanofluid	Porosity (%)	Permeability (Darcy)	Connate water saturation	Water flooding	EOR	Total recovery
Sand pack 9	Copper oxide + xanthan gum	41.51	2.18	17.99	49.33	16.66	65.69

CONCLUSION

After injecting water at twice the volume of the void space, the flooding results with 0.1% sodium chloride by weight were 87.46%. Also, the oil recovery in all four tubes using aluminum oxide (Al_2O_3), titanium dioxide (TiO_2), silica (SiO_2), and copper oxide (CuO) nanoparticles at ambient temperature is 48.7%, 50.9%, 52.9% and 51.1% of the oil produced in situ. Although the results show that the highest oil recovery occurred at this stage, this rate is not at the desired level based on previous predictions and conditions. After that, by injecting a suspension of nanofluids, a significant improvement in oil recovery has been achieved; So that by injecting a silica nanofluid ion suspension into the first tube, 15.66%, by injecting an aluminum oxide nanofluid suspension into the second tube, 12.38%, by injecting a titanium dioxide nanofluid suspension into the third tube, 18.27%, and by injecting a copper oxide nanofluid suspension into the fourth tube, 12.38% of the initial oil was produced in situ.

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