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Asphaltene laboratory assessment of a heavy onshore reservoir during pressure, temperature and composition variations to predict asphaltene onset pressure

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Abstract−An Iranian heavy oil reservoir recently encountered challenges in oil production rate, and further investigation has proven that asphaltene precipitation was the root cause of this problem. In addition, CO_2 gas injection could be an appropriate remedy to enhance the production of heavy crudes. In this study, high pressure-high temperature asphaltene precipitation experiments were performed at different temperatures and pressures to investigate the asphaltene phase behavior during the natural depletion process and CO_2 gas injection. Compositional modeling of experimental data predicted onset points at different temperatures which determine the zone of maximum probability of asphaltene precipitation for the studied heavy oil reservoir. Also, the effect of CO_2 gas injection was investigated as a function of CO_2 concentration and pressure. It was found that a CO_2-oil ratio of 40% is the optimum for limiting precipitation to have the least formation damage and surface instrument contamination.

Keywords: Asphaltene Precipitation, Solubility, CO_2 Gas Injection, Onset Pressure, Heavy Oil

INTRODUCTION

With the appearance of new technologies and the increasing demand for hydrocarbon fuels, the oil industry has tended to use deep reservoirs and heavy crude oils. Deposition of heavy organic compounds, especially the asphaltene component in heavy crude oils, can cause severe and undesirable problems in many industrial processes, including plugging problems, decreasing oil recovery, production and rock wettability alternation [1,2]. Resolving these problems can be very difficult and expensive. These heavy crude compounds are defined as polyaromatics with aliphatic chains composed of Fe, N, V and heteroatoms such as O, N, and S [3]. Asphaltene in crude oil appears as a colloidal particle stabilized by resin. Adding normal alkanes, such as n-Heptane and n-Pentane, weakens the interaction between resin molecules and the asphaltene particles' surface [4]. When asphaltene particles come into contact with each other in areas that are not covered by resin, these particles may attach to each other and start to agglomerate [5,6]. The agglomeration process will cause asphaltene particles to enlarge to a visible size (onset point) based on the technique used.

Precipitation occurs due to change in thermodynamic conditions: pressure and temperature composition. The effect of pressure has been studied in the literature [7-9]; however, it is still not well understood. Some research has shown that asphaltene precipitation increases with temperature [10], while others have found that precipitation is inversely affected by temperature [11,12].

Due to the high interest in gas injection in EOR processes, especially CO_2 injection in heavy oil reservoirs [13,14], it is important to understand the thermodynamic and asphaltene phase behavior of oil at reservoir conditions in the presence of different CO_2 mole percentages with respect to reservoir pressure variation. Modeling of experimental observations is a promising tool to predict the asphaltene precipitation under different conditions.

In oil fields, asphaltene particles will deposit at different conditions. To have a better understanding about asphaltene precipitation in a wide range of conditions, different models have been generated. Asphaltene models are divided into four groups: solubility models, solid models, colloidal models and association equation-of-state (EOS) models [1]. One of the most common solubility models in literature, the Flory-Huggins model, describes asphaltene stability in terms of reversible solution equilibrium [15]. In this model the reservoir oil mixture is divided into liquid and vapor phases by vapor/liquid equilibrium calculations of Soave-Redlich-Kwong EOS [16]. The liquid phase is then split into the asphaltene component and the de-asphaltene fluid. Kawanaka et al. [17] and Cimino et al. [18] developed a precipitation model on the basis of polymer-solution thermodynamics. In solid models, the asphaltene particles are considered as pure solids, while a cubic EOS is used to model the oil and gas phases. Earlier single-component solid model could not model asphaltene precipitation successfully [19]. Nghiem et al. [20] proposed a model by which asphaltene precipitation prediction was improved based on the single component solid model. This model was further refined by Nghiem et al. [21,22]. Leontaritis and Mansoori [23] proposed a model that assumes asphaltene solid particles are stabilized in the oil by resins on their surfaces. In this colloidal model there is an equilibrium between the resins in the oil phase and the resins on the asphaltenes' surfaces. The asphaltene precipitation model proposed by DBR (a Schlumberger company),

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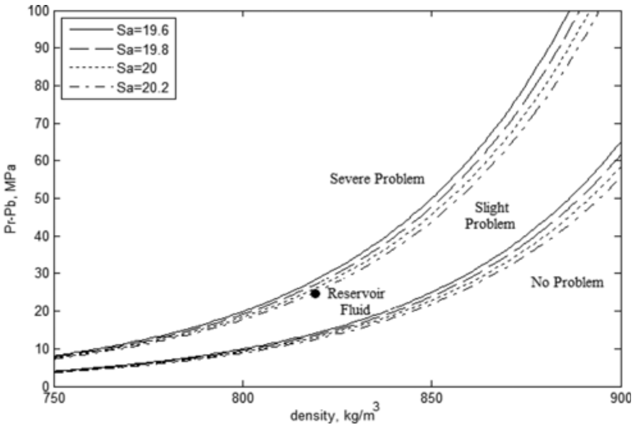

Fig. 1. De boer plot for reservoir sample.

is an association EOS model which makes four main assumptions: (1) Asphaltene molecules are considered as monomers in the bulk crude oil and polymers in the precipitation phase; (2) Asphaltene association leads to asphaltene precipitation; (3) The asphaltene precipitation process is thermodynamically reversible; (4) The asphaltene precipitation phase is a pseudo liquid phase [24].

The purpose of this work was to perform some experiments and then to create a solid model to predict fluid phase behavior at different temperatures, pressures, and compositions.

ASPHALTENE DEPOSITION FEASIBILITY

There are different methods to predict asphaltene deposition. One of the common methods for screening of asphaltene precipitation is the de Boer plot [25], which is based on experimental PVT data. Fig. 1 shows the potential of asphaltene precipitation for oil at reservoir pressure. Equation 1 describes the relation between different parameters of the de Boer plot:

$$(\mathrm{P}_r - \mathrm{P}_b) = \frac{(\Delta \mathrm{S}/\mathrm{S})}{\frac{\mathrm{v}_a}{\mathrm{v}_o}\kappa_o + \frac{2\mathrm{v}_a}{\mathrm{RT}}(\delta_a - \delta_o)\left(\frac{\partial \delta_o}{\partial \mathrm{d}_o}\right)_T \mathrm{d}_o \kappa_o} \qquad (1)$$

SARA analysis (ASTM D4124) was performed to divide the oil into saturate, aromatic, resin, and asphaltene components [26]; the results of the analysis are shown in Table 1. Another asphaltene feasibility method is defined as the ratio of saturates/aromatics (S/A) to asphaltenes/resins (A/R), which was proposed by Stankiewicz et al. [27] and is displayed in Fig. 2. The asphaltene-resin ratio can give a good estimate of asphaltene precipitation potential. Resins are peptizing agents in crude mixtures, and a higher asphaltene-resin ratio means that the crude mixture is more stable. Experiments indicated that if this ratio is higher than 0.35 the crude sample is unstable [28]. For the studied oil sample, the ratio is equal to 0.76. It is seen that, for the de Boer prediction method, the oil sample is slightly in the problem zone, while for other prediction methods it is on the unstable region. All these methods considered that this oil sample would be in high risk zone at the beginning period of production. Therefore, a comprehensive study of asphaltene precipitation behavior is necessary.

Table 1. SARA analysis results

Component	wt%
Saturate	24.8
Aromatic	45.6
Resin	16.8
Asphaltene	12.8

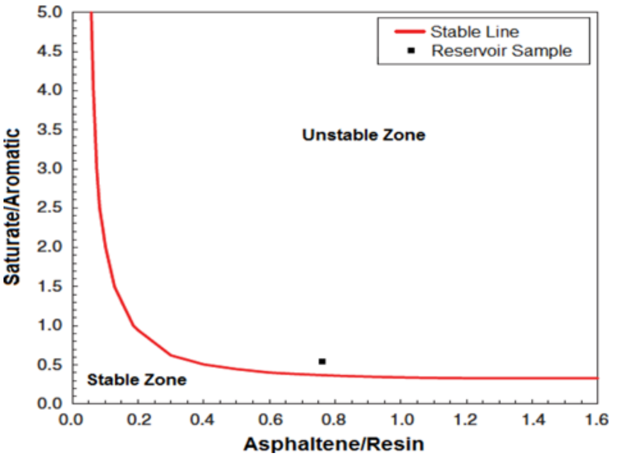

Fig. 2. Saturate/Aromatic vs. Asphaltene/Resin plot for reservoir sample.

EXPERIMENTAL SECTION

1. Fluid Characteristics

Asphaltene precipitation tests were performed on an Iranian heavy oil sample from a reservoir in the southwest region of Iran. Original oil in place (OOIP) is estimated around 28 billion barrels,

Table 2. Composition and properties of crude oil

Components	mole%
N_2	0.39
CO_2	1.74
C_1	20.55
C_2	7.31
C_3	5.34
i-C_4	1.00
n-C_4	3.65
i-C_5	3.10
n-C_5	4.75
C_6	5.48
C_7	3.23
C_8	1.32
C_9	2.27
C_{10}	2.19
C_{11}	1.81
C_{12}+	35.87
Total	100
Molecular weight of C_{12+} fraction	370
Sp.Gr. of C_{12}+ fraction at 60 °F	0.9769

Table 3. Oil field properties

Reservoir name		Asmari-Sarvak
Location		South of Iran
Reservoir depth	[m]	2692-2725
Reservoir temperature	[°C]	96.1
°API degree		20.32
Initial reservoir pressure	[MPa]	30.82
Bubble point pressure	[MPa]	9.88

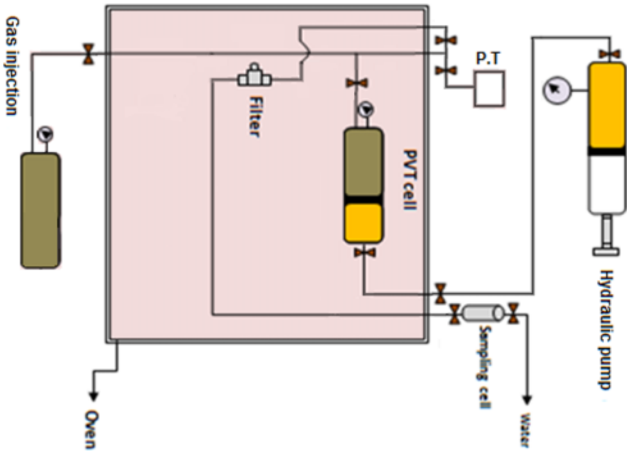

Fig. 3. Experimental setup.

which makes it one of the largest oil fields in the world in terms of oil in place. Compositions of the live oil sample, plus fractions properties, are summarized in Table 2. This reservoir is characterized as a heavy oil reservoir, and the potential of asphaltene precipitation problem is recognized for this reservoir in primary production, especially in the tubing, production lines and surface facilities. The asphaltene content is 12.8 wt%. Table 3 also shows oil field properties directly related to this study.

2. High Pressure-high Temperature Apparatus

A high pressure-high temperature filtration setup was designed to perform natural depletion and CO_2 gas injection tests under reservoir temperature. Fig. 3 shows the experimental setup scheme. The designed setup can give proper results at the range of pressure and temperature up to 41.368 MPa and 148.9 °C. It consists of PVT cell, shaker, hydraulic pump, high pressure metal filter, oven, pressure transducer, high pressure sampler, pressure gauges, and recombination cell apparatus. The most important part of this equipment is the SWAGELOK 0.5 micron metal filter that is appropriate for the separation of asphaltene particle from original fluid sample in PVT cell.

3. Experimental Specification Procedure

We designed a set of experiments, including pressure depletion and CO_2 gas injection tests to study the effect of pressure, temperature, and composition variations on asphaltene precipitation behavior. The live reservoir fluid sample was collected at reservoir pressure in a recombined cell. The recombined cell consists of a moving piston that separates the hydraulic fluid and the live reservoir fluid. The collected reservoir fluid is maintained at or above reservoir pressure and is transported to the laboratory. Experiments were started by transferring live reservoir fluid from the recombined cell into the PVT cell using a digital hydraulic pump at constant pressure. An isobaric fluid transfer was necessary to avoid unwanted asphaltene precipitation along lines and the PVT cell. Also, keeping the fluid at the original conditions at the recombined cell for the next set of experiments is the other part of safe fluid transferring. For natural depletion test, the temperature of the oven was increased to a desirable temperature, and then the PVT cell was shaken for 24 hours to regain reservoir thermodynamic condition. The PVT cell remained at a stationary position for another 24 hours to ensure that the system reached its asphaltene stability and thermodynamic equilibrium. Asphaltene drops out from the original sample, then the oil passed through the filter and it was transferred to the high pressure sampler. The pressure and temperature of the oil were reduced gently and it was transferred to a beaker. Then, the asphaltene content was measured using the IP-143 method (ASTM 6560-00) [29]. Finally, the result was subtracted from the total asphaltene content of the dead oil to measure the asphaltene particle behind the metal filter. After achieving the precipitated asphaltene behind metal filter for the first point, the pressure of the system was lowered to the next desired pressure at a constant temperature and another 24 hours was given to the system to reach the thermodynamic equilibrium and to do the rest of the mentioned steps. This process continued at different pressure steps in a constant temperature to get all needed points in natural depletion test. At each step the metal filter was evacuated and washed to recover entrained fluid and asphaltene particles behind it. To reach a reliable result for each step, a measurement of the IP-143 method was carried out five times on the sample to calculate the exact amount of precipitated asphaltene.

Also, CO_2 gas injection tests were carried out in reservoir conditions. First, a volume amount of CO_2 gas was calculated based on the selected mole ratio of gas and oil. In the second step, CO_2 was transferred from a gas cylinder to PVT cell, and then a calculated oil volume was added to it in the PVT cell. The PVT cell was shaken for 24 hours to achieve maximum interaction of oil components and injected gas. Finally, the cell remained at stationary position for another 24 hours to reach thermodynamic equilibrium. When the pressure gauge stayed at a constant pressure, it meant a fully miscible condition had occurred. Now, the homogeneous mixture of oil and gas was prepared at reservoir condition. Therefore, sampling was done and the test was continued like natural depletion to obtain the amount of asphaltene precipitation behind the filter using the IP-143 standard test.

EXPERIMENTAL RESULTS AND DISCUSSION

1. Pressure Effect

The asphaltene precipitation experimental results for depletion tests at three different temperatures are shown in Table 4 and Fig. 4. Solubility theory is used to describe asphaltene precipitation behavior during the depletion test. According to this theory [30], above the bubble point, pressure reduction causes more expansion in the light end components than in the heavy end components of crude oil and a less aromatic state is being formed. As a result, the density of oil and the asphaltene solubility parameter decreases, and then more asphaltene precipitation is observed. Especially, it is very drastic during pressure depletion until saturation pressure. Maxi-

Table 4. Pressure depletion data

96.1 °C		80 °C		60 °C	
Pressure (MPa)	Precipitated Asph. (wt%)	Pressure (MPa)	Precipitated Asph. (wt%)	Pressure (MPa)	Precipitated Asph. (wt%)
30.33	0.27	30.89	0.51	30.61	0.14
20.69	0.93	20.82	1.13	19.65	0.6
10.69	1.55	10.96	1.61	7.00	0.89
7.03	1.08	6.96	1.74	3.58	0.73
		3.58	1.08		

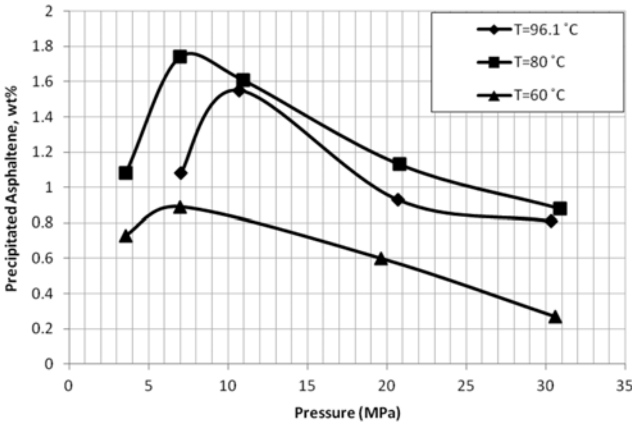

Fig. 4. Experimental results for crude oil.

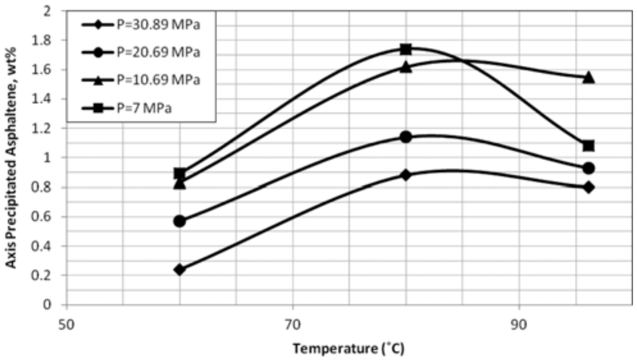

Fig. 5. Effect of temperature on asphaltene precipitation at different pressures.

Table 5. Experimental bubble point pressures at different temperatures

Temperature (°C)	Bubble point pressure (Mpa)
96.1	9.88
80	9.10
60	8.32

mum asphaltene precipitation occurs at bubble point pressure. For this reservoir fluid, CCE tests were carried out at 96.1 °C, 80 °C and 60 °C to obtain the bubble points at these temperatures (Table 5) and to ensure that maximum precipitation happens at these pressures. Below the bubble point, pressure reduction causes solution gas to be liberated from the liquid phase; hence the crude oil becomes richer in heavy end components. Consequently, an increase in the solubility parameter was observed. Results show that the solubility of asphaltene particles is very sensitive to pressure changes under saturation pressure at different temperatures in the objective reservoir.

2. Temperature Effect

The effect of temperature at different points shows the trend of asphaltene precipitation varies under temperature gradient. Fig. 4 shows that the effect of temperature on asphaltene precipitation is considerable. Based on the obtained results, asphaltene precipitation increases from 60 °C to 80 °C and it decreases from 80 °C to 96.1 °C. Since the stabilizing action of the resins works through the mechanism of polar interactions, their effect becomes weaker as the temperature rises, i.e., flocculation will occur as the temperature increases. However, at a specified temperature, solution entropy

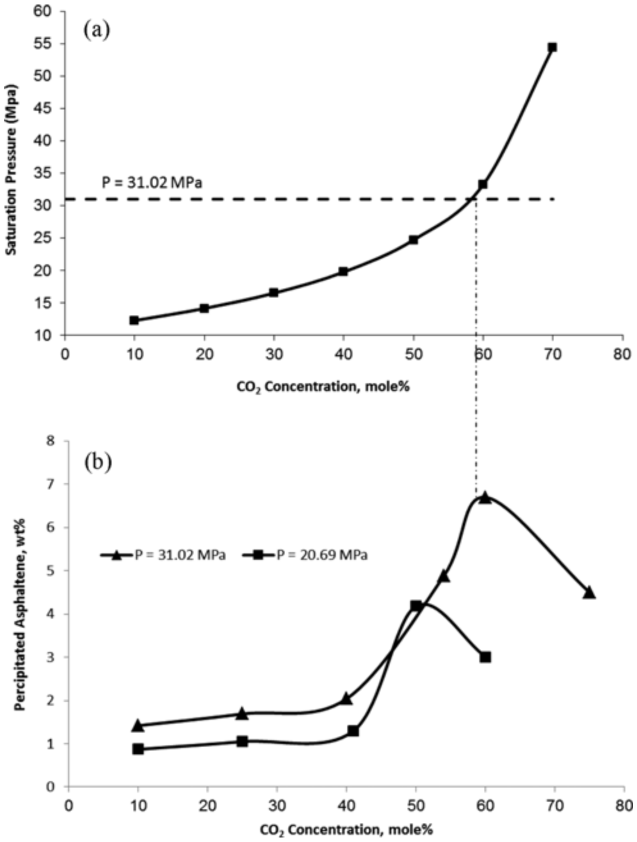

Fig. 6. (a) Saturation pressure of the mixture during CO_2 gas injection, (b) Effect of CO_2 gas injection on asphaltene precipitation.

increases and asphaltene melts and redissolves in the oil as temperature increases.

3. Effect of CO_2 Gas Injection

Fig. 6(b) shows CO_2 gas injection laboratory tests carried out at different CO_2 mole percent and two pressures. By adding more CO_2 into the system, saturation pressure of the mixture increases, as it is shown in Fig. 6(a). The minimum miscibility pressure (MMP) of CO_2 is 26.2 MPa based on the slim tube experiment using reservoir oil. When the pressure of the system is 31.02 MPa, CO_2 is miscible in the fluid and there will be a mass transfer between CO_2 and the fluid; thus, the injected CO_2 gas will mix with the fluid. This behavior continues until the saturation pressure reaches the test pressure. When the test pressure is lower than the saturation pressure, all dissolved gas leaves the mixture and density of the oil increases and as a consequence asphaltene precipitation increases based on the solubility theory. The experiments performed at the set point pressure of 20.69 MPa that is below the MMP, so CO_2 does not mix with oil and there will be a small transfer of light end hydrocarbons into the CO_2. Hence, less precipitation will occur compared to set point pressure of 31.02 MPa. One important fact from this figure is that there is a small increase in asphaltene precipitation when the CO_2 mole percent is lower than 40% in reservoir pressure, which could help for future EOR plans to have less damage during the CO_2 gas injection. The maximum amount of asphaltene that can precipitate out of the fluid is 6.7 wt%, which occurs in the reservoir pressure and 60% of CO_2 mole%.

However, comparing the results of natural depletion and CO_2 gas injection tests, it can be found that asphaltene precipitation is much higher in CO_2 injection tests than in natural depletion. Thus, it is recommended to produce oil in the beginning of a reservoir's life until the reservoir pressure has reached 20.69 MPa, and then inject CO_2 gas to prevent more reservoir pressure decline and less formation and facility damage.

PREDICTING OF ASPHALTENE PRECIPITATION

To predict asphaltene precipitation at different pressure points during natural depletion tests, it is necessary to use an efficient model with reasonable accuracy. The solid model proposed by Nghiem et al. [20] is applied for prediction of the asphaltene precipitation, which is based on the fugacity coefficient. The precipitated asphaltene is represented as a pure solid while the oil and gas phases are modeled with PR EOS with volume shift parameters. The equations for thermodynamic equilibrium of oil, gas and solid are presented in Eqs. (2) and (3):

$$\ln f_{ig} - \ln f_{io} = 0,\ i = 1, \ldots, n_c \tag{2}$$

$$\ln f_g - \ln f_{n,o} = 0 \tag{3}$$

where n_c is the number of components in the oil and gas phases and is considered as an index for asphaltene component, f_{io} and f_{ig} are the component fugacities in the oil and gas phases calculated with an EOS, and f_s is the fugacity of solid phase asphaltenes calculated from:

$$\ln f_g = \ln f_g^* + \frac{V_g(P - P^*)}{RT} \tag{4}$$

V_s is the asphaltene molar volume and f_g and f_g^* are the fugacities of pure solid asphaltene at pressures p and p^*, respectively. In this model, asphaltene deposit considers as a pure and dense solid particle. There is solid precipitation if

$$\ln f_{n,o} \geq \ln f_g$$

An important step in the modeling of asphaltene precipitation is the separation of the heaviest component of crude oil (C_{31+}) into precipitating (C_{31+A}) and non-precipitating (C_{31+B}) components. These two components have the same critical properties and acentric factors, but they have different binary interaction coefficients with light end hydrocarbons. The precipitating component has a higher binary interaction with light end hydrocarbons. As the interaction coefficients increase, the compatibility of precipitating component with light components in the fluid decreases. As a result, the precipitating component tends to precipitate as the amount of light components in solution increases. The interaction coefficients between hydrocarbons are calculated from:

$$d_{ik} = 1 - \left(\frac{2V_{ci}^{1/6}V_{ck}^{1/6}}{V_{ci}^{1/3} + V_{ck}^{1/3}}\right)^e \tag{5}$$

where V_{ci} and V_{ck} are the critical volume of component i and k, and e is an adjustable parameter. Normally, different values of e are used for C_{31+A} and C_{31+B}, with $e(C_{31+A}) > e(C_{31+B})$.

To use Eq. (4), the reference fugacity (f_g^*) is considered equal to the fugacity of the precipitating component, which is calculated from the equation of state (EOS). Equation 6 shows the calculation of the mole fraction of the precipitating.

$$Z_{precipitating} = \frac{w_{asphaltene} * M_{oil}}{M_{precipitating}} \tag{6}$$

For modeling of asphaltene precipitation, solid molar volume and binary interaction coefficients between precipitating and light end components are the matching parameters. The amount of asphaltene precipitation at saturation pressure is controlled by solid molar volume that is considered slightly higher than the one predicted by EOS. Binary interaction coefficients also control the precipitating and dissolution of asphaltene particles under saturation pressure [12]. Table 6 shows the matching parameters used in modeling, and Fig. 7 represents the results of modeling in natural depletion tests at different temperatures. Upper and lower onset pressures at different temperatures are obtained from Fig. 7, and the results of onset pressures are summarized in Table 7. The asphaltene deposition envelope (ADE) is represented by Fig. 7. It has been observed that with decrease of temperature, the upper onset pressure increases until a maximum pressure, and with further decrease of temperature it will decline (Fig. 8). In this work to see in what tempera-

Table 6. Molar volume and interaction coefficient at different temperatures

Temperature (°C)	Solid molar volume (m^3/kmole)	Interaction coefficient C1-C5
96.1	0.6456	0.181
80	0.6398	0.210
60	0.6288	0.165

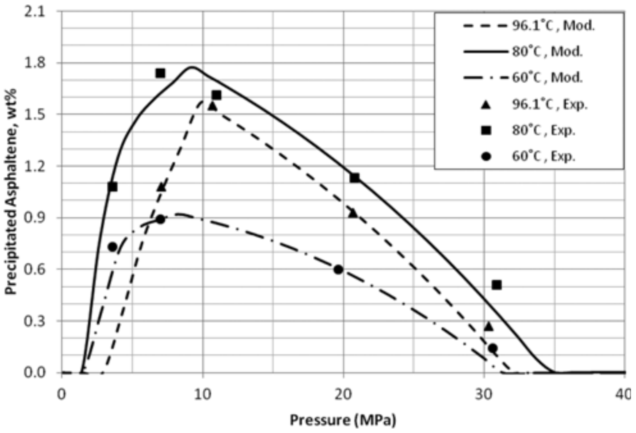

Fig. 7. Results of modeling for pressure depletion tests.

Table 7. Upper and lower onset pressures at different temperatures

Onset-temperature	T=96.1 °C	T=80 °C	T=60 °C
Upper onset pressure (MPa)	32.34	34.96	31.36
Lower onset pressure (MPa)	2.96	1.45	1.54

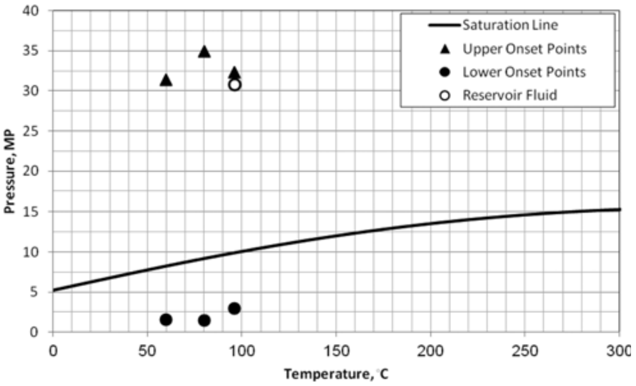

Fig. 8. Asphaltene deposition envelope (ADE).

ture the maximum upper onset pressure occurs, we needed more experimental data in a wide range of temperatures. Lower onset pressure also falls as temperature decreases and at a specified temperature this behavior will be reversed. This figure indicates that the studied reservoir fluid has a high potential of asphaltene formation and originally is unstable even at an early stage of production. Inside the reservoir, after precipitation has occurred, the asphaltene particles can remain in suspension and flow within the oil phase, or can deposit onto the rock surface.

CONCLUSION

1. Decreasing pressure above the saturation pressure leads to asphaltene precipitation increases, but below the saturation pressure causes the asphaltene content to decrease at different temperatures.

2. At high temperatures, asphaltene content increases by decreasing temperature, so the colloidal phenomenon is dominant.

3. At low temperatures, by decreasing temperature, asphaltene particles resolve into crude oil and the solubility phenomenon controls the process.

4. In CO_2 gas injection tests, a small increase in asphaltene precipitation was seen when the CO_2 mole percent was lower than 40. There is also less asphaltene precipitation at the pressure of 20.69 MPa compared to 31.02 MPa, and The maximum amount of asphaltene that can precipitate out of the fluid is 6.7 wt%, which occurs in the reservoir pressure and 60% of CO_2 mole%.

5. Asphaltene precipitation is much higher in CO_2 injection tests than natural depletion at early production stages of reservoir.

6. A good estimation of the asphaltene deposition envelope (ADE) at high temperatures was obtained for this heavy crude oil.

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NOMENCLATURE

d_{ik} : interaction coefficients between components i and k
d_o : density of oil sample [kg /m^3]
e : adjustable parameter for interaction coefficients between hydrocarbons
f_{ig} and f_{io} : fugacity of component i in gas and oil phase [MPa]
$f_{n,o}$: fugacity of components in oil phase [MPa]
f_g : solid fugacity [MPa]
f_g^* : reference solid fugacity [MPa]
M_{oil} : molecular weight of oil [kg/kmole]
$M_{precipitating}$: molecular weight of the precipitated component [kg/kmole]
n_c : number of components in the oil and gas phases
P_b : bubble pressure [MPa]
P_r : reservoir pressure [MPa]
P^* : reference pressure [MPa]
R : universal gas constant [8.314 kPa m^3/kmole K]
S : saturation [fraction]
SARA : saturated-asphaltene-resin-aromatic
T : temperature [°C]
v_a : molar volume of asphaltene [m^3/kmole]
v_o : molar volume of oil sample [m^3/kmole]
V_s : solid molar volume [m^3/kmole]
$w_{asphaltene}$: weight percent of the precipitated asphaltene [gr/gr]
V_{ci} and V_{ck} : critical volume of component i and k [m^3/kmol]
$Z_{precipitating}$: mole fraction of the precipitating component

Greek Letters

δ_a : solubility parameter of asphaltene [Mpa$^{1/2}$]
δ_o : solubility parameter of oil [Mpa$^{1/2}$]
κ_o : compressibility factor of oil [kpa^{-1}]

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