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## Improving Fluid Flow through Low Permeability Reservoir in the Presence of Nanoparticles: An Experimental Core Flooding WAG Tests

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### Highlights

- Introducing two nanoparticles of calcium oxide and silica in carbonate porous media;
- Using calcium oxide and silica nanoparticles to improve two-phase relative permeability parameters;
- Selecting optimum concentration and best nanoparticles based on the results;
- Improving water alternating gas tests in the presence of best-selected nanoparticles.

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### Abstract

Recently, nanoparticles have been used to improve oil and gas production volume and enhance oil recovery (EOR). Based on our recent research, using nanoparticles such as silica and calcium oxide has a good potential for changing mechanisms in the porous media, such as interfacial tension and wettability. Low permeability carbonate plugs were selected to determine the application of nanoparticles in the porous media. Two main steps were used: 1) Using CaO and SiO<sub>2</sub> nanoparticles for wettability alteration, interfacial tension reduction, and improving fluid flow through porous media, and 2) Surveying the application of nanoparticles to the water alternating gas (WAG) (nanoparticles (NCs)-assisted WAG) test. The zeta potential values were stable at  $-56.4 \pm 2$  mV and  $-44.0 \pm 3$  mV for calcium oxide and silica nanoparticles, respectively, at an optimum nanoparticle concentration of 15 ppm. Calcium oxide and silica nanoparticles effectively altered the wettability from oil-wet to water-wet by surveying the intersection of two-phase relative permeability. Moreover, CaO nanoparticles performed better in low permeability carbonate porous media than SiO<sub>2</sub> nanoparticles regarding wettability alteration to water wetness. Based on the results and a better grade of CaO, it was selected for performing NCs-assisted WAG tests at WAG ratios of 1:1, 40 °C, and 15 ppm. The recovery factor increased from 42.9% to 73% in the presence of CaO during NC-assisted WAG tests, and residual oil saturation decreased from 40.9% to 19.4%.

**Keywords:** Calcium Oxide Nanoparticles, Silica, WAG, Wettability, Low Permeability Reservoir

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### 1. Introduction

A considerable volume of crude oil remains in oil reservoirs after performing primary oil recovery. Other enhanced oil recovery (EOR) methods, such as gas injection, are used to improve the production rate in different scenarios (Kulkarni et al., 2005). One of the essential factors that poses a force in finding a solution for gas scenarios is low sweep efficiency due to its early breakthrough. Accordingly, water alternating gas (WAG) methods combine primary water flooding and gas injection to enhance

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ultimate oil recovery (Awan et al., 2005; Crogh et al., 2002). It is shown that using the WAG method is cost-effective in comparison to gas scenarios, and the primary purposes of using the WAG method are lowering fingering issues and mobility ratio and improving effective mechanisms (Changlin-lin et al., 2013; Ahmadi et al., 2015; Claridge et al., 1982; Agbalaka et al., 2008; Latil et al., 1980; Craig et al., 1971; Matthews et al., 1988; Yokoyama et al., 1981; Jones et al., 1989; Gorell et al., 1990; Fayers et al., 1990; Hanssen et al., 1987; Sorbie et al., 1987). However, there are still problems with ultimately reaching these goals. Using chemicals such as nanoparticles, polymers, surfactants, and alkaline is ordinary for improving mobility ratio and fingering problems in routine WAG tests by considering effective mechanisms such as wettability and interfacial tension (Samanta et al., 2012).

Among different mentioned chemical methods, nanoparticles show advantages in passing quickly through pores, stability, and changing main mechanisms in the reservoirs such as interfacial tension (IFT) and contact angle, especially in harsh conditions (Ko et al., 2019; Ahmadi et al., 2018; Ahmadi et al., 2020). Ahmadi et al. (2018 and 2020) performed laboratory research in the presence of nanoparticles, including calcium oxide and silica nanoparticles, for different purposes, such as improving water–oil relative permeability, reducing interfacial tension, changing wettability, and asphaltene adsorption on nanoparticle surfaces. It was concluded that calcium oxide and silica nanoparticles changed wettability from oil-wet to water-wet, and the IFT decreased in the presence of calcium oxide and silica nanoparticles. Moreover, Ahmadi et al. (2015) performed experimental work on different WAG scenarios, and according to their results, associated gas had the best results for increasing ultimate oil recovery.

This paper used calcium oxide and silica nanoparticles to perform interfacial tension, wettability, and displacement tests to obtain the optimum nanoparticle concentration. Then, CaO nanoparticles were used to perform NCs-assisted WAG tests in the obtained condition.

## 2. Material and methods

### 2.1. Materials

Crude oil and plugs were selected from one of the Iranian oil carbonate reservoirs, and the viscosity and oil density are 9.9 cP and 0.864 g/cm<sup>3</sup> at 40 °C, respectively. Tables 1 and 2 list the specifications of plugs for reservoir oil-associated gas compositions, respectively.

**Table 1**  
Reservoir oil composition

Components	Reservoir oil (mole %)	Associated gas (mole %)
H <sub>2</sub> S	0.01	(-)
N <sub>2</sub>	0.08	5.13
CO <sub>2</sub>	0.56	41.28
C <sub>1</sub>	3.44	20.11
C <sub>2</sub>	1.88	15.17
C <sub>3</sub>	3.45	2.52
iC <sub>4</sub>	1.23	7.14
nC <sub>4</sub>	5.77	2.12
iC <sub>5</sub>	3.70	2.40
nC <sub>5</sub>	5.30	2.12
C <sub>6</sub>	5.92	0.96
C <sub>7+</sub>	68.67	1.43

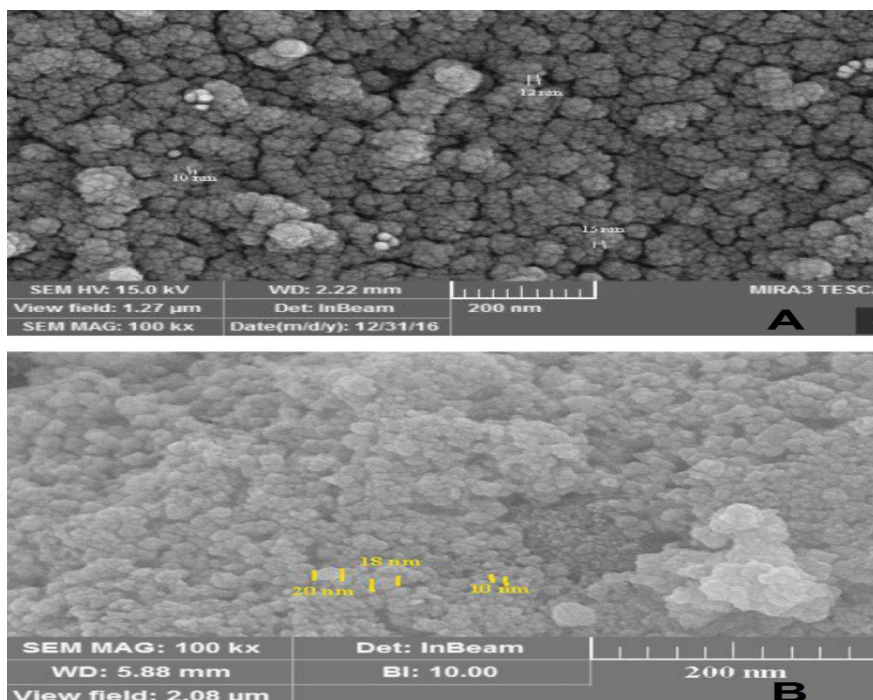
**Table 2**  
Plugs specification

Properties	Plug 1	Plug 2
Mineralogy	Carbonate	Carbonate
Length (cm)	4.98	4.82
Diameter (cm)	3.85	3.84
Gas pore volume (cc)	10.78	10.59
Gas permeability (cc)	9.63	9.77
Gas porosity (%)	18.60	18.97
Brine pore volume (cc)	11.35	11.43
Brine porosity (%)	19.57	20.45
Brine permeability (mD)	0.16	0.18

Merck toluene and ethanol were used for asphaltene extraction in the IP143 test and cleaning the IFT needle apparatus, respectively. After contacting oil with a needle valve, the needle should be placed in ethanol to prevent it from becoming oil-wet. The formation water at 50000 ppm was used to make nanofluids. Viscosity and brine density were 0.880 cP and 1.025 g/cc at 40 °C, respectively. Silica nanoparticles were provided from the commercial Houston brand, and calcium oxide nanoparticles were synthesized according to the procedure given below (Singh et al., 2007):

1. Mixing 5 mL succinic, tartaric, citric acids with 10 g of CaCO<sub>3</sub>
2. Drying the mixture at 100 °C for two hours;
3. Heating the mixture at 900 °C for two hours;

The SEM and properties of calcium oxide and silica are presented in Figure 1 and Table 3, respectively.



**Figure 1**

The SEM of (a) silica and (b) calcium oxide nanoparticles

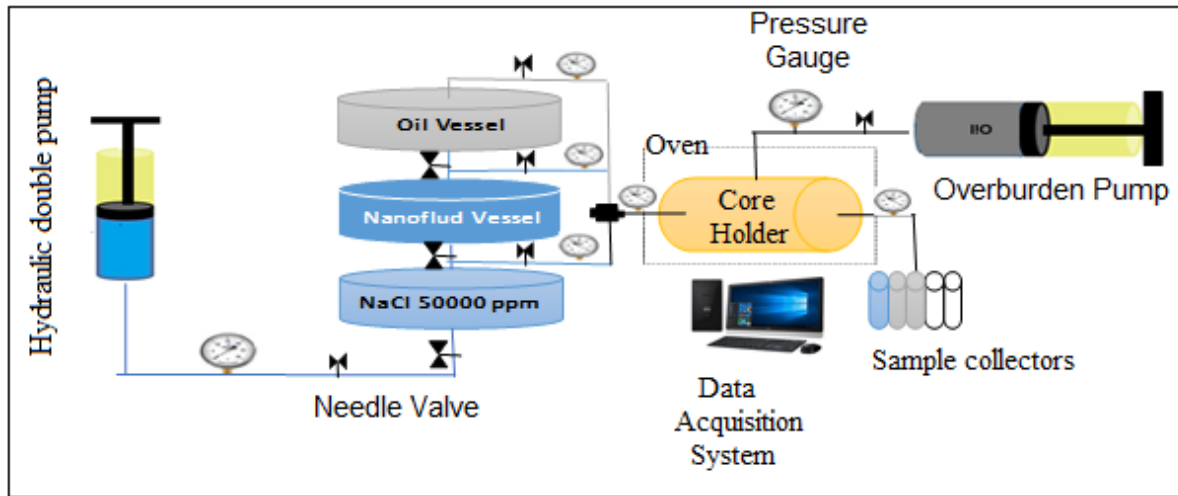
**Table 3**  
Specification of the nanofluids

Nanofluids type/properties	Silica	Calcium oxide
Concentration (ppm)	15.00	15.00
Density (g/cc)	0.99	0.99
Nanoparticle size (nm)	20.00	20.00
Viscosity (cP)	1.29	1.65
Nanoparticles purity (%)	95.00	95.00
pH (-)	6.71	6.75

## 2.2. Methods

### 2.2.1. Displacement tests

Ten displacement tests were done to obtain the optimum amount of calcium oxide and silica nanoparticles in low-permeable carbonate porous media. Attention to an optimum nanoparticle concentration is essential in preventing any blockage in porous media (Teng et al., 2017). A dynamic apparatus schematic was used to perform 10 displacement tests in the presence of CaO and SiO<sub>2</sub> nanoparticles in Figure 2.



**Figure 2**

The displacement test apparatus (Ahmadi et al., 2018)

The displacement test setup contained three transfer vessels of crude oil, brine, and nanofluids; two pumps connected to the transfer vessels with high-pressure and high-temperature lines; a core holder; oven; pressure gauges; hand pump; pressure; and the acquisition system. The main procedure for performing the displacement test is summarized as follows:

- Core saturation of cores with brine;
- Obtaining initial water saturation ( $S_{wi}$ ) by performing crude oil injection in low permeable carbonate porous media;
- Performing aging conditions for three weeks;
- Calculation of Kabs for crude oil;

- e. Obtaining tertiary oil recovery;
- f. Injection of prepaid nanofluids;
- g. Repeated tertiary oil calculations in the presence of CaO and SiO<sub>2</sub> nanoparticles.

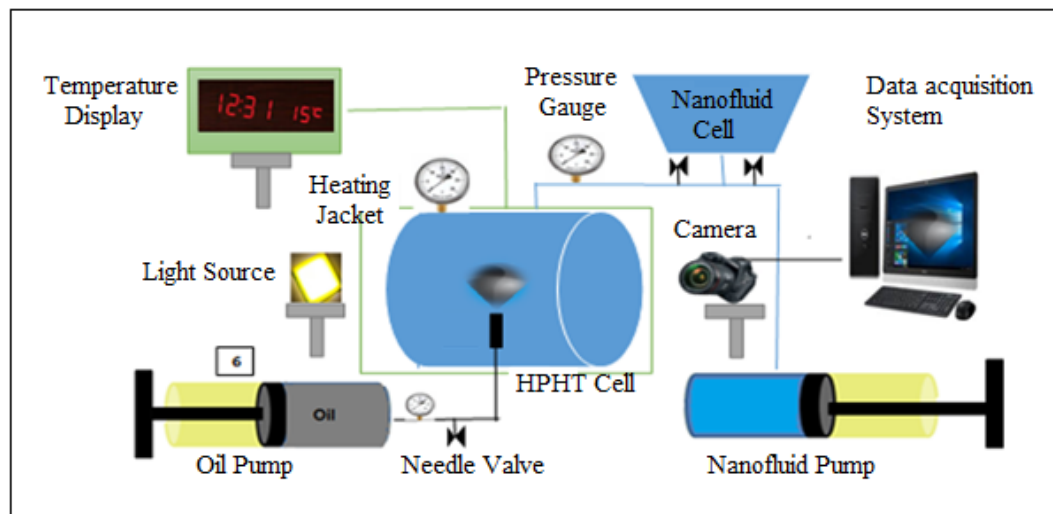
### 2.2.2. Oil–water relative permeability

The main procedure of performing relative permeability is summarized as follows:

- a. Porosity and permeability measurements (ASTM Standard D2434-68);
- b. Saturation of carbonate cores with brine;
- c. Obtaining  $S_{wi}$  with an injection of crude oil;
- d. Performing aging condition for three weeks;
- e. Calculation of the oil–water relative permeability parameters;
- f. Introducing 0.5 PV nanofluid in the porous media;
- g. Calculation of the oil–water relative permeability parameters in the presence of calcium oxide and silica nanoparticles.

### 2.2.3. Oil–water interfacial tension

A schematic of the high-pressure and high-temperature IFT setup is shown in Figure 3. It contains a high-pressure and high-temperature vessel, a heating jacket, and fluid pumps. Crude oil is transferred through pumps to capture oil droplets after transferring fluid to the primary cell and removing trapped air from the top. Finally, raw data are analyzed with acquisition software.



**Figure 3**

The high-pressure and high-temperature IFT setup (Ahmadi et al., 2018)

### 2.2.4. WAG and nanoparticles-assisted WAG tests

The experimental procedure for performing the WAG tests is summarized as follows :

- a. Measuring the dry weight of a core sample;
- b. Vacuum process on carbonate plugs;
- c. Saturation of carbonate plugs with brine;
- d. Measurement of the wet weight of the core sample;
- e. Measurements of  $K_{abs}$  with Darcy's law;
- f. Obtaining  $S_{wi}$  by the injection of oil;

- g. Aging conditions for at least three weeks;
- h. Performing WAG tests at a gas/water cycle ratio, injection rate, and temperature of 1:1, 15 and 8 cc/h, and 40 °C, respectively;
- i. Repeating the experimental procedure for performing NC-assisted WAG tests.

### 3. Results and discussion

#### 3.1. Displacement test

Researchers have used different nanoparticles for increasing recovery factors such as SiO<sub>2</sub>, CaO, TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and CaCO<sub>3</sub> according to effective mechanisms such as wettability and interfacial tension (Agi et al., 2018; Bayat et al., 2015; Ju et al., 2012; Onyekonwu et al., 2010; Roustaei et al., 2013). Researchers' main concerns are calculating optimum nanoparticle values and stability (Dishon et al., 2009). Thus, 10 different tests were designed in this paper to obtain the optimum values by performing displacement tests, as listed in Table 4. Five different nanoparticle concentrations of 5, 10, 15, 20, and 25 ppm were selected during the displacement tests, and these concentrations were selected based on our previous work (Ahmadi et al., 2020) and preventing any blockage in the selected carbonate core; the brine permeability was 0.16–0.18. According to the results for CaO and SiO<sub>2</sub> nanoparticles, tertiary oil recovery increased to 15 ppm concentration. Then, after 15 ppm, it was seen that the tertiary oil recovery decreased. Thus, 15 ppm was selected as an optimum nanoparticle concentration in the two-phase relative permeability, WAG, and NCs-assisted WAG tests. Moreover, the zeta potential values were  $-56.4 \pm 2$  mV and  $-44.0 \pm 3$  mV for calcium oxide and silica nanoparticles, respectively. The absolute quantity of zeta potential is essential, balancing between electrostatic repulsive forces and van der Waals attractive. Values above 30 mV are stable solutions (Rezk et al., 2019).

**Table 4**  
Displacement tests results

Nanoparticles type	Nanoparticle concentration (ppm)	Primary recovery factor after primary water flooding (%)	Final recovery factor after using nanoparticles (%)	Tertiary recovery (%)
Hydrophilic calcium oxide	5	55.29	61.25	5.96
	10	55.15	63.50	8.35
	15	55.00	65.50	10.50
	20	55.12	64.70	9.58
	25	55.39	63.90	8.51
Hydrophilic silica	5	63.41	67.20	3.79
	10	63.32	68.50	5.18
	15	63.50	71.50	8.00
	20	63.58	69.70	6.12
	25	64.12	68.30	4.18

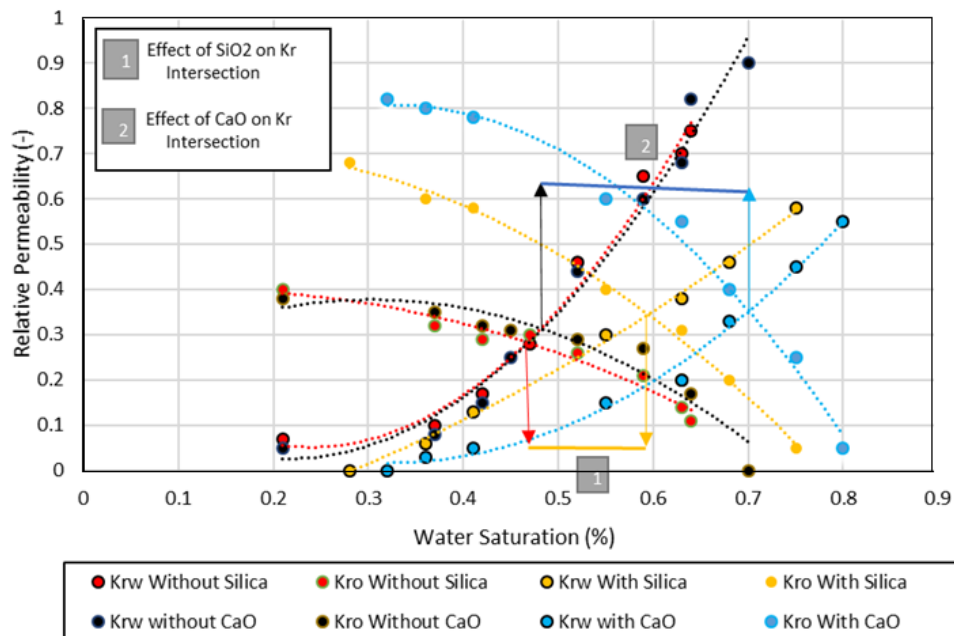
#### 3.2. Relative permeability and interfacial tension tests

This paper used the unsteady-state method following the work of Toth et al. (2002) to obtain relative permeability. Figure 4 shows relative permeability versus water saturation in the presence of calcium oxide and silica nanoparticles. Both calcium oxide and silica nanoparticles effectively altered the wettability from oil-wet to water-wet, and the intersection moved further to the right side for calcium

oxide compared to silica (Point 1 and Point 2). Before using both calcium oxide and silica nanoparticles, the oil–water relative permeability intersection was less than 50% of water saturation, indicating the oil-wet condition of the cores. Both nanoparticles changed intersections by more than 50%, and these trends could be interpreted as the wettability of the cores changing from oil-wet to water-wet in the presence of nanoparticles. The calcium oxide nanoparticle had a better function for changing wettability from oil-wet to water-wet than the silica nanofluid. It was found that due to the evaporation of the light component of crude oil while raising the temperature, heavy oil was deposited, and due to a reaction between the carbonate surface and the heavy component, the surface became oil-wet (Khoramian et al., 2019). A layer of nanofluids is formed during adsorbing NCs on the carbonate rock surface. Although the formed layer depends on different factors such as NC concentration, size of NCs, and rock characteristics, the interaction of rock and NCs makes a carbonate oil reservoir from oil-wet to water-wet.

It is also shown that while increasing the nanoparticles concentration, the main reason for altering wettability from oil-wet to water-wet is the impact of repulsion forces. Moreover, IFT is known as one of the main parameters in EOR due to its effects on capillary forces. IFT changes can minimize the contact angle and maximize disjoining pressure. Furthermore, it is observed that adding nanoparticles has a substantial impact on the wettability of the carbonate reservoir up to a particular nanoparticle concentration. After that, permeability reduction occurs (Al-Anssari et al., 2016).

Next, it was observed that calcium oxide decreased oil–water interfacial tension more than silica: silica reduced it from 46.414 to 39.408 mN/m, while calcium oxide reduced it from 46.414 to 28.773 mN/m. IFT is known as one of the main parameters in EOR due to its effects on capillary forces in reservoirs (Abramov et al., 2019). Finally, calcium oxide nanofluid improved oil flow in porous media more than commercial silica fluid and had better efficiency in the carbonate oil reservoir. Based on comparing the displacement tests and relative permeability parameters of calcium oxide and silica, calcium oxide was selected to perform other WAG and nanoparticle-assisted WAG experimental tests.

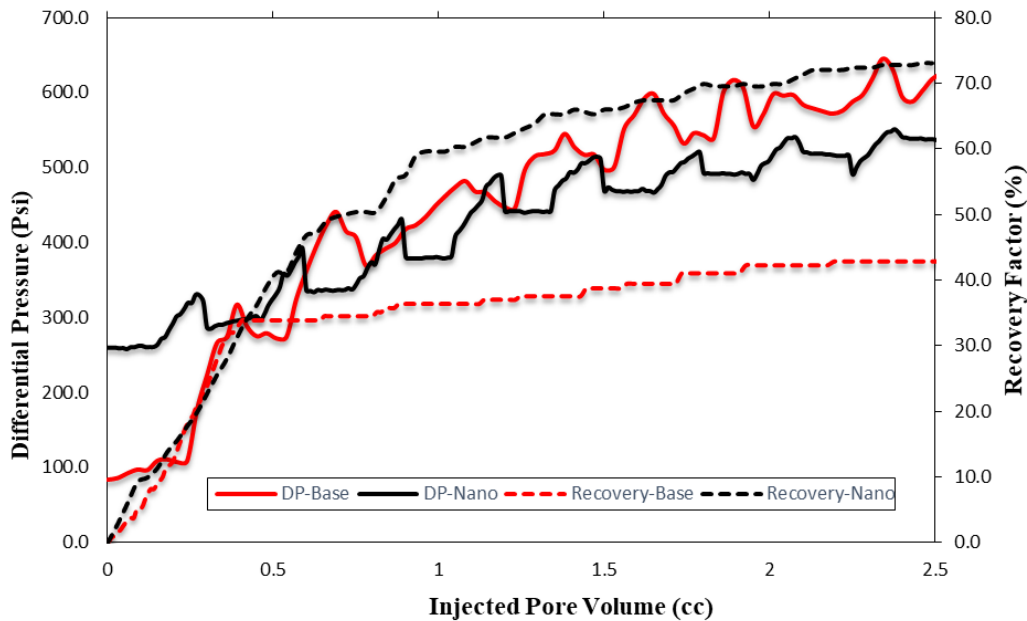


**Figure 4**

The effects of calcium oxide and silica nanoparticles on oil–water relative permeability parameters

### 3.3. WAG and nanoparticle-assisted WAG

In this paper, calcium oxide was selected to improve WAG tests as one of the standard chemically enhanced WAG methods at a concentration of 15 ppm. Figure 5 shows the effect of calcium oxide nanoparticles on the recovery factor and differential pressure at 15 ppm. The recovery factor increased from 42.9% to 73.0% in the presence of calcium oxide nanoparticles. Moreover, average WAG and NCs-assisted WAG injection pressures were 700 and 2500 psi, respectively, and it was seen that during NCs-assisted WAG tests, the recovery factor increased until reaching a plateau. The mentioned plateau started with a delay due to the addition of calcium oxide nanoparticles. Increasing recovery factor (RF) and delaying in the plateau were two main effects of adding CaO nanoparticles in NC-assisted WAG tests. Calcium oxide nanoparticles decreased IFT from 46.414 to 28.773 mN/m at 15 ppm, and the main reason was changing the state of fluid from Newtonian to non-Newtonian state (pseudo-plastic) (Cheraghian et al., 2016; Suleimanov et al., 2011). Furthermore, residual oil saturation decreased from 40.9% to 19.4% in the presence of calcium oxide nanoparticles, and the amount of water produced in the presence of nanoparticles decreased compared to the routine WAG test. These results indicated that calcium oxide increased the efficiency of the standard WAG test.



**Figure 5**

Recovery factor and differential pressure of the WAG and NCs-assisted WAG tests

## 4. Conclusions

This paper compared two nanoparticles of calcium oxide and silica to improve fluid flow through low-permeable carbonate porous media. Calcium oxide offered better results than silica nanoparticles concerning IFT reduction, wettability alteration from oil-wet to water-wet, and enhancing fluid flow through porous media. Accordingly, calcium oxide at an optimum concentration of 15 ppm was selected for improving the water alternating gas tests, and the following results were extracted during the NCs-assisted WAG tests:

- Increasing the recovery factor from 42.9% to 73.0% in the presence of calcium oxide nanoparticles;
- Decreasing the residual oil saturation from 40.9% to 19.4% in the presence of calcium oxide nanoparticles;
- Decreasing the produced water in the presence of calcium oxide nanoparticles;

At an optimum concentration of 15 ppm, calcium oxide nanoparticles can be promising for improving water alternating gas and two-phase relative permeability parameters.

## Nomenclature

CaO	Calcium oxide
cP	centipoise
EOR	Enhanced oil recovery
IFT	Interfacial tension
Kabs	Absolute permeability, mD
NCs	Nanoparticles
ppm	Parts-per-million
PV	Pore volume, cc
RF	Recovery factor, %
SEM	Scanning electron microscope
SiO <sub>2</sub>	Silicon dioxide
$S_{wi}$	Initial water saturation
WAG	Water alternating gas

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