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RESEARCH PAPER

Experimental Investigation and Modelling of Asphaltene Precipitation during Gas Injection

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Abstract

Due to the limited crude oil resources, the role of enhanced oil recovery (EOR) techniques in the production of the oil that has not been extracted during the primary and secondary oil production techniques is crucial. Gas injection is known as an important EOR technology, but one of the main concerns during gas injection is asphaltene precipitation and deposition within reservoir formation. In this study, the effect of temperature (ranges 376-416 K) and concentration of injected gas (N_2 (10, 20 and 40, mole percent) and first separator gas (20, 40 and 60, mole percent)) on the onset pressures and amount of asphaltene precipitation in one of the Iranian oil reservoirs were investigated. Two series of experiments were accomplished on live oil by gravimetric method; first: injection of different concentrations of nitrogen and first separator gas at reservoir temperature and under different pressures (3000-8000 psia) and second: natural depletion at different temperatures. Besides, the experimental data of asphaltene precipitation due to N_2, first separator gas, and also CO_2 injection were compared together. Finally, the experimental data were modeled with a solid model. The results indicate that the amount of asphaltene precipitation due to N_2 injection (0.1-0.2 wt %) is lower than the first separator gas and CO_2 injection at the same concentration. Experiments show that in the range of experimental temperatures the asphaltene precipitation changes up to 0.06 wt %. For pressures below the bubble pressure (~ 4700 psi), precipitation changes directly with temperature, and indirect relation is observed for pressures above the bubble point pressure.

Keywords:
Asphaltene Precipitation,
Gas Injection,
Natural Depletion,
Solid Model,
Temperature

Introduction

In oil industries, enhanced oil recovery (EOR) techniques are used for improving oil recovery and producing reservoirs of residual oil that can’t be recovered through primary and secondary processes. Among different EOR techniques, gas injection is an increasingly widespread technology used to increase oil production. The most common gases for the EOR technique are nitrogen, carbon dioxide, and natural gas. N_2 injection is an effective EOR technology in oilfield reservoirs [1-2]. The past studies proved that nitrogen injection could recover up to 45-90% of initial reserves [3]. It is important to state that when solvents are used in EOR processes, asphaltene deposition can occur in the field [4].

Asphaltenes are the heaviest and the most polar fraction of crude oil, typically soluble in some substances such as aromatics (e.g., benzene, toluene, and pyridine), and insoluble in some others such as n-alkanes (e.g., n-pentane). Furthermore, it is the most complicated fraction which contains more than 100,000 different molecules [5-8]. The chemical structure of asphaltene includes recondensed aromatic rings and aliphatic chains that are combined in different ways, forming

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molecules with a wide range of molecular weight values [9-10]. Thus there has been widespread debate on its characteristics such as molecular weight. There are many reviews and experimental works on asphaltene molecular weight. The asphaltene molecular weight presented by different authors has a range of 500 to 1000 (gr/mole) which depends on several parameters such as, oil and its origin, nature of the solvent, the solution temperature, and the measurement method [11]. However, the most used value is reported by Mullins et al. [12] and Soleymanzadeh et al. [13] to be around 750 g/mole. Also, asphaltene can form aggregates with a molecular weight distribution of 10^3 to 10^5 [7].

Asphaltene is a matter which tends to cause some issues during crude oil production, transport, and refinement [14]. Asphaltene precipitation and deposition cause severe operational, economic, and environmental problems to the petroleum industry. Some of these problems include serious damages around the wellbore and reservoir by reduction of permeability and oil recovery via wettability alteration. They also cause plugging the pores with an additional pressure drop in wellbore tubing. In another word, Asphaltene deposition can reduce flow diffusivity and cause fouling problems during transportation and processing [8-9,15-16].

In the literature, controversial results have been reported for the effect of temperature on the amount of asphaltene precipitation. Mohammadi et al. [17] found that the depressurization process at higher temperatures results in higher asphaltene onset pressure or earlier formation of asphaltene. Mahmoudi and Zare-Reisabadi [18] showed that the solubility of asphaltene increases with temperature increasing. Santos et al. [15] showed that temperature has a small influence on asphaltene precipitation onset pressure and solubility parameter of the oil.

In this study, the effect of temperature and concentration of injected gas on the onset pressures and amount of asphaltene precipitation in one of the Iranian oil reservoirs were investigated. Two series of experiments were accomplished. The first set of experiments were performed at reservoir temperature and under different pressures for different concentrations of nitrogen and first separator gas. Results of these experiments were also compared with results of Ashoori et al. [19] which was done by injection of different mole fractions of CO_2. The second set of experiments were conducted at different temperatures for the natural depletion of reservoir oil. Finally, results were modeled by the solid model.

In the experimental part of this work, the gravimetric method was used to measure the onset and amount of asphaltene precipitation for a live oil sample with *an* isothermal depressurization process in a PVT cell. The accuracy of this method depends on the accuracy of the analytical method and the selection of pressure steps [14].

Experimental

In this study, the gravimetric method was used for gathering experimental data of asphaltene precipitation and onset pressures in live oil samples from an Iranian oil reservoir. For making live oil samples, dead oil from the stock tank was mixed with associated gas from different stages of the production unit (based on the gas-oil ratio (GOR) of reservoir oil at reservoir pressure and temperature). The reservoir oil composition and its properties are represented in Tables 1 and 2.

Experimental PVT cell consists of an air bath, oil sample cylinder, injection gas storage cylinder, and heart of the system as a variable volume and visual equilibrium cell which are schematically shown in Fig. 1. The working pressure and temperature of the system are 10000 psia and 150°C (423.15 K), respectively.

Experimental Procedure for measuring the amount of asphaltene precipitation is as below:

1- The cell of the experimental apparatus was set at the desired temperature.
2- The known volume of the live oil sample was charged to the cell at the reservoir pressure. Note: Temperature must be unchanged during the transfer of oil into the PVT cell.
3- The cell pressure was decreased step by step (1000 psi) from 8000 to 3000 psia.

Note 1: For each step, the sample was mixed for 30 minutes. For stabilization, the sample was left for about 72 hours.

Note 2: At any temperature, asphaltene precipitation would start at a pressure above the bubble point pressure which is named the upper onset pressure (UOP), afterwards, asphaltene precipitation increases with the reduction of pressure until it reaches the bubble point pressure. Near the bubble point and the onset pressures, a smaller pressure interval was used for increasing the accuracy of the experiments.

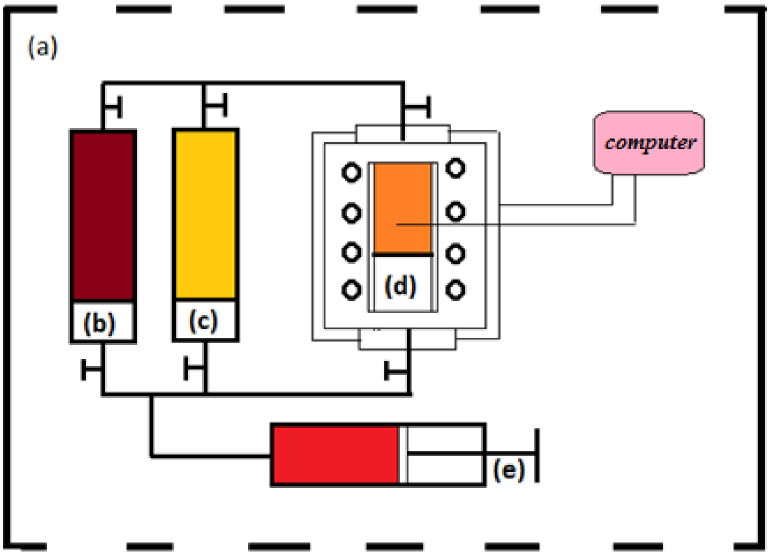


Fig. 1. Schematics of the high pressure – high-temperature asphaltene precipitation system. a) air bath, b) oil sample cylinder, c) injection gas storage cylinder, d) windowed equilibrium cell, e) injection pump

Table 1. Composition of Live Oil

Component	Mole Percent
CO_2	2.31
H_2S	0.65
N_2	0.11
C_1	53.31
C_2	6.60
C_3	3.91
i-C_4	0.66
n-C_4	1.60
i-C_5	0.73
n-C_5	1.06
C_6	2.95
C_7	3.30
C_8	2.85
C_9^+	19.96
Sum	100%

4- A known volume of sample was gathered from the top of the cell.
Note: Below bubble point pressure, liberated gas was removed from the top of the cell, and then, the sample was gathered from the remained liquid oil in the cell.

5- The amount of asphaltene in each sample was determined from titration by normal alkanes (n-C_5 and n-C_6).
Note: To measure the asphaltene content of samples, IP/143 [20] was employed.

6- Asphaltene precipitation amount is the difference of asphaltene content in each step with asphaltene content of reservoir oil.

7- To investigate the effect of gas injection, a predetermined amount of nitrogen or first separator gas with a composition at reservoir pressure (presented in Table 3) was injected into the cell after step 2, and it was mixed for 3 hours. The contaminant of the cell would be liquid in this step. Then for each amount of gas injection concentration, steps 3 to 6 were repeated.
8- To examine the effect of temperature, steps 1 to 6 were repeated for different temperatures (376.15K, 396.15K, and 416.15K).

Table 2. Properties of Reservoir Oil

Properties	Amount
Reservoir Temperature	143°C (416.15K)
Initial Reservoir Pressure	10000 psia
Bubble Point Pressure	4700 psia
Gas Oil Ratio (SCF/STB)	1650
Molecular Weight of Live-oil	69.82
Molecular Weight of Dead oil	197.46
Density of Dead oil (g/cc)	0.827
Wt% of Asphaltene in Dead oil	1.15
Mole% of C_{7+} in Dead oil	85.98

Table 3. Composition of First Separator Gas

Component	Mole Percent
CO_2	3.06
H_2S	0.92
N_2	0.20
C_1	79.63
C_2	7.55
C_3	4.57
i-C_4	0.74
n-C_4	1.57
i-C_5	0.49
n-C_5	0.58
C_6	0.38
C_7	0.24
C_8	0.05
C_9^+	0.02
Sum	100%

Theoretical Procedure

In addition to the experimental investigation, the solid model was used to model asphaltene precipitation during natural depletion and gas injection. A solid model has been used by many authors such as Hajizadeh et al. [19], Zanganeh et al. [22], Ashoori et al. [23], and Nghiem et al. [24]. In the solid model, asphaltene is assumed as a single component in the dense phase (solid or liquid). The base of the solid model is that the fugacity of each component in all phases in the equilibrium is equal and modeled with a cubic equation of state (EOS) [21]. Nghiem (1998) split the heaviest component of the oil phase into non-precipitating and precipitating components and then he considered the precipitating component to be asphaltene [25]. In this work, this assumption of Nghiem (1998) was used and any component which was satisfied in Eq. 1, was assumed to have the probability of precipitation and to be a fraction of precipitating pseudo component (asphaltene). For fugacity calculations, two common equations of state, SRK and PR equations were used.

$$f_i(P,T,z) \geq f_i^{pure}(P,T) \tag{1}$$

Here, precipitated asphaltene was assumed to be a dense liquid phase which was in equilibrium with reservoir fluid which has the following properties: MW equals 750 [12,26] and acentric factor, critical temperature (K) and pressure (bar) is 1.535, 1040, and 15.44, respectively [26]. With knowing these properties of asphaltene, properties of the non-precipitating pseudo components were tuned with experimental data of the bubble point pressure of the live oil at reservoir condition. Also, the binary interaction parameter of asphaltene with lighter hydrocarbons was tuned with experimental data of upper onset pressure (UOP) of the reservoir oil.

Results and discussion

Gas Injection Effect

To investigate the effect of N_2 injection on asphaltene precipitation, different amounts of nitrogen (10, 20, and 40 mole %) were charged into PVT cell at reservoir temperature and pressure. The results of asphaltene precipitation for different amounts of injections are shown in Fig. 2. As shown in this figure, titration by normal pentane shows more asphaltene precipitation rather than using normal hexane. According to Fig. 2, by increasing the injection gas concentration, bubble point pressure would be increased, which means increasing the difference between bubble point pressure and test pressure, so the amount of asphaltene precipitation would be decreased. At low pressures, nitrogen injection reduces the amount of asphaltene precipitation in any concentration. This observation shows that at low pressures, the oil sample cannot dissolve nitrogen; therefore, nitrogen injection leads to the liberation of a part of light components from oil and the heavier remaining oil can dissolve more asphaltene.

The effect of nitrogen injection on asphaltene precipitation at constant pressures and the results of modeling by PR-EOS and SRK-EOS are presented in Fig. 3. According to this figure, for pressures below the bubble point pressure (Figs. 3a and 3b), by increasing concentration of nitrogen, asphaltene precipitation is decreased and finally, for higher concentrations of N_2 injection, precipitation does not happen since lower onset pressure (LOP) lies above the test pressure. This observation shows that at low pressures, the oil sample cannot dissolve nitrogen. Nitrogen injection leads to the liberation of a part of light components from oil and the heavier remaining oil can dissolve more asphaltene.

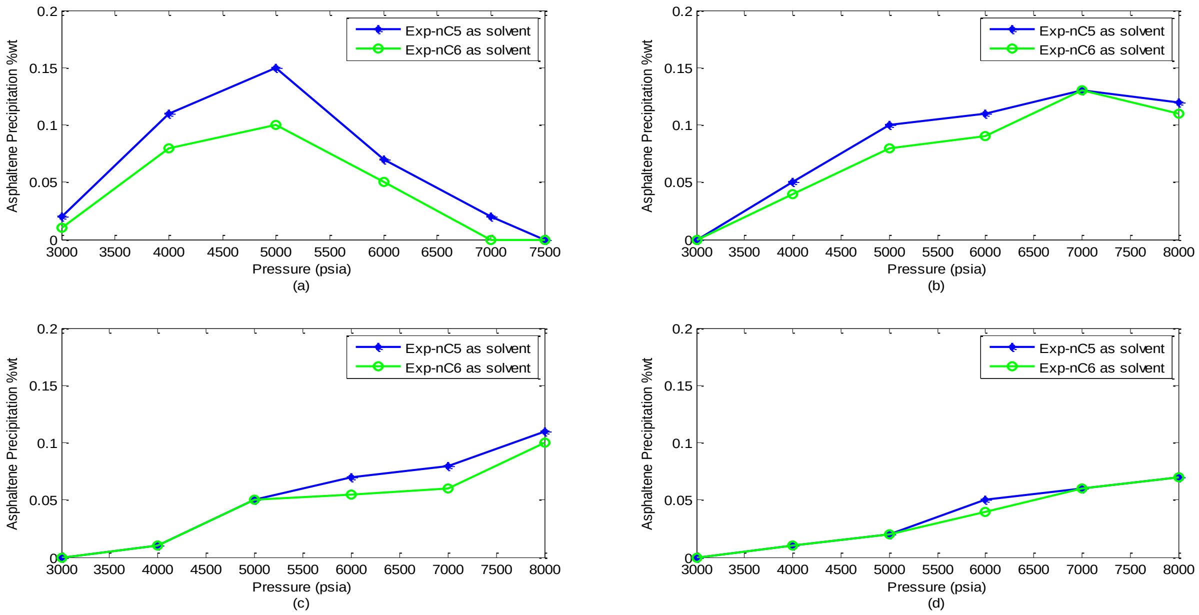


Fig. 2. Asphaltene precipitation vs. pressure at different N_2 concentrations
a) 0%, b) 10%, c) 20%, and d) 40%

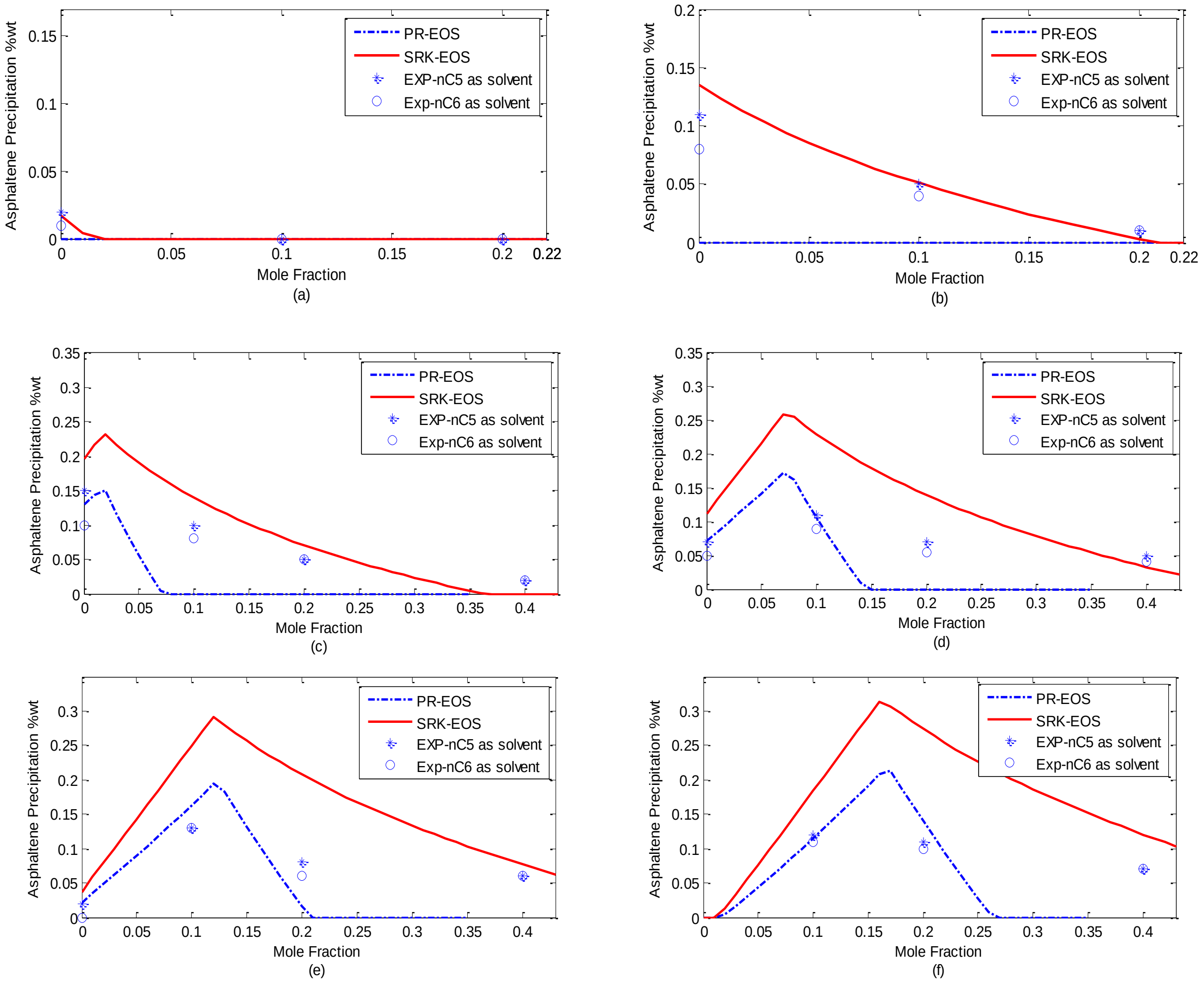
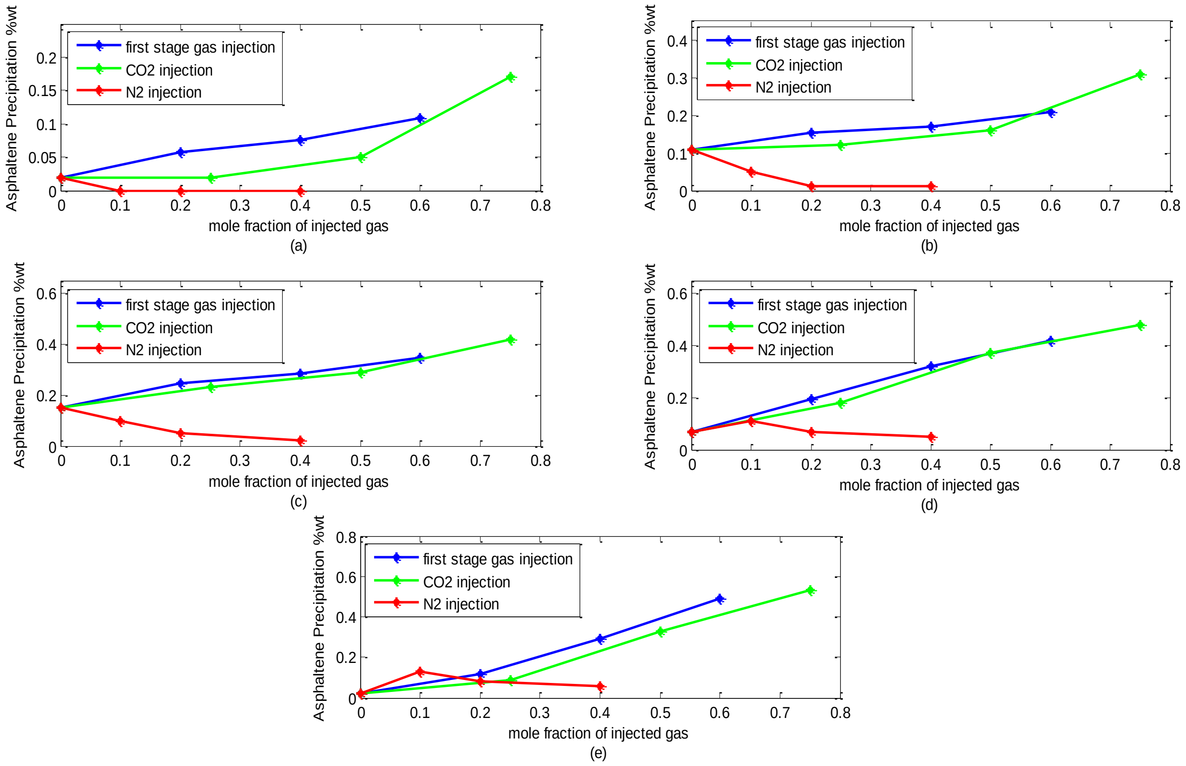


Fig. 3. Asphaltene precipitation vs. mole fraction of injected gas (N_2) at different pressures
a) 3000, b) 4000, c) 5000, d) 6000, e) 7000, and f) 8000 psia

For pressures above 4000 psia (Figs. 3c, 3d, 3e, and 3f), in comparison to previous figures (Figs. 3a and 3b), asphaltene precipitation is high. Due to high pressures, the ability of the oil to dissolve some nitrogen increases and oil becomes lighter and cannot dissolve asphaltene; which results in increasing precipitation amount. But it should be noted that in these pressures, at first, by increasing concentration of nitrogen, asphaltene precipitation increases up to a maximum point, and after that precipitation decreases. The fluid with maximum precipitation refers to the bubble point pressure which is equal to the test pressure.

Also, it is inferred from Fig. 3 that for lower pressures, SRK-EOS shows good accordance with experimental data whereas PR-EOS is better matched with experimental data for higher pressures. To compare the effect of different gas injection on asphaltene precipitation, 20, 40, and 60 mole percent of first separator gas was injected into live oil sample and the amount of precipitation as a result of the injection of this gas and N_2 injection were compared with CO_2 at different pressures. It is noticeable that all experiments in this step were done at reservoir temperature (416.15 K) and the results are depicted in Fig. 4. According to this figure, for all pressures, at the same mole fraction of injected gas, nitrogen injection results in the least precipitation except for test pressure of 7000 psia and 0.1 mole fraction of nitrogen which has the bubble point pressure near 7000 psia. This figure also shows that the first separator gas causes the highest amount of precipitation at approximately all conditions. Therefore, the order of asphaltene precipitation as a result of the injection of different gases is generally as follows: first separator gas > CO_2 > N_2. Some authors also presented similar results. They concluded that the asphaltene will be easily precipitated and deposited by CO_2 injection but N_2 injection will not cause serious asphaltene problems [1].

Injection of gas to the system reduces the heat of vaporization and increases the molar volume of fluid (oil without asphaltene) which leads to a reduction of the solubility parameter. Following this fact and as presented in Fig. 4, injecting the first separator gas and CO_2 into the system will increase the amount of asphaltene precipitation. However, as mentioned before, this pattern is different for nitrogen injection because of the low solubility of nitrogen in the oil sample and the liberation of light components during nitrogen injection at low pressures.



(a)

(b)

(c)

(d)

(e)

Fig. 4. Comparison of different injected gas at different pressures
a) 3000, b) 4000, c) 5000, d) 6000, and e) 7000 psia

The effect of nitrogen injection on bubble point and onset pressures is presented in Figs. 5 and 6, respectively. According to these figures, increasing the mole fraction of the injected gas increases the bubble point and the UOP and it also has very little influence on the LOP. According to Fig. 6, the range of asphaltene precipitation pressures (UOP to LOP) increases with nitrogen injection. This observation was also reported by other authors [1].

Figs. 5 and 6 reveal that both SRK and PR equations of state show a relatively good match with the experimental data of the bubble point and the UOP, but the deviation of the PR equation of state from the LOP is relatively high.

Temperature Effect

To investigate the effect of temperature on asphaltene precipitation, natural depletion of the reservoir oil at three different temperatures (376.15, 396.15, and 416.15 K) was performed. For each temperature and pressure, the asphaltene content of the oil samples was measured at atmospheric pressure by titration with normal pentane. Asphaltene precipitation was calculated according to the initial asphaltene content of the reservoir oil.

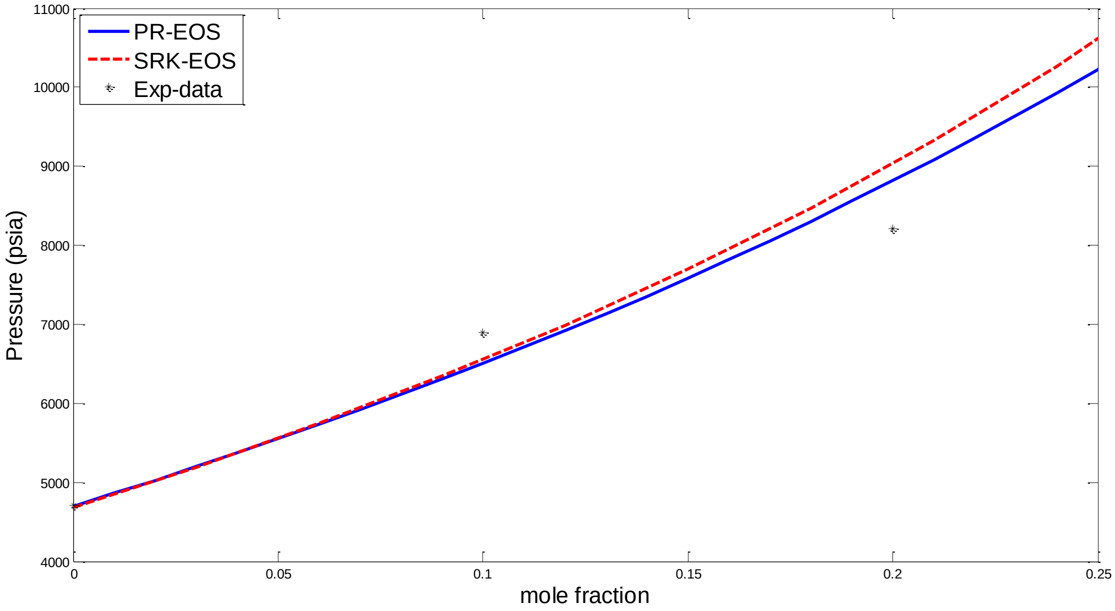


Fig. 5. Bubble point pressure vs. mole fraction of injected gas (N_2) (Measurement of bubble point pressure for nitrogen concentration of 0.4 was not possible due to device working pressure limitation)

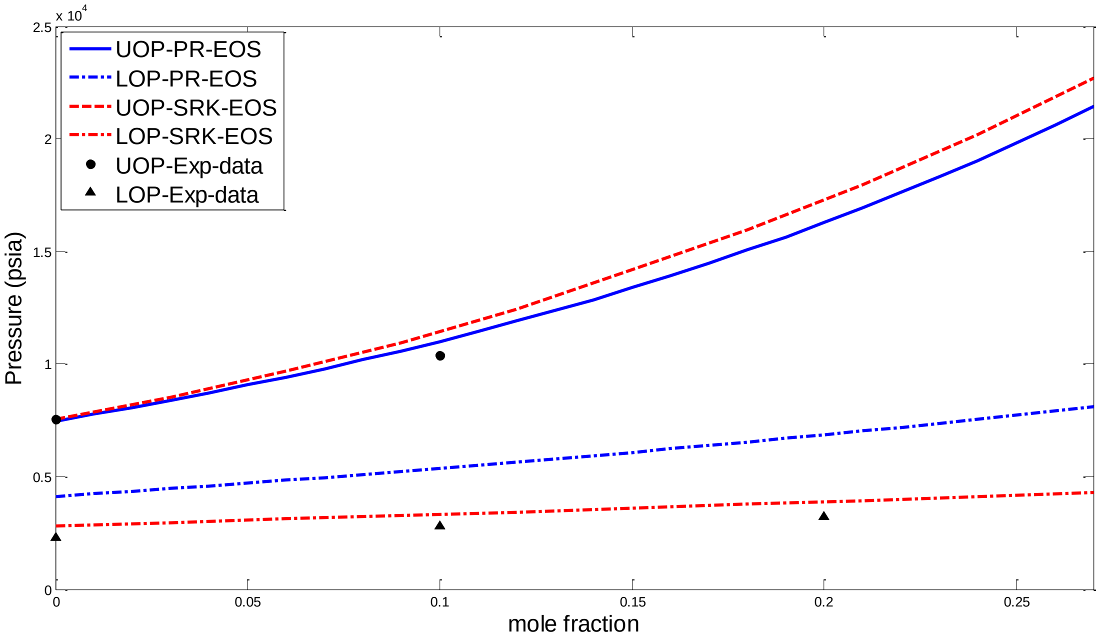


Fig. 6. Onset pressures vs. mole fraction of injected gas (N_2) (Measurement of upper onset pressure for nitrogen concentrations more than 0.1 was not possible due to device working pressure limitation)

A comparison between precipitation data and the results of modeling by PR-EOS and SRK-EOS is shown in Figs. 7 and 8, respectively. The results indicate that upper onset pressure decreases at higher temperatures i.e. the precipitation begins at lower pressures. The results also show that at pressures above the bubble point pressure, asphaltene precipitation decreases with increasing the temperature, but near the LOP, precipitation increases with increasing the temperature. This observation was also mentioned by some authors [9]. However, some other studies reported different results such as the direct relationship between precipitation and temperature at high pressures presented by Mohammadi et al. [17] and reverse relations between them at all pressures reported by Mahmoudi and Zare-Reisabadi [18].

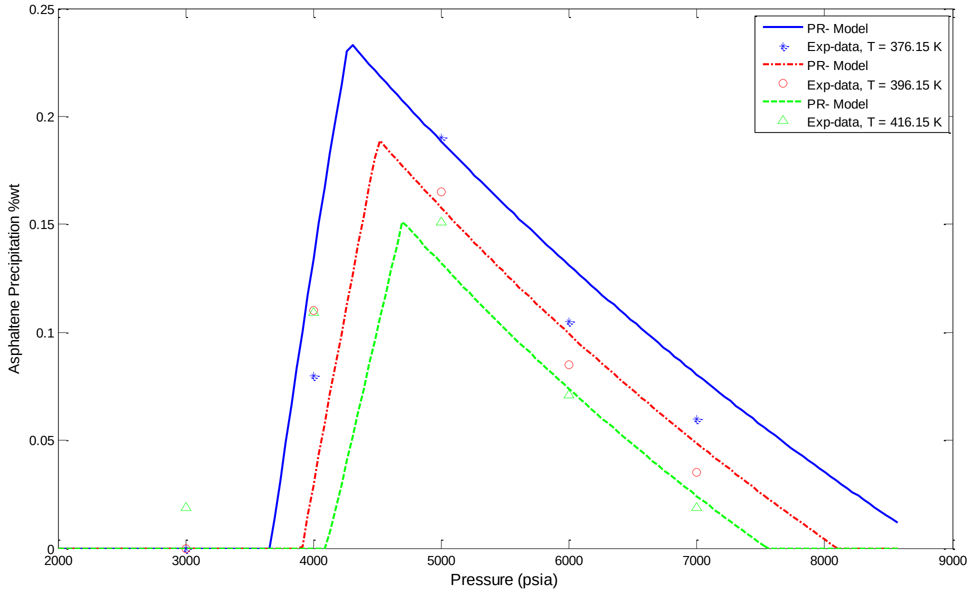


Fig. 7. Asphaltene precipitation vs. pressure; during primary depletion for dtifferent temperatures (experimental data and PR-EOS)

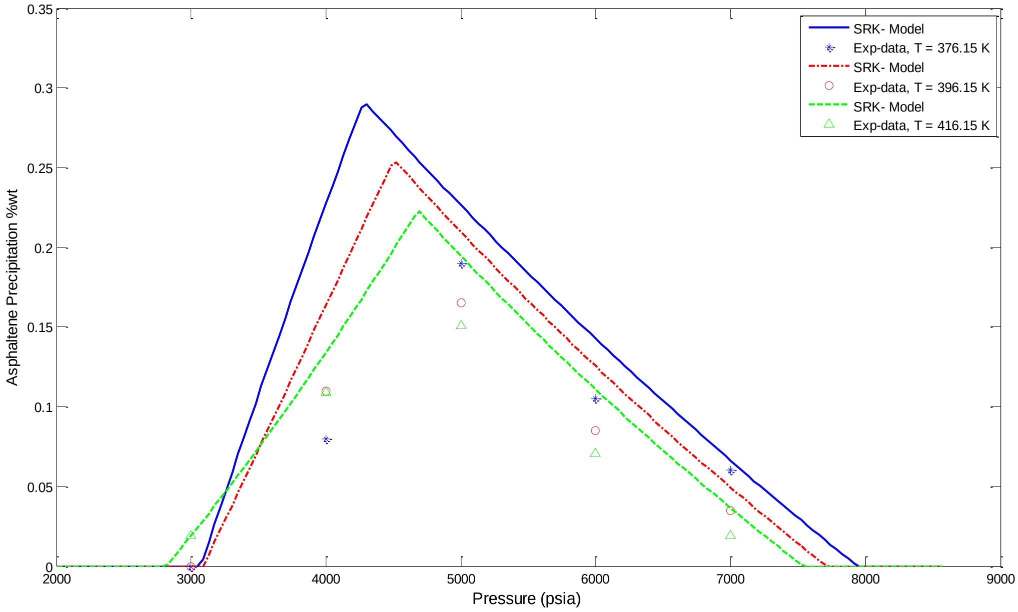


Fig. 8. Asphaltene precipitation vs. pressure; during primary depletion for different temperatures (experimental data and SRK-EOS)

Fig. 7 also shows that for higher pressures, PR-EOS could predict the present experimental data properly, but it is not an appropriate model for predicting the trend and the amount of asphaltene precipitation for pressures near the LOP. As shown in Fig. 8, SRK-EOS overestimates the experimental data for all pressures but it could predict the trend of asphaltene precipitation correctly even for pressures near the LOP. The reason for this observation was investigated by examination of temperature effect on the onset pressures.

Experimental and modeling results of the onset and bubble point pressures for different temperatures are depicted in Fig. 9. As shown in this figure, by increasing the temperature, the bubble point is increased but the UOP and LOP are decreased. This trend is shown by SRK-EOS.

Therefore, higher temperatures have higher precipitation in lower pressures (near LOP). By increasing the temperature, the slower onset pressure increases up to about 390 K, and after that, the process is inversed.

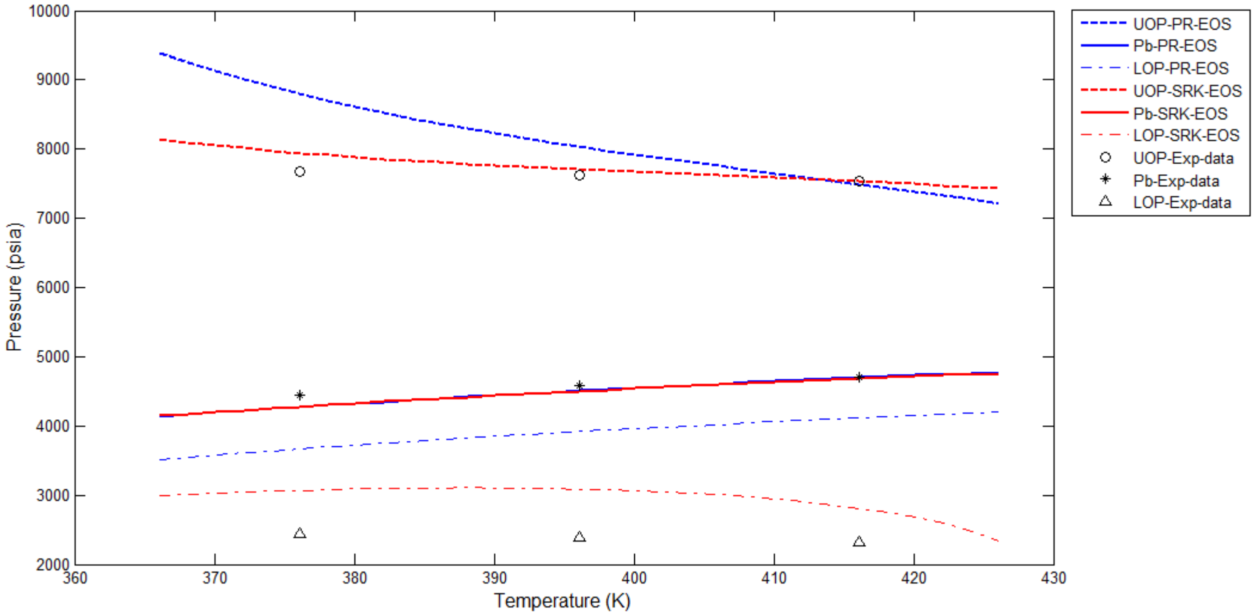


Fig. 9. Effect of temperature on bubble point and onset pressures of reservoir oil

The deviation percent of PR and SRK-EOS data from experimental data is listed in Table 4. According to this table, calculated bubble point pressure by PR and SRK are identical and have about a 2% average deviation from experimental data. For the upper onset pressure, SRK-EOS average deviation is a quarter of the PR-EOS deviation, and for the lower onset pressure, it is half of the PR-EOS deviation.

Table 4. Deviation Percent of Calculated Bubble Point and Onset Pressures by EOS from Experimental Data

Temperature (K)	**SRK**			**PR**		
	Bubble Point Pressure	**Upper Onset Pressure**	**Lower Onset Pressure**	**Bubble Point Pressure**	**Upper Onset Pressure**	**Lower Onset Pressure**
376	3.85	3.38	25.87	3.94	14.48	50.40
396	1.60	1.22	29.66	1.47	5.50	64.54
416	0.56	0.09	21.12	0.25	0.52	43.56
Average	2.00	1.56	25.55	1.89	6.83	52.83

Conclusions

In this work, to investigate the effect of temperature, pressure, and gas injection on asphaltene precipitation in an Iranian oil reservoir, gravimetric method, and modeling by SRK and PR-EOS were used. Following conclusions were extracted from this study.

1- For all pressures in the same mole fraction of injected gas, the order of asphaltene precipitation by injection of different gases is generally as follows: First separator gas > CO_2 > N_2.
2- Increasing the mole fraction of injected gas increases the bubble point and the upper onset pressures and it has very little influence on the LOP.
3- For pressures above the bubble point pressure, asphaltene precipitation decreases with increasing the temperature, but near the LOP, precipitation increases with increasing the temperature.

4- For high pressures, PR-EOS can predict the experimental data properly, but it is not a suitable model for predicting the trend and the amount of asphaltene precipitation in pressures near the LOP.
5- SRK-EOS overestimates the experimental data for all pressures at different temperatures but it could predict the trend of asphaltene precipitation correctly even for pressures near the LOP.
6- By increasing the temperature, the bubble point is increased but the UOP and LOP would be decreased.
7- The effect of temperature on LOP could be predicted by SRK-EOS. By increasing the temperature, the slower onset pressure increases at first, and after that decreases.
8- For different temperatures, SRK-EOS average deviation for UOP is a quarter of PR-EOS deviation and for the LOP, it is half of the PR-EOS deviation.

Nomenclature

f_i	Fugacity of ith component
f_i^{pure}	Fugacity of ith component in pure state
P	Pressure
T	Temperature
z	Mole fraction
LOP	Lower onset pressure
UOP	Upper onset pressure

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