

Contents lists available at ScienceDirect

Fuel

journal homepage: www.elsevier.com/locate/fuel



Improving oil recovery from shale oil reservoirs using cyclic cold nitrogen injection – An experimental study



Khalid Elwegaa, Hossein Emadi*

Texas Tech University, 2500 Broadway, Lubbock, TX 79409, USA

ARTICLE INFO

Keywords: Unconventional reservoir Cyclic gas injection Oil recovery factor Thermal shock Nitrogen gas

ABSTRACT

In this experimental study, effects of injecting temperature and pressure on oil recovery factor (RF) of shale oil reservoir was investigated by implementing cyclic cold nitrogen injection on Eagle Ford core samples.

Four outcrop core samples from Eagle Ford were used in this study. Nitrogen was injected at various temperatures ($-26\,^{\circ}\text{C}$ ($-15\,^{\circ}\text{F}$), $-18\,^{\circ}\text{C}$ ($0\,^{\circ}\text{F}$), $0\,^{\circ}\text{C}$ ($32\,^{\circ}\text{F}$), and $23\,^{\circ}\text{C}$ ($74\,^{\circ}\text{F}$)) and pressures ($6.9\,\text{MPa}$ ($1000\,\text{psi}$), $10.3\,\text{MPa}$ ($1500\,\text{psi}$), $13.8\,\text{MPa}$ ($2000\,\text{psi}$), and $20.7\,\text{MPa}$ ($3000\,\text{psi}$)) into the saturated core samples. Oil recovery factor for each experiment was calculated during three days of production period. Using average Young's modulus and Poisson's ratio of the samples, applied thermal stress due to the thermal shock was calculated. Furthermore, Computed Tomography (CT) scanner was deployed to scan the core samples prior to and after performing the experiment.

The results demonstrated that injecting nitrogen at low temperatures increases oil RF than does injecting nitrogen at ambient temperature. Injecting cold gas resulted in increase in the cumulative oil recovery factor by 10% and the highest recovery factor was observed at the operating pressure and temperature of 20.7 MPa (3000 psi) and $-26\,^{\circ}\text{C}$ ($-15\,^{\circ}\text{F}$), respectively. The results demonstrated that thermal stress applied to the core samples owing to injecting cold nitrogen resulted in creating new cracks and/or extending the existing ones. Additionally, the results demonstrate that injecting cold nitrogen resulted in widening the existing crack in the core samples. Hence, implementing cyclic cold nitrogen injection could potentially improve the efficacy of the current industry practice of cyclic gas injection technique in shale oil reservoirs.

1. Introduction

The oil industry is still investigating how to increase recovery from shale-oil and shale-gas reservoirs. In these reservoirs, production quickly declines owing to very low permeabilities. Also, these reservoirs do not produce economically unless hydraulic fractures are created to open a path between the wellbore and the reservoir matrix [16,9,12,17,18]. Experimental works have been and continues to be done to increase oil recovery from shale oil reservoirs [20,7,28]. Cyclic Gas Injection (CGI), as a successful technique, has been researched to stimulate the area surrounding the wellbore and increase oil recovery factor (RF) by injecting nitrogen (N_2) , carbon dioxide (CO_2) , and methane (CH_4) .

1.1. Cyclic gas injection method

It has been known for decades that Cyclic Gas Injection (CGI) is a successful method to increase oil production from conventional

reservoirs. Recent researches demonstrate that it can also be successfully implemented in unconventional reservoirs [17]. CGI also known as huff-and-puff technique is an efficient Enhanced Oil Recovery (EOR) method in which a slug of gas is injected into a reservoir either in miscible or immiscible condition (huff cycle). Then the well is shut-in for a "soak" period to allow the injected gas to interact with the formation oil and reach equilibrium. At the end of the soaking period, the production is resumed through the same well (puff cycle). Contributory mechanisms in increasing oil recovery in cyclic injection processes are oil viscosity reduction, oil expansion due to dissolution of injected gas in crude oil, solution gas drive aided by gravity drainage, vaporization of lighter components of oil, interfacial tension reduction, and relative permeability alteration [19,21]. Formation oil viscosity increases during the huff cycle owing to low temperature of injected gas, then it goes back to its original value during the soaking stage, which is substantially longer than the huff cycle. Experimental studies have been conducted to investigate the applicability of CGI in shale formations, and the results have been very promising [22,26,7,28]. Nitrogen is the

E-mail address: h.emadibaladehi@ttu.edu (H. Emadi).

^{*} Corresponding author.

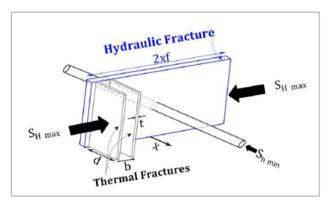


Fig. 1. Thermal fractures created perpendicular to the hydraulic fracture [6].

best option among all the gases used in this technique due to its low cost and availability.

1.2. Cold fracturing method

Cold Fracturing is a technique designed to improve oil recovery from unconventional reservoirs. In this technique, a gas at a low temperature is injected into a hot reservoir. This process is called Thermal Shock, which leads to create thermal stress on the rock. Temperature difference between the injected gas and the wall of the wellbore results in an increase in tensile stress and if the increase is adequate, a secondary crack is initiated. If the cold gas is injected into a fractured well, the temperature difference causes an increase in tensile stress and a decrease in the maximum horizontal effective stress (stress in the direction parallel to the main fracture in case of transverse fracture). If the decrease in maximum horizontal effective stress and the increase in tensile stress are adequate, a secondary crack is initiated resulting in an increase in the fracture conductivity yielding to higher oil recovery (Fig. 1).

Thermal stress applied to a solid body due to temperature change is calculated using the following equation (1).

$$\sigma_{thermal} = E(1+\nu)\alpha [T_i - T_s]$$
 (1)

where $\sigma_{thermal}$ is thermal stress, E is Young's modulus, ν is Poisson's ratio, T_i is initial temperature of solid body, and T_s is temperature of solid surface exposed to cold fluid.

Existence of natural fractures reduces rock tensile strength that helps thermal shock technique be successfully implemented resulting fracture creation and extension (Fig. 2).

Geyer and Nemat-Nasser [8] thermally induced parallel edge cracks in a half-plane consisting of brittle material. A glass plate was heated to a uniform temperature and then was exposed to a liquid bath cooled by dry ice. When the edge was in contact with the cold liquid bath, a

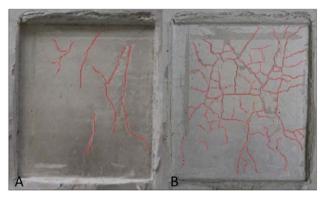


Fig. 2. An artificial rock sheet: A) existing fracture before cryogenic treatment, B) new fractures after cryogenic treatment [29].

thermal edge developed in the solid. This caused the solid edge to thermally contract, creating tensile cracks. These cracks propagated perpendicular to the cooled edge. Kim and Kemeny [14] conducted laboratory tests to study effects of rapid cooling on existing cracks. Granite, diabase with ore veins and KVS (Cretaceous Volcanic Sediments, a crystal tuff) samples were tested in the study. The samples were slowly heated to 100 °C (212 °F) and rapidly cooled afterwards. The results showed that crack growth, caused by thermal shock, occurred in some rock types. Tarasovs and Ghassemi [23] developed a simulation model to study the process of thermal stimulation. The results indicated that secondary cracks were mainly determined by temperature distribution in a geothermal reservoir. Kumar and Gutierrez [15] developed a two-dimensional transient heat flow model to estimate thermal induced effects on fracture geometry. A cold fluid was injected into hot rocks causing the rocks surrounding the fracture become relatively cool resulting in changes in tensile stresses and expansion of the fracture volume. Using a 2D plane strain simulation model, Tran et al. [24] studied effects of temperature difference between injected cold fluid and a hot reservoir on crack initiation. The results demonstrate that thermal stresses could create secondary fractures. The results also demonstrated that even a short-term injection could create and propagate the secondary cracks. Hamdi et al. [10] conducted a non-isothermal compositional simulation to examine the efficacy of low temperature CO2 injection process in hot reservoirs. The results showed that the oil RF increased by 8.73% after 24 years of production.

In the last few years, several experimental works have been conducted to study the cold fracturing technology and its applications. Using liquid nitrogen, Alqahtani [1] carried out cryogenic fracturing tests on concrete, tight sandstone, and Niobrara shale samples. The results demonstrated that cold fracturing enhances the samples' permeabilities without causing any formation damage. Zhao et al. [29] measured mechanical properties of an artificial rock while the rock samples were subjected to liquid nitrogen. The results showed that subjecting the samples to liquid nitrogen significantly reduces shear strength and tensile strength of the samples. The thermal shock resulted from the cryogenic treatment could create new microfractures and extend the existing microfractures. Yao [27] conducted cryogenic fracturing experiments to investigate effects of thermal shock on permeabilities of synthetic rock samples (concrete). 20.32 cm (8-inch) rock cubic samples were used in the experiments. Eight tiny thermocouples were embedded in the samples to monitor the temperature changes during the tests. Liquid nitrogen was circulated into the samples through 15.24 cm (6-inch)-deep holes while the samples were subjected to triaxial compression. The results showed that the permeability of the affected area increased. Elwegaa & Emadi [3] and Elwegaa et al. [4,5] conducted several experiments to study the effect of thermal shock on porosity, permeability, and rock mechanical properties of Eagle Ford core samples. The results showed that the porosity and permeability of the core samples were enhanced due to creating new fractures and/or extending the existing ones. Furthermore, implementing the thermal shock technique on the core samples altered their Young's moduli and Poisson's ratios and increased their brittleness indices. Carpenter [2] concluded that cryogenic fracturing is a stimulation technology that deploys cryogenic fluids to fracture unconventional oil and gas reservoirs.

Effects of injecting cold nitrogen in conjunction with CGI method on oil recovery of the Eagle Ford shale oil reservoir was examined in this study. Since Eagle Ford is one of the largest unconventional resources in the U.S., core plugs from its outcrop were obtained and used in this study. Additionally, nitrogen was used as the injection fluid due to its availability, low cost, and successful implementation in EOR operations. Nitrogen was injected into four core samples at different pressures and temperatures and the oil RF at the end of each test was calculated. The results were then analyzed to assess effects of injecting pressure and temperature on the oil RF. Computed Tomography (CT)

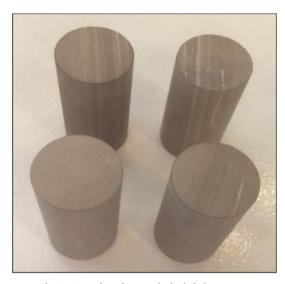


Fig. 3. Core plugs from eagle ford shale outcrop.

scanner was used to scan the core samples prior to and after conducting the experiments.

2. Core preparation and saturation

Four core plugs were used in this experimental study (Fig. 3). The cores were cut from Eagle Ford outcrop parallel to the bedding planes, all 3.81 cm (1.5") in diameter and 7.62 cm (3") in length. First, the cores were numbered and placed in a vacuum oven dryer to evaporate all water present in the pore spaces. Since Eagle Ford shale formation has very low permeabilities, the cores were saturated at 6.9 MPa (1000 psi) pressure.

The apparatus used to saturate the core samples comprised of an ISCO syringe pump, a vacuum pump, a stainless-steel high-pressure vessel, Soltrol 130 mineral oil, and a pressure gauge (Fig. 4). The core saturation process was conducted in the following steps:

- The core plugs were named (EF#1, EF#2, EF#3 and EF#4) and their dry weights were measured using a lab balance scale.
- Core plug was placed in the vessel; the vessel was secured.
- The vacuum pump was turned on for 24 h to evacuate the air from

the pore spaces.

- The syringe pump was turned on to saturate the core sample for 24 h.
- The pump was turned off and the pressure was bled gradually to avoid creating any crack inside the core plug.
- The sample was kept in the vessel for 24 h till its internal and external pressures reach equilibrium.
- The sample was removed from the vessel and its saturated weight was measured using the lab scale.
- At this point, the sample was saturated and ready for the cold nitrogen injection experiment.

3. Cyclic cold nitrogen injection experiment

First, the core plugs were heated to the reservoir temperature (82 °C (180 °F)). Next, nitrogen was injected into the samples at different pressures (6.9 MPa (1000 psi), 10.3 MPa (1500 psi), 13.8 MPa (2000 psi), and 20.7 MPa (3000 psi)) and temperatures ($-26\,^{\circ}\text{C}$ ($-15\,^{\circ}\text{F}$), $-18\,^{\circ}\text{C}$ (0 °F), 0 °C (32 °F), and 23 °C (74 °F)). Fig. 5 illustrates the design of the experiment.

The apparatus used to conduct the experiment composed of a Quizix pump (QX-6000), two high-pressure stainless-steel accumulators, a high-pressure stainless-steel core holder, a high-pressure nitrogen gas cylinder, a plastic cooling bath, a high-temperature heating jacket ($T_{max} = 121\,^{\circ}\text{C}$ (250 °F)), hydraulic oil, insulation tape, pressure gauges, and a fresh water tank. Fig. 6 presents the schematic diagram of the setup.

The hydraulic oil used in the experiment has minimum working temperature of -55 °C (-67 °F), which does not freeze during the tests. Table 1 illustrates the specifications of the hydraulic oil.

Ice, Sodium Chloride (NaCl) crystals, and Calcium Chloride Hexahydrate (CaCl₂.6H₂O) were used to obtain the desired temperatures of the cooling bath. Table 2 shows the salt-ice cooling mixtures.

3.1. Experimental procedure

At the beginning of the test, all tubing and fittings were connected and sufficiently tightened to prevent any gas leakage from happening. The experimental procedure is presented below:

- The saturated core plug was placed inside the core holder.
- The core holder was secured and connected to the High-Pressure

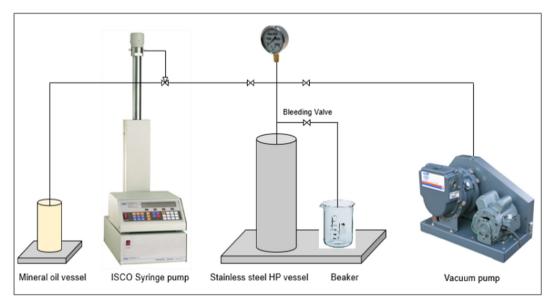


Fig. 4. Schematic diagram of the setup for the saturation experiment.

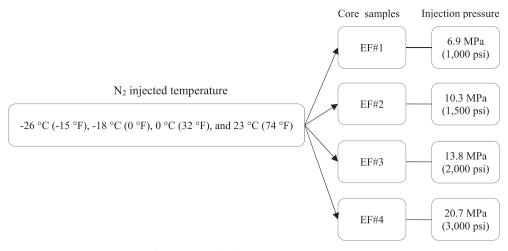


Fig. 5. Design of cyclic gas injection experiment.

(HP) gas accumulator.

- The heating jacket was turned on for one hour to heat the core holder and the core sample to 82 °C (180 °F).
- The cooling bath was prepared by mixing ice with the selected salt to achieve the desired temperature (as shown in Table 2).
- The HP gas accumulator was submerged in the ice-salt cooling mixture.
- The nitrogen cylinder valve was opened to let the gas flow and accumulate in the 2nd HP gas accumulator.
- The gas was left in the accumulator for 30 min to reach temperature equilibrium.
- The valve between the core holder and the gas accumulator was opened, the gas flowed, and the core sample was subjected to the cold nitrogen. This process is called thermal shock.
- The safety pressure for the QX pump was set and pump started operating to reach desired pressure inside the core holder.
- The pump was turned off and the valve between the core holder and the 2nd accumulator was closed for four hours (soaking time).
- The bleeding valve was opened gradually to release pressure inside the core holder.
- Finally, the core was removed from the core holder and placed on the balance scale to monitor and measure its weight in real time. The production period started, and the core weight was measured at the following intervals: 0.5 h, 2 h, 4 h, 8 h, 12 h, 24 h, 48 h, and 72 h.

3.2. Recovery factor (RF) calculation

Weight of the core plugs were measured before saturation, after saturation, and during production. The results were then used to calculate the oil RF using the following equation:

$$Oil RF = \frac{wt_2 - wt_3}{wt_2 - wt_{dry}} \tag{2}$$

where wt_2 is weight of the saturated core plug, wt_3 is weight of core plug at each production time, and wt_{dry} is weight of core plug before the saturation. At the end of this stage, core sample was again scanned using the CT scanner.

4. Brittleness index calculation

Jarvie et al. [13] defined brittleness index based on mineral content of the rock. The following formula is used to calculate the brittleness index based on the volumes content of Quartz, Calcite and Clay.

$$BI = \frac{V_{Quartz}}{V_{Quartz} + V_{Calcite} + V_{Clay}}$$
(3)

Wang and Gale [25] then proposed another definition of brittleness index based on mineral content taking into account Dolomite content and Total Organic Content (TOC) as following.

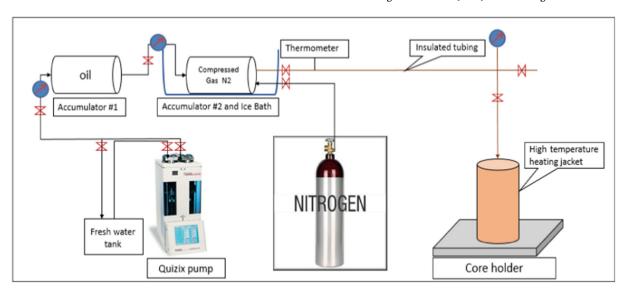


Fig. 6. Schematic diagram of cold fracturing and cyclic gas injection setup.

Table 1 Properties of the hydraulic oil.

Name	Min. operating temp.	Max. operating temp.	Pour point	Specific gravity
Synthetic hydraulic oil	− 55 °C (−67 °F)	266 °C(510 °F)	− 55 °C (−67 °F)	0.826

 Table 2

 Salt-ice mixtures and the resulting temperatures.

No.	Cooling agent	Solvent	Resulting Temperature	Ratio
1. 2. 3.	Ice Ice Ice	Sodium Chloride Calcium Chloride Hexa-hydrate	0 °C (32 °F) -18 °C (0 °F) -26 °C (-15 °F)	About 1–3 (w/w) ratio of salt to ice About 1–0.9 (w/w) ratio of salt to ice

Table 3
Eagle Ford XRD results (mineral content).

Wt.%
14.2
3.0
1.3
81.6

$$BI = \frac{V_{Quartz} + V_{Dolomite}}{V_{Quartz} + V_{Dolomite} + V_{Calcite} + V_{Clay+}V_{TOC}}$$
(4)

X-ray diffraction (XRD) analysis was conducted on the samples and the results are presented in Table 3.

5. Results and discussion

To investigate efficiency of the thermal shock technique via the cyclic injection of nitrogen, cumulative oil RF at each pressure and temperature was calculated. The results demonstrated that decrease in the injection temperature results in an increase in the ultimate oil RF. The results also showed that increase in the injection pressure improves the cumulative oil RF. Also, the results demonstrated that thermal stress resulted from implementing the thermal shock technique plays a crucial role in creating induced fractures. Using equation (1) presented in Section 1.2, magnitudes of the thermal stresses were calculated. In addition, the results of CT scanner depicted that existing cracks in the core samples become wider after implementing the tests at low temperatures (-18 °C (0 °F) and -26 °C (-15 °F)).

5.1. Cumulative oil recovery at constant temperatures

5.1.1. Cumulative oil recovery at ambient temperature (23 $^{\circ}$ C (74 $^{\circ}$ F))

In the first set of experiments, nitrogen was injected into the core samples at the ambient temperature (23 °C (74 °F)). The results (Table 4 and Fig. 7) demonstrate a direct relationship between the injection pressure and cumulative oil RF. The results also show that most of the oil produced during the first 24 h of the production period. At 20.7 MPa (3000 psi), the cumulative oil RF was 7.0% higher than 6.9 MPa (1000 psi) after 30 min of production time and the difference kept increasing and reached 10.57% after 72 h of production.

Table 4 Results of oil RF at 23 °C (74 °F).

44.15
46.89
51.95
54.72

5.1.2. Cumulative oil recovery at 0 °C (32 °F)

Table 5 and Fig. 8 present the results of oil RF at 0 °C (32 °F).

The results demonstrate that injecting nitrogen at 0 $^{\circ}$ C (32 $^{\circ}$ F) results in higher RF than injecting at the ambient temperature. The effect of injection pressure on cumulative RF was consistent with the results observed at the ambient temperature. The results display that cumulative oil recovery at 20.7 MPa (3000 psi) was 14% higher than for 6.9 MPa (1000 psi) after 72 h of production.

5.1.3. Cumulative oil recovery at -18 °C (0 °F)

The production results of the tests conducted at $-18\,^{\circ}\text{C}$ (0 $^{\circ}\text{F}$) are presented in Table 6 and Fig. 9.

The effects of both temperature and injection pressure on the ultimate oil RF are consistent with the previous results. In the other words, increase in injection pressure and decrease in injecting gas temperature improve cumulative oil RF. Increasing injection pressure from 6.9 MPa (1000 psi) to 20.7 MPa (3000 psi), resulted in an increase of 12.65% in the ultimate RF after 72 h of production. The effect of injection pressure on oil RF is noticeable even at the early stages of the production phase. After 30 min of production, oil RF at 20.7 MPa (3000 psi) injection pressure is 9.34% higher than recovery factor at 6.9 MPa (1000 psi). Decrease in temperature from 23 °C (74 °F) down to -18 °C (0 °F) at the constant injection pressure of 20.7 MPa (3000 psi), resulted in an increase of 7.83% in ultimate oil RF.

The results also show that cracks were observed in all four core samples after finishing the tests at $-18\,^{\circ}\text{C}$ (0 °F). Fig. 10 shows the core sample EF#1 before and after conducting the test. A crack, which has a positive contribution to oil production, was patently created in the core sample after injecting the gas at $-18\,^{\circ}\text{C}$ (0 °F).

Fig. 11 shows the CT scan results of core sample EF#4 before and after conducting the thermal shock technique. The figure clearly shows that conducting thermal shock caused an increase in the crack width.

5.1.4. Cumulative oil recovery at $-26 \,^{\circ}\text{C}$ ($-15 \,^{\circ}\text{F}$)

Table 7 and Fig. 12 present the results of oil production at -26 °C (-15 °F)

In light of injection pressure, the results demonstrate that injecting gas at 20.7 MPa (3000 psi) resulted in the highest oil RF. The results also show that decrease in the temperature of the injection gas results in an increase in the oil RF.

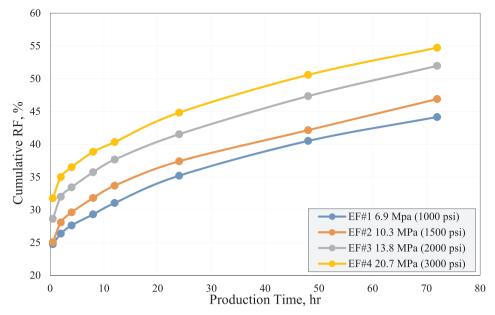


Fig. 7. Results of oil RF at 23 °C (74 °F).

Table 5Results of oil RF at 0 °C (32 °F).

Core no.	Inj. Pressure, MPa (psi)	Production Time, hour	0.5	2	4	8	12	24	48	72
EF#1	6.9 (1000)	RF, %	29.60	31.69	32.46	34.00	34.98	37.89	42.18	45.30
EF#2	10.3 (1500)		29.92	32.33	33.30	34.91	36.19	39.75	45.94	49.52
EF#3	13.8 (2000)		31.22	34.72	36.07	38.12	39.57	43.85	49.19	52.53
EF#4	20.7 (3000)		33.03	36.94	38.30	40.45	42.15	46.80	54.40	59.32

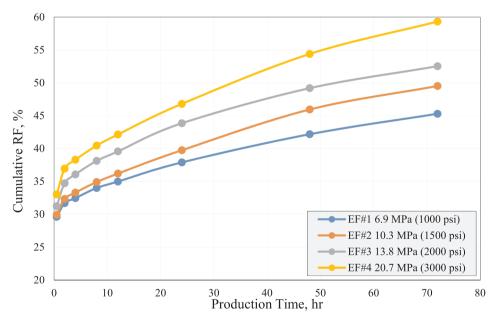


Fig. 8. Results of oil RF at 0 $^{\circ}$ C (32 $^{\circ}$ F).

Table 6 Results of oil RF at -18 °C (0 °F).

Core no.	Inj. Pressure, MPa (psi)	Production Time, hour	0.5	2	4	8	12	24	48	72
EF#1	6.9 (1000)	RF, %	24.24	27.87	29.51	31.53	33.54	39.15	45.76	49.90
EF#2	10.3 (1500)		26.27	29.95	32.48	34.39	36.30	41.16	47.25	51.45
EF#3	13.8 (2000)		29.61	35.11	37.41	40.43	42.74	48.75	56.10	60.68
EF#4	20.7 (3000)		33.58	38.29	39.99	42.90	44.97	50.55	57.60	62.55

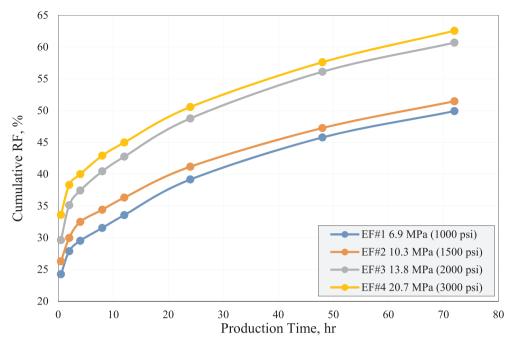


Fig. 9. Results of oil RF at -18 °C (0 °F).

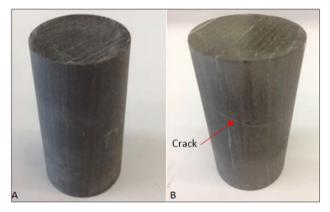
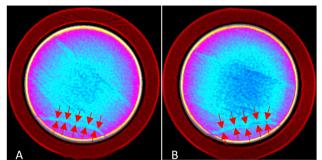


Fig. 10. Core plug EF#1: A) Before thermal shock. B) After thermal shock.



shock.

Fig. 11. CT scan results of EF#4 A. before thermal shock, B. after thermal

5.2. Cumulative oil recovery at constant injecting pressures

Fig. 13, Fig. 14, Fig. 15, and Fig. 16 present the results of cumulative recovery factors at constant pressures (6.9 MPa (1000 psi), 10.3 MPa (1500 psi), 13.8 MPa (2000 psi), and 20.7 MPa (3000 psi)). The results indicate that decrease in the injection temperature increases the ultimate oil RF at any injecting pressure. The temperature difference between the core holder and the injected gas results in an additional pressure increase inside the core holder, which enhances the oil RF. It also results in a thermal shock, which extends the existing fractures inside the core sample (Fig. 11) and consequently, improves the oil RF.

5.3. Increase in oil RF

Fig. 17 depicts ultimate oil RF at each pressure and temperature. The results patently demonstrate that there is a direct relationship between the oil RF and the injecting pressure. It is also clear that lowering the temperature of the injecting gas results in an increase in the oil RF owing to creating new cracks and/or extending the existing ones and increasing the pressure inside the core holder by 689-1379 KPa (100-200 psi) than the target pressure.

It is worth noting that the least increase in RF was observed when the temperature decreases from $-18\,^{\circ}\text{C}$ (0 °F) to $-26\,^{\circ}\text{C}$ (-15 °F) owing to a relatively slight change in temperature compared to the other cases.

5.4. Thermal stress results

At each temperature, thermal stress for each core sample was calculated using equation (1) and the rock properties presented in Table 8.

Table 7	
Results of oil recovery factor at	$-26 ^{\circ}\text{C} (-15 ^{\circ}\text{F}).$

Core no.	Inj. Pressure, MPa (psi)	Production Time, hour	0.5	2	4	8	12	24	48	72
EF#1	6.9 (1000)	RF, %	23.31	27.36	29.38	31.62	33.73	39.44	46.21	50.71
EF#2	10.3 (1500)		28.42	33.06	34.91	37.54	39.55	45.30	52.24	56.96
EF#3	13.8 (2000)		33.25	37.99	39.72	42.24	44.76	50.44	57.44	62.03
EF#4	20.7 (3000)		38.13	41.25	42.72	45.01	47.27	52.32	58.11	63.07

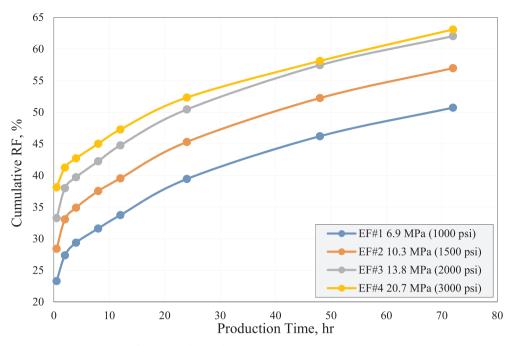


Fig. 12. Results of oil recovery factor at -26 °C (-15 °F).

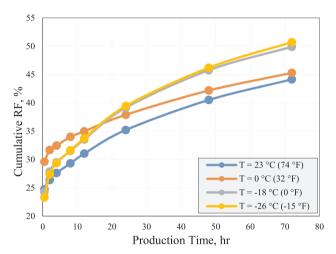


Fig. 13. Results of cumulative RF of EF#1 at 6.9 MPa (1000 psi).

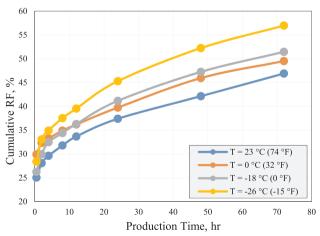


Fig. 14. Results of cumulative RF of EF#2 at 10.3 MPa (1500 psi).

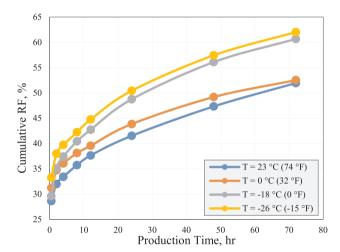


Fig. 15. Results of cumulative RF of EF#3 at 13.8 MPa (2000 psi).

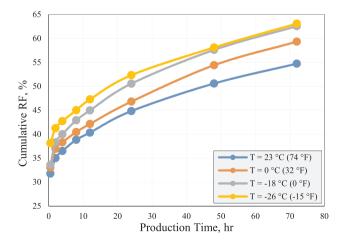
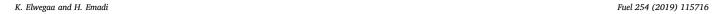


Fig. 16. Results of cumulative RF of EF#4 at 20.7 MPa (3000 psi).



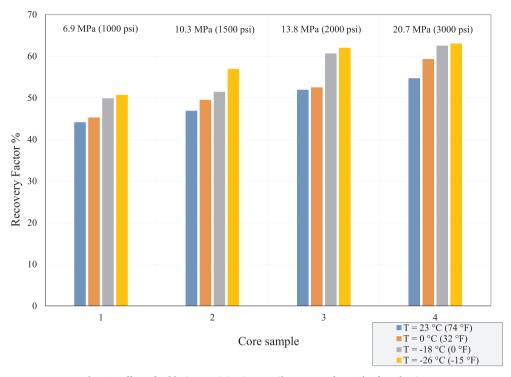


Fig. 17. Effect of cold nitrogen injection on oil recovery after 72 h of production.

Table 8
Core sample properties.

Core sample	Young's modulus (E), GPa (psi×10 ⁶)	Poisson's ratio (v)	Thermal expansion coefficient (α), K^{-1}	Thermal expansion coefficient (α), F^{-1}
EF#1	38.61 (5.60)	0.27	1×10 ⁻⁵	5.55×10 ⁻⁶
EF#2	41.78 (6.06)	0.26		
EF#3	37.44 (5.43)	0.25		
EF#4	36.20 (5.25)	0.27		

Table 9
Thermal stress results.

Initial Temperature (T _i), °C (°F)	Injection Temperature (Ts), °C (°F)	ΔT, °C (°F)	$\sigma_{ m thermal}$, psi	$\sigma_{ m thermal}$, MPa
82 (180)	23 (74) 0 (32)	59 (106) 82 (148)	4250 5824	29 40
	-18 (0)	100 (180)	7083	49
	-26 (-15)	108 (195)	7674	53

Dynamic Young's moduli and Poisson's ratios were obtained from ultrasonic velocity test measurements, which were conducted on Eagle Ford core sample from the previous experimental work [4].

Thermal stresses applied to the samples were calculated at each temperature difference (ΔT) and the results are presented in Table 9. The results demonstrate that increase in temperature difference between the injected gas and the rock sample results in an increase in the thermal stress. Thus, injecting nitrogen at lower temperatures causes an increase in the magnitude of the thermal stress, which results in creating new cracks and/or extending existing cracks in the core sample.

5.5. Brittleness index results

Using the mineral content of the core samples and equations (3) and (4), the calculated brittleness index of the core samples is 14, which seems too low due to high volume content of Calcite and low volume of Quartz. Using Rickman's formula, calculated brittleness index was 60, which is similar to the previous studies [11].

6. Conclusions

Nitrogen was injected into four core samples taken from Eagle Ford reservoir at four different pressures and temperatures. Effects of injecting pressure and temperature on oil RF were investigated.

- The results of this experimental study demonstrate that injecting cold nitrogen via cyclic gas injection could be used as an Improved Oil Recovery (IOR) method in shale oil reservoirs.
- The results also demonstrate that injecting cold nitrogen causes a
 pressure increase around the core sample resulting in higher oil RF.
- The results also showed that increase in the injection pressure improves the cumulative recovery factor. For instance; at the lowest gas temperature of $-26\,^{\circ}\text{C}$ ($-15\,^{\circ}\text{F}$), the oil recovery was 13% at 20.7 MPa (3000 psi) higher than at 6.9 MPa (1000 psi).
- The CT scan results clearly show that injecting cold nitrogen caused an increase in the width of the existing fracture resulting in higher fracture conductivity and oil RF.
- The results show that injecting nitrogen at $-26\,^{\circ}\text{C}$ (-15 $^{\circ}\text{F}$)

- increases oil RF by up to 10% than injecting nitrogen at the ambient temperature (23 $^{\circ}$ C (74 $^{\circ}$ F)).
- The thermal stress results demonstrate that increase in temperature difference between the injected gas and the rock sample results in an increase in the magnitude of the thermal stress. Injecting cold nitrogen at −26 °C (−15 °F) into hot formations results in applying thermal stress of 53 MPa (7674 psi) to the core samples leading to creating new cracks and/or extending the existing ones.

References

- Alqahtani N. Experimental study and finite element modeling of cryogenic fracturing in unconventional reservoirs. Golden, Colorado, USA: PhD Dissertation, Petroleum Engineering Department, Colorado School of Mines, 2015.
- [2] Carpenter C. Cryogenic-fracturing treatment of synthetic-rock with liquid nitrogen. J Petrol Technol 2017:70–1.
- [3] Elwegaa K, Emadi H. The effect of thermal shocking with nitrogen gas on the porosities, permeabilities, and rock mechanical properties of unconventional reservoirs. Energies 2018:11(8):2131.
- [4] Elwegaa K, Emadi H, Ramezanian M. Investigating effects of thermal shock technique on unconventional reservoir rock mechanical properties. New York, NY, USA, In: Proceedings of the 53rd US rock mechanics/geomechanics symposium, 2019.
- [5] Elwegaa K, et al. Improving oil recovery from shale oil reservoirs using cyclic cold carbon dioxide injection – an experimental study. Fuel 2019;254.
- [6] Enayatpour S, Patzek T. Thermal Shock in Reservoir Rock Enhances the Hydraulic Fracturing of Gas Shales. Denver, Colorado, USA, Unconventional Resources Technology Conference, 2013.
- [7] Gamadi T, Sheng J, Soliman M. An experimental study of cyclic gas injection to improve shale oil recovery. New Orleans, Louisiana USA, SPE-166334-MS, 2013.
- [8] Geyer JF, Nemat-Nasser S. Experimental investigation of thermally induced interacting cracks in brittle solids. Int J Solids Struct 1982;18(4):349–56.
- [9] Gutierrez G et al. Improvements in multistage fracturing, Remolino Field, Mexico. The Woodlands, Texas, USA, Society of Petroleum Engineers, 2014.
- [10] Hamdi Z, Awang M, Moradi B. Low temperature carbon dioxide injection in high temperature oil reservoirs. Kuala Lumpur, Malaysia, International petroleum technology conference, 2014.
- [11] Hu Y. et al. A novel model of brittleness index for shale gas reservoirs: confining pressure effect. Brisbane, Australia, In: Proceedings of the SPE Asia Pacific unconventional resources conference and exhibition, 2015.
- [12] Janszen M, Bakker T, Zitha PLJ. Hydraulic fracturing in the Dutch Posedonia Shale.

- Budapest, Hungary: Society of Petroleum Engineers; 2015.
- [13] Jarvie DM, Hill RJ, Ruble TE, Pollastro RM. Unconventional shale-gas systems: the Mississippian Barnett Shale of North-Central Texas as one model for thermogenic shale-gas assessment. AAPG Bull 2007;91:475–99.
- [14] Kim K, Kemeny J. Effect of Thermal Shock and Rapid Unloading on Mechanical Rock Properties. Asheville, North Carolina, USA, ARMA-09-084, 2009.
- [15] Kumar D, Gutierrez M. Effects of Temperature on Two-Dimensional Hydraulic Fracturing in Impermeable Rocks. San Francisco, California, USA, ARMA 11-220, 2011.
- [16] Langedijk RA et al. Optimization of Hydraulic Fracturing in a Deep, Multilayered, Gas-Condensate Reservoir. Dallas, Texas, USA, Society of Petroleum Engineers, 2000
- [17] Mansour A, Gamadi T, Emadibaladehi H, Watson M. Limitation of EOR applications in tight oil formation. Kuwait city, Kuwait: Society of Petroleum Engineering; 2017.
- [18] Mansour AG, Khalil R, Gamadi T. Compositional simulation evaluation of miscible gas injection performance in tight oil formation. Society of Petroleum Engineering: Bakersfield, California, USA; 2017.
- [19] Mohammed-Singh LJ, Singhal AK, Sim S. Screening Criteria for CO2 Huff 'n' Puff; Puff Operations. Tulsa, Oklahoma, USA, SPE/DOE Symposium on Improved Oil Recovery, 2006.
- [20] Morsy S, Sheng J, Soliman MY. Improving hydraulic fracturing of shale formations by acidizing. Pittsburg, Pennsylvania, USA: Society of Petroleum Engineers; 2013.
- [21] Shi R, Schnitzler J, Kantzas A. Preliminary results of enhanced heavy oil recovery by CO₂ cyclic injection. Edmonton: World Heavy Oil Congress; 2008.
- [22] Song C, Yang D. Performance evaluation of CO2 huff-n-puff processes in tight oil formations. Calgary, Alberta Canada, SPE-167217-MS, 2013.
- [23] Tarasovs S, Ghassemi A. Propagation of a system of cracks under thermal stress. San Francisco, California, USA, ARMA 11-558, 2011.
- [24] Tran D, Settari A, Nghiem L. Initiation and propagation of secondary cracks in thermo-poroelastic media. Chicago, Illinois, USA, ARMA 12-252, 2012.
- [25] Wang F, Gale JFW. Screening criteria for shale-gas systems. Gulf Coast Assoc. Geol. Soc. Trans. 2009;59:779–93.
- [26] Wan T, Sheng J, Soliman M. Evaluate EOR potential in fractured shale oil reservoirs by cyclic gas injection. Denver, Colorado, USA, SPE-168880-MS, 2013.
- [27] Yao B et al. Experimental study and modeling of cryogenic fracturing treatment of synthetic rock samples using liquid nitrogen under tri-axial stresses. Calgary, Alberta, Canada, SPE-185050-MS, 2017.
- [28] Yu Y, Sheng J. An experimental investigation of effect of pressure depletion rate on oil recovery from shale cores by cyclic N2 injection. San Antonio, Texas, USA, SPE-178494-MS, 2015.
- [29] Zhao B, Zhang G, Lin Q. The application of cryogenic treatment during refracture process – laboratory studies. Houston, Texas, USA, ARMA 16-552, 2016.