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TÍTULO: Un estudio experimental de la inyección de gas alternante nanofluida para la recuperación mejorada de petróleo del depósito fracturado Asmari con carbonato.

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RESUMEN: En esta investigación, se intenta detallar el efecto de los nanofluidos basados en nanohíbridos MWCNT-SiO2 para la mejora de la humedad de agua de la roca de yacimiento y la reducción de la tensión interfacial entre la inyección de nanofluido y el aceite de yacimiento. Se ha demostrado experimentalmente que el Nano-Alternating-Gas (NAG) tiene la capacidad de mejorar tanto la humectabilidad como la tensión interfacial (IFT) para aumentar el factor de recuperación del depósito de aceite Asmari.

PALABRAS CLAVES: Depósito de carbonato, depósito fracturado, nanosurfactante, humectabilidad, tensión interfacial .

TITLE: An Experimental Study of Nano-Fluid Alternating Gas Injection for Enhanced Oil Recovery of Carbonate Asmari Fractured Reservoir.

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ABSTRACT: In this research, it is attempted to detail the effect of MWCNT–SiO_2 nanohybrid based nanofluids on improvement of water wetness of reservoir rock and reduction in interfacial tension between injecting nanofluid and reservoir oil. It has been experimentally shown that Nano-Alternating-Gas (NAG) has the capabilities to improve both wettability and interfacial tension (IFT) to increase recover factor of Asmari oil reservoir.

KEY WORDS: Carbonate reservoir, fractured reservoir, Nano Surfactant, Wettability, Interfacial tension.

INTRODUCTION.

On average, the primary oil recovery of fractured carbonate reservoirs is less than one-third of the original oil in place (OOIP). The rest of the oil will be trapped in the rock due to high capillary forces that prevent oil from flowing through the matrix rock into the nearby fracture network. Any process that involves injection of fluid(s) to supplement natural reservoir energy by interacting with the rock-oil-brine system to create favorable conditions for maximum oil recovery is known as an enhanced oil recovery (EOR) process (Green, et al. 1998). These favorable interactions to maximize oil recovery may be oil swelling, lowering the interfacial tension, rock wettability modification, and oil viscosity reduction.

Secondary recovery are recovery techniques used to enhance the natural energy of the reservoir by artificially injecting fluid (gas or water) into the reservoir to force the oil to flow into the wellbore and the surface (Speight, 2009). The main objective of a secondary recovery program is to sweep the oil towards the production wells to increase productivity. Secondary recovery is also used to restore and maintain the reservoir pressure, which normally declines during the primary recovery phase.

Water alternating gas injection (WAG) is considered an enhanced recovery process. WAG injection involves drainage (D) and imbibition (I) taking place simultaneously or in cyclic alternation in the reservoir (Nezhad et al. 2006). Due to their low viscosities, gases have high mobility which results in poor macroscopic sweep efficiency (Hustad et al. 1992).

The injection of water after gas helps to control the mobility of the gas and stabilizes the displacement front. WAG recovery techniques combine the benefits of both water and gas injection, i.e., improved macroscopic sweep efficiency of water flooding with the high displacement efficiency of gas injection in order to increase incremental oil production (Kulkarni et al. 1980). Righi et al. (2004) conducted an experimental study of tertiary immiscible WAG injection by flooding several 38 mm diameter core plugs with water and gas slugs. Their experimental results show that WAG injection significantly increases tertiary oil recovery efficiency, leading to final residual oil saturations as low as 13% pore volume (PV).

Nanotechnology has shown its potential to revolutionize the petroleum industry for both upstream and downstream sectors in the recent years. Many types of research, especially for nano-EOR, have been done in the past few years and shown a promising result on the recovery improvement.

The experimental studies of Zhang 2009 and Espinoza 2010 have shown that the surface-coated silica NPs can stabilize emulsion which improves mobility control in EOR process. Ju et al. (2002 and 2006) based on the results of their experimental works, have shown that Hydrophilic silica NPs has the capabilities to alter the rock wettability and reduce interfacial tension.

Onyekonwu et al. (2010) studied several parameters of nanoparticles such as size, concentration, ionic compositions and NP type in relation to EOR and showed that silica NPs dispersed in ethanol could improve oil recovery up to 38%. Hendraningrat et al. (2013) showed that the optimal silica NPs concentration was 0.05wt % and concluded that the smaller size of NPs leads to better recovery. They have proved that nanoparticles were able to form adsorption layers on the surface of the grain and significantly change the wettability and interfacial tension of reservoir system.

Most of the clean sedimentary rocks are strongly water-wet, and generally, it was assumed that water films prevent oil from touching the surface of the reservoir. The strong water-wetness can be altered by the adsorption of polar compounds from oil and deposition of organic matter, i.e. aging. When the polar compound is adsorbed, the hydrocarbon end is exposed, making the surface more oil-wet.

In mixed wet carbonate rock, there exist two paths for fluid movement, i.e. smaller water wet pore network which contain connate water and larger oil wet pores containing reservoir oil. Initially, the reservoir oil is in contact with connate water and any improvement in water wetness and interfacial tension will have positive effect on increasing the recovery factor by increasing the capillary number (Nc).

Due to injection of Nanofluid Alternating Gas (NGA), oil will be displaced by two mechanisms, i.e. imbibition by wetting phase (nanofluid) and drainage by non-wetting phase (gas). Highly fractured Asmari carbonate reservoirs are a class of reservoirs characterized by high conductivity fractures surrounding low permeability matrix blocks. In these mixed wet reservoirs, wettability alteration is a key method for recovering oil from oil wet pores. When the oil wet pores are alternated to water wet, both imbibition (by water) and drainage (by gas) will be improved in WAG injection. In this study, nanofluid solution was used to enhance spontaneous imbibitions between the fractures and the matrix by both alternating wettability and lowering interfacial tensions. The wettability alternation and reduction in interfacial tension were determined by using contact angle and pendent drop measurement respectively.

Wettability and wettability alteration.

In the flow of two immiscible fluids in a porous media, wettability is the tendency of one fluid to adhere to the surfaces of the porous medium in the presence of the other fluid (Donaldsonet al. 2008). The fluid distribution, relative permeability, capillary pressure and residual oil saturation of reservoir rock after flooding by an immiscible phase depend on wettability. The wetting phase tends to filled up the smaller pores while the non-wetting phase occupies the bigger ones. The distribution of the fluids will affect the recovery of the oil. When the surface of the rock is water wet in a brine-oil reservoir, the water will tend to occupy the smaller pores and wet the surface of the bigger pores. While the rock surface is oil wet, the oil will adhere to the smaller pores by displacing the water and recovering the oil will be much difficult.

The most common way of defining wettability is using the contact angle (θ) which is measured through the denser fluid. The three broad classification of homogenous wettability are water-wet ($\theta < 70°$), intermediate-wet ($70° < \theta < 115°$), and oil-wet ($\theta > 115°$). Some of the parameters that affect the wettability of a porous medium are surface roughness, types and composition of fluids, etc. In addition, there exists heterogeneous state of wettability which is mixed wet, i.e. the smaller pores are water wet and occupies by connate water, while the wettability of oil saturated larger pores have been altered to oil wet state.

Nano particles.

Multiwalled carbon nanotube (MWCNT)-Silica nanohybrid structures are very suitable material for enhanced oil recovery in order to their excellent interfacial activity and wettability enhancement. In the oil/water interface, they could change the oil properties to reduce the capillary pressure and accelerate mobilization of the reservoir oil.

Multiwalled carbons nanotubes, which have been used in this research, were collected from Research Institute of Petroleum Industry (RIPI). In order to decrease the cost of raw material, RIPI has been constructed the MWCNT production Demo plant with high production capacity. Very low dosage, i.e. 0.2 wt % nanohybrid is necessary for the preparation of nanofluid, and periodic gas injection in Nanofluid Alternating Gas (NAG) injection will cause more reduction in the amount of required nanohybrid and enhancement in rock water wetness.

Nano particles used in this study were silica nanopowder with spherical morphology, which dispersed in deionized water as aqueous phase by ultrasonic radiation probe apparatus for 30 minutes. The nanohybrid sample was characterized by XRD, and SEM analyses. The details of nanofluid preparation procedure was the same which presented by Ershadi, et al. 2015.

Core flooding experimental work.

Materials and Methodology.

Ten 35cm length and 5 cm diameter cylindrical rock samples without micro fractures or vugs were cut from a homogeneous outcrop of Asmari carbonate reservoir (92% calcite and 8% dolomite) located in the south of Iran. To simulate the reservoir fracture network, the cylindrical models were cut by two diagonal fractures. Impermeable spacers were put between four pieces of each model to hold the fractures opened during the experiments. The reservoir oil and gas samples were collected from test separator of Ahwaz production unit.

The interfacial /surface tension of two immiscible fluids, i.e. oil/gas, water/oil, nanofluid/oil were measured by a pendant-drop apparatus. Density and viscosity of test fluids, i.e. oil, gas, water, nanofluid were measured by Anton Paar DMA 4500 density meter and Rollring ball viscometer apparatus respectively.

All the experiments were performed using 20000 ppm NaCl brine and a reservoir oil with viscosity of 17 cp at ambient condition. Chemical materials including silica nanoparticle were received from Research Institute of Petroleum Industry (RIPI).

Tetraethylorthosilicate (TEOS) 99%, absolute ethanol (EtOH) 99.5%, ammonia solution 25% were used as received from Merck Company. MWCNT was received from Research Institute of Petroleum Industry (RIPI) that was prepared by chemical vapor deposition (CVD) method. The Nanohybrid sample was characterized by Field Emission Scanning Electron Microscope (FE-SEM), using a Holland Phillips XL30 microscope. XRD patterns of the samples were recorded in ambient air using a Holland Philips X-ray powder diffraction (Cu Ka, k = 1.5406 Å), at scanning speed of 2^{o}/min from 20^{o} to 80^{o}. Transmission Electron Microscopy (TEM) was performed with a Philips EM 208 FEG instrument operating at 90 kV (Ershadi et al., 2015).

Flooding Experimental procedure.

The secondary recovery, water alternating gas and nanofluid alternating gas experiments were carried out in a fractured carbonate rock model. The cylindrical fractured physical mode has approximately 35cm length and 5 cm diameter. Before running each test, the glass micro model was cleaned to assure no oil is remained after previous run. During cleaning procedure, distilled water and toluene were injected into core holder to circulation with several cycles. The flooding apparatus involved are: 1- Pump, 2- Gas container, 3- Brine continer, 4- Nanofluid solution, 5- Oil container, 6- Pressure regulator, 6-Pressure regulator, 7- Gas flow meter, 8- Core holder, 9-

Pressure pump, 10- Back pressure regulator, 11- Pressure sensor, 12- Gas-liquid separator, 13- Cylinder, 14- Computer, 15- Thermostat.

All seven experiments were performed at the same initial condition, i.e. oil and water saturation at atmospheric pressure and 60 °F. The injection rates were set at the same value, i.e. 0.5 cc/hr in all experiments for both liquid (water and Nano) and Gas. The experiments were carried out by flooding fractured carbonate models saturated with Asmari oil (So = 82%) and brine (Scw = 18%) by natural gas, water, Nano solution, WAG and NAG.

The physical models with two diagonal fractures after saturated with reservoir oil and connate water (So = 82 %, Scw = 12%) were placed horizontally in core holder. To compare the performance of different injecting phase, i.e. gas, water and nanofluid on oil recovery, 1.2 pore volume of gas, water and nanofluid were injected at 0.5 cc/hr for the first series of experiments, i.e. experiments no.1 to no. 3.

In second series of experiments, i.e. experiments no.4 to no. 7, the water alternating gas injection (WAG) and the Nano-fluid alternating gas injection (NAG) experiments were performed in two scenarios, i.e. liquid-gas-liquid and gas-liquid-gas. Each experiment in the second series was performed in three alternating 0.4 pore volume injection periods of gas and liquids.

CONCLUSIONS.

The performed experiments indicated that water-based nanofluid increases the ultimate recovery considerably both as secondary recovery and nanofluid alternating gas injection. The capabilities of nanofluids to overcome the WAG challenges by reduction in surface tension and wettability alteration to water wetness have been shown experimentally.

The key results of the experiments are listed as following:

- Our study showed that adsorption of SiO2 nanoparticles on the rock surface changed the wettability of the oil-wet pores (ϴ = 124^{o}) to strongly water-wet (ϴ = 22^{o}), which affects the oil recovery performance.
- Reduction of IFT (from 37 mN/m to 18 mN/m) occurred due to placement of the nanoparticles on the oil and water interface.
- In mixed wet fractured carbonate rock, the performance of gas injection is about 30% higher than water injection.
- Due to reduction in surface tension (from 37 mN/m to 18 mN/m) and wettability alteration to water wetness (contact angle altering from 124^{o} to 22^{o}), the ultimate recovery of nanofluid injection is about 19% higher than the water injection.
- Experimental results demonstrated more than 11% and 26% incremental in recovery factor by nanofluid alternating gas injection process in comparison to the conventional WAG in gas-liquid-gas and liquid-gas-liquid cyclic injection scenarios respectively.
- Nano-gas-nano scenario has more effect on surface tension reduction and wettability alteration compare to gas-nano-gas injection scenario, since the amount of nano in N-G-N was twice that in G-N-G, which causes 13% increase in ultimate recovery.

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