Experimental Investigation and Modeling of Asphaltene Precipitation due to Gas Injection

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ABSTRACT: *Asphaltene instability is one of the major problems in gas injection projects throughout the world. Numerous models have been developed to predict asphaltene precipitation; The scaling equation is an attractive tool because of its simplicity and not involving complex properties of asphaltene. In this work, a new scaling model is presented to account for asphaltene precipitation due to gas injection at reservoir conditions. Extensive published data from literature have been used in model preparation. To check predictive capability of the equation, miscible gas injection experiments are conducted for a southwest Iranian oil reservoir. Experimental results show that methane injection has significant effect on asphaltene precipitation and direct effect of temperature is less severe than other parameters. In addition to the accuracy and simplicity, the proposed equation provides universal parameters which make this approach novel for evaluation of future gas injection projects when simple PVT data are available.*

KEY WORDS: *Asphaltene precipitation, Miscible gas injection, Scaling equation.*

INTRODUCTION

It is well known that the injection of carbon dioxide, nitrogen and hydrocarbon gases changes the solubility of heavy components in the reservoir oil and causes asphaltene instability [1-3]. Numerous thermodynamic-based models have been presented to predict asphaltene precipitation. *Hirschberg et al*. [4] applied Flory-Huggins polymer solution theory to present a thermodynamic model for prediction of asphaltene precipitation. This model considers asphaltene as a heavy liquid and performs phase behaviour calculations for gas-oil and oil-asphaltene systems. It was later modified to include asphaltene polydispersity [5]. *Yang et al.* [1] proposed a modified Hirschberg solubility model and a new solubility parameter correlation as a function of specific gravity, molecular weight and boiling point. *Pazuki et al.* [6] defined an interaction parameter between polymer and solvent and correlated it with molecular weight of solution and asphaltene. This parameter is estimated by

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tuning three matching parameters from experimental data. Later, they used the perturbation theory for presentation of a new equation of state to study phase behavior of crude oil and predict asphaltene precipitation [7]. *Nikookar et al.* [8] also modified an Equation Of State (EOS) for calculation of phase behavior of fluids and solubility parameter. *Mofidi et al.* [9] excluded asphaltene molecular weight and presented a simplified thermodynamic approach. *Vafaei-Sefti et al.* incorporated an association term in Peng-Robinson equation of state by a simple association factor obtained from molecular weight distribution (fractional molecular weight) and average molecular weight of asphaltene. Since they related EOS parameters of asphaltene linearly to the fractional molecular weight, there was no need to define asphaltene critical properties (e.g. critical temperature and pressure). The results of their model were in good agreement with experimental data and other previous attempts [10, 11]. *Soulgani et al.* [12] introduced a kinetic for precipitation and also investigated deposition of asphaltene in the wellstring which is a common problem in production wells.

Leontaritis & Mansoori [13] presented a colloidal model by applying statistical thermodynamics. They defined a critical resin concentration (CRC to estimate critical chemical potential of resins. Asphaltene would precipitate if chemical potential of resins in the mixture is less than the critical value. *Escobedo & Mansoori* [14] concluded that this model is accurate for determination of onset of asphaltene precipitation and does not quantify the precipitates. Later, *Wang et al.* [15] mentioned that some experimental evidences were against this model.

Pan & Firoozabadi [16, 17] proposed thermodynamic micellization model that describes asphaltene precipitation by minimizing total free Gibbs energy of system (including asphaltene monomers and micelles). Although this model considers colloidal nature of oil, it must be optimized to comprise effect of resins and precipitants successfully.

Zahedi et al. [18] investigated the ability of artificial neural networks in the modeling and prediction of the asphaltene precipitation and compared the results to conventional thermodynamic models. It was observed that the most suitable algorithm with appropriate number of neurons in the hidden layer which provides the minimum error is the Levenberg–Marquardt (LM) algorithm.

Almost all mentioned models involve asphaltene properties (e.g. density, molecular weight) in their calculations. Accurate determination of asphaltene mean molecular weight and molecular weight distribution of aggregates is difficult because of association of asphaltene particles and their polydisperse nature in molecular size and polarity [19,20]. To overcome this difficulty, common thermodynamic models consider asphaltene fraction as a heavy pseudo-component and tune its properties to match experimental data. This regression is accurate only at static test conditions (i.e. specified pressure range, temperature and injected gas concentration); at reservoir conditions, where different grid blocks are different in injected gas saturation and temperature, results of original regressed model would be misleading.

Rassamdana et al. [21,22] conducted asphaltene precipitation experiments by n-alkanes (n-C_5 to n-C_{10}) at standard conditions. They observed that conventional thermodynamic model of Flory-Huggins was not able to predict their experiment results accurately. As a result, the following scaling equation was developed:

$$X = \frac{R}{M^{Z}} \qquad Y = \frac{W}{R^{Z'}} \tag{1}$$

Where R is the ratio of the volume of injected solvent to the weight of the crude oil, M is the molecular weight of the solvent and W is the weight percent of precipitated asphaltene.

For Z'= -2 and Z = 0.25, all data points would collapse onto a single third-order polynomial curve in general form of:

$$Y = a_0 + a_1X + a_2X^2 + a_3X^3 \qquad (X \geq X_C) \tag{2}$$

Hu et al. [23] checked the predictive capability of the scaling equation by comparison with literature precipitation data. They also examined the universality of exponents Z and Z'. It was concluded that Z'= -2 was a universal constant regardless of oil composition and type of solvent and the optimum value of Z varied between 0.1 and 0.5.

Ashoori et al. [24] performed asphaltene precipitation experiments on Iranian dead oil samples at different temperatures and introduced the effect of temperature in the original scaling equation:

$$X = \frac{R}{T^{n}M^{Z}} \qquad Y = \frac{W}{R^{Z'}} \tag{3}$$

Temperature exponent, n, was defined in the range of 0.1 to 0.25 whereas other constants (Z, Z') were unchanged.

Bagheri et al. [25] also performed some titration experiments on dead oil samples and proposed the following scaling equation:

$$X = \frac{R_m \times C_t \times GOR}{X_{C31+} \times R_t} \qquad Y = \frac{W \times R_m \times R_t}{C_t} \tag{4}$$

Where R_m is solvent to oil dilution ratio (cm^3/g), GOR is the gas oil ratio (SCF/STB), R_t and C_t are resin and asphaltene contents (wt %), W is amount of precipitated asphaltene (wt %) and X_{C31+} is the mole fraction of C_{31+} fraction in the oil sample. Model results were compared to original scaling predictions; experimental asphaltene precipitation data (in ambient pressure and temperature) were predicted better by the new model.

In this paper, the basics of original scaling equation are applied to develop a new model for prediction of asphaltene precipitation due to gas injection at reservoir conditions (high pressure and temperature). Subsequently, miscible gas injection experiments are conducted to verify predictive capability of presented model and finally, a sensitivity analysis is performed to check universality of the model parameters.

MODELING

Conventional scaling equation is initiated for standard conditions. In this study, a new model is presented to predict asphaltene precipitation due to High Pressure High Temperature (HPHT) gas injection.

Compared to standard conditions, more effective parameters have to be accounted for the HPHT gas injection. Reservoir pressure and temperature, bubble point pressure of sample, mole percent of precipitant (injected gas) and gas molecular weight were considered in the new model.

Different configurations of mentioned parameters were examined considering physical effect of each parameter on precipitation phenomenon. The most simple and accurate definition for X and Y was found to be as:

$$X = \frac{R_m}{M^Z T^n} \qquad Y = \frac{W}{R_m{}^{Z'}} \tag{5}$$

Where

$$R_m = e^{-h\left(\frac{P_b}{P}-1\right)^2} * R \tag{6}$$

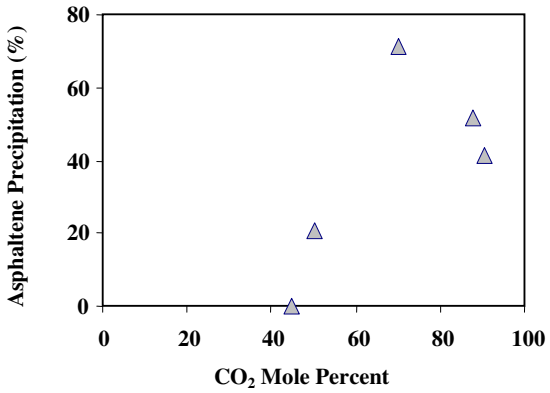

Fig. 1: Asphaltene precipitation vs. mole% of injected gas at 1900 psi and 149 ˚F [26].

M is the molecular weight of injected gas, W is weight percent of precipitated asphaltene, P is pressure, P_b is bubble point pressure of mixture, T is temperature in ˚F and R is the ratio of the volume of injected gas to the weight of crude oil.

Exponential function was used to model both increasing and decreasing effects of pressure on asphaltene precipitation above and below bubble point pressure.

Different sets of gas injection data were collected from literature to determine model exponents. Integrated data were mostly on CO_2 injection and data for other gases were sparse.

Novosad et al. [26] evaluated stability of asphaltene in a 29 ˚API reservoir under CO_2 injection (Fig. 1). It is observed that when CO_2 concentration increases, asphaltene particles become unstable at critical concentration of 45 mole percent. Asphaltene re-dissolution is due to loss of miscibility at high gas concentration.

Srivastava et al. [2] presented results of dynamic and static precipitation tests to investigate the asphaltene deposition problems in southeast Saskatchewan's Weyburn reservoir. Tests were conducted at the reservoir temperature and pressure conditions for three different oil samples (Fig. 2).

Takahashi et al. [27] investigated effect of CO_2 concentration on asphaltene precipitation at reservoir temperature for a range of pressures. As mentioned by authors, some data were not satisfactory which were excluded from modeling approach to assure accuracy of model parameters. Approved experimental results are shown in Fig. 3.

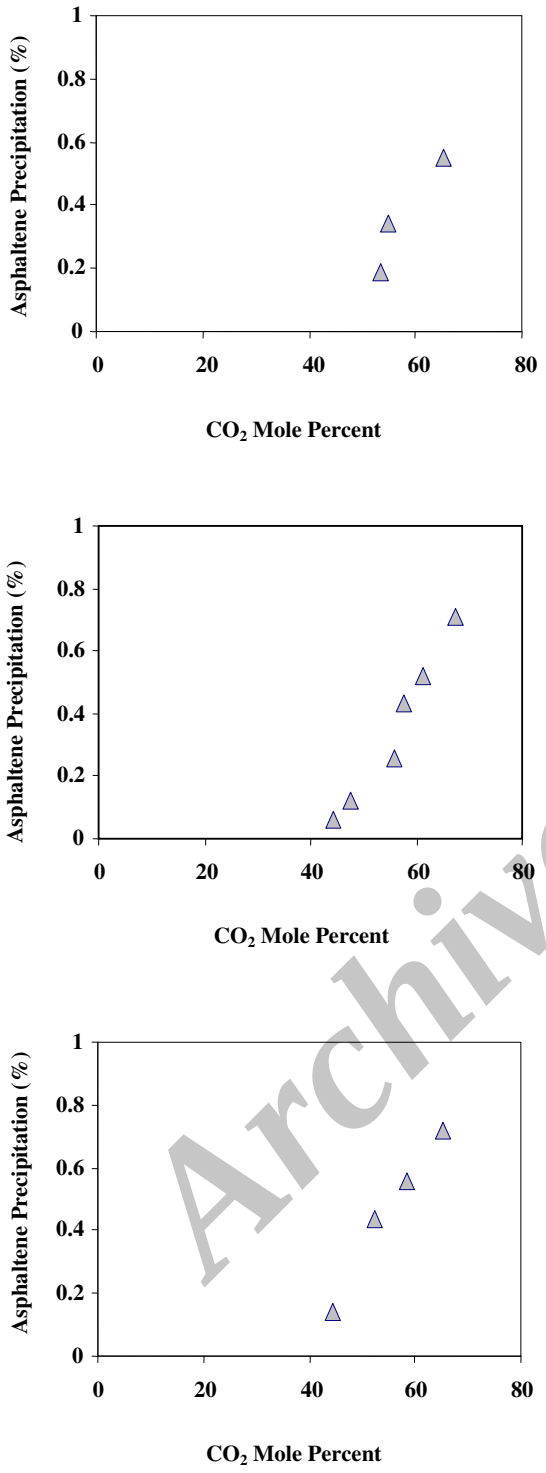

Fig. 2: a) Sample W1 at 16 MPa and 59˚C, (b) Sample W2 at 16 MPa and 61˚C, (c) Sample W3 at 16 MPa and 63 ˚C [2].

Other investigations with vague experiment details or incomplete data were excluded from modeling. *Jamaluddin et al.* [3] investigated effect of nitrogen injection on asphaltene instability by isothermal depressurization with 5, 10 and 20 mole percent of nitrogen at reservoir temperature. *Hu et al.* [28] studied asphaltene precipitation of two reservoir oil samples collected from Jinlin oil field. No asphaltene precipitation was detected during the pressure depletion processes without CO_2 injection. For the CO_2-injected oil systems, the effects of operating pressure, injected CO_2 concentration, and multiple-contact on the onset and amount of asphaltene precipitation were studied under the reservoir temperature. *Negahban et al.* [29] discussed experimental work associated with the evaluation of asphaltene precipitation due to injection of a synthetic gas for a field in UAE. *Idem et al.* [30] used a molar CO_2 programmed titration technique to evaluate the kinetics of CO_2-induced asphaltene precipitation from three Saskatchewan crude oils under isothermal (in the range of 300-338 K) and isobaric (at 17.2 MPa) reservoir conditions and formulated a kinetic for achieved data.

Using defined X and Y parameters, it is observed that the plot of Y vs. X collapses all literature data on the following third-order polynomial function (Fig. 4).

$$Y = 3.3877X^3 + 0.4831X^2 - 0.0099X + 8\times 10^{-6} \qquad (7)$$

The exponents, Z, Z', n and h were carefully determined for best fit of the literature experimental data. Afterwards, the coefficients of 3rd order polynomial (a_i) could be extended to other samples.

EXPERIMENTSAL SECTION

Crude oil sample characterization

Single phase bottom-hole samples were taken from a southwest Iranian oil reservoir. Molecular weight, °API, solution gas oil ratio and composition of samples were determined (Tables 1 -3). The oil was also characterized for saturates (S), aromatics (A), resins (R) and asphaltenes (A) using the SARA method as described by Institute of Petroleum handbook [30].

Asphaltene precipitation tests

High pressure high temperature filtration test was conducted to quantify the amount of asphaltenes that may precipitate at a given pressure. The schematic diagram of

Table 1: Properties of crude oil.

Temperature	°F	255
Saturation Pressure	psia	1722
Solution GOR	SCF/STB	450
API Gravity of Residual Oil	°API	20.17

Table 2: SARA analysis.

Saturated	wt.%	30.10
Aromatic	wt.%	42.10
Resin	wt.%	13.36
Asphaltene	wt.%	13.75

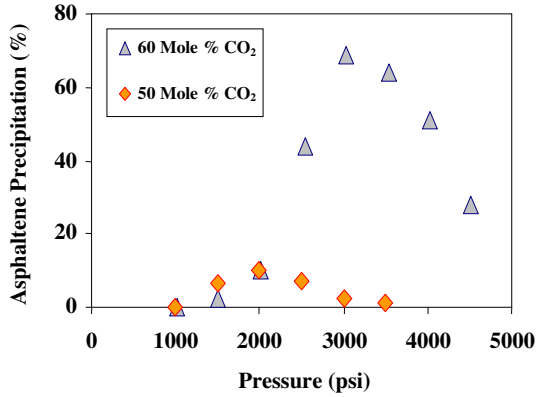

Fig. 3: Asphaltene precipitation vs. pressure at 212 ˚F for 50 and 60 mole% of CO_2 [27].

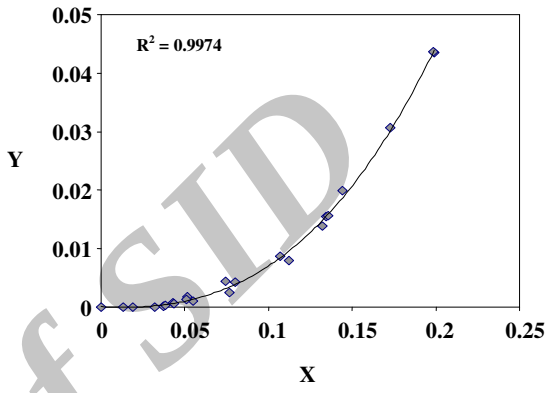

Fig. 4: Collapse of literature gas injection data by new scaling equation.

the experimental setup is shown in Fig. 5. The main part of the system is a mercury-free, visual JEFRI equilibrium cell. The working temperature range of the cell is -30-200 °C and the maximum working pressure is 10000 psi.

First, the conventional pressure depletion process was performed at reservoir temperature to determine asphaltene precipitation happening in the absence of gas injection.

The experimental procedure is described as follows:

1- The equilibrium cell was thoroughly cleaned and maintained at a reservoir temperature (255 °F).

2- A known volume of oil was charged into the cell under single-phase conditions at reservoir pressure (5500 psi). The sample was allowed to equilibrate overnight. A magnetic stirrer was used to agitate the sample to accelerate the equilibrium process.

3- Cell pressure was declined in subsequent predefined steps; at each point, a high pressure filtration process was conducted with a 0.2μ filter paper. During the filtration process, it was important that the sample remain monophasic as it passes through the filter manifold. High pressure helium was used to maintain a back-pressure on the downstream of the filter. As a result, the filtration process was very close to an isobaric process.

4- Filtered oil was flashed in a separator and asphaltene content of residual oil was measured using the standard IP procedure. The difference between asphaltene content of original and filtered oil at each pressure determines the weight percent of precipitated asphaltene. Experimental results are presented in Fig. 6.

Further experiments were performed to investigate effect of gas injection on asphaltene precipitation behaviour. First, a swelling test was conducted to investigate effect of gas injection on bubble point pressure of the mixture at different temperatures (Fig. 7).

The working procedure for gas injection experiments is almost similar to natural depletion; After feeding a known volume of oil sample into the equilibrium cell, methane was injected at GOR of 600 SCF/STB while the cell pressure maintained above the mixture saturation pressure to prepare a uniform single phase sample. All steps were repeated at 180 ˚F to investigate effect of temperature on asphaltene precipitation. Experimental results are shown in Fig. 8.

It is observed that effect of temperature is less severe and change in asphaltene precipitation curve is mostly due to variation of bubble point pressure with temperature.

Table 3: Composition of oil sample.

Component	Mole Percent
H_2S	2.70
N_2	0.21
CO_2	5.14
C_1	22.00
C_2	7.10
C_3	5.34
i-C_4	0.99
n-C_4	2.78
i-C_5	1.12
n-C_5	1.41
C_6	5.55
C_7	3.84
C_8	4.02
C_9	2.85
C_{10}	2.83
C_{11}	2.58
C_{12+}	29.54
MW of C_{12+} Fraction	418
MW of Reservoir oil	149
SG. of C_{12+} Fraction @ 60/60 °F	0.9760

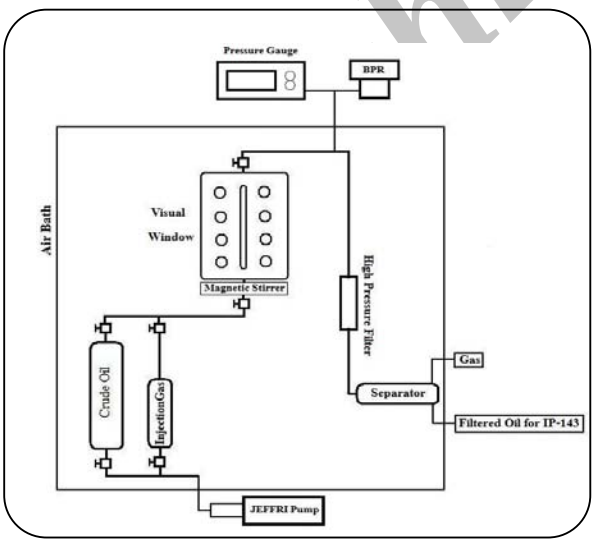

Fig. 5: Schematic diagram of HPHT gas injection experimental setup.

Effect of temperature seems to be different above and below bubble point pressure. Above P_b, asphaltene becomes more soluble in oil as the temperature decreases; it can be expected that the energy of the mixture to prevent the interaction of asphaltene clusters to aggregate and precipitate increases with decreasing temperature (i.e., the solubility parameter of oil decreases with increasing temperature). Below P_b, effect of temperature on mixture solubility is insignificant compared to gas evolution and asphaltene re-dissolution rate is controlled by amount of liberated gas rather than temperature.

RESULTS AND DISCUSSION

Experimental Results show that methane injection increases asphaltene precipitation more than three times compared to natural depletion (see Table 4). As a result, asphaltene deposition may cause serious reduction in the field productivity.

Our experimental data were used to evaluate new model. Although the general equation is regressed for literature CO_2 data, Fig. 9 shows that experimental data of methane injection are also collapsed on the general curve.

Working procedure for estimation of asphaltene precipitation due to gas injection by applying scaling equation would be summarized as follows:

- Preliminary characterization of oil and gas samples (i.e. measurement of oil and injected gas molecular weight and mixture bubble point pressure).
- Determination of asphaltene content by SARA analysis.
- Calculation of X parameter from Eq. (5) for desired pressure, temperature and gas concentration.
- Evaluation of Y parameter from general 3rd order polynomial (Eq. (7)).
- Determination of precipitated asphaltene weight percent from Y definition (Eq. (5)).

Fig. 10 compares experimental results with predictions from new scaling model. It is observed that proposed model successfully predicts our experimental data.

One of main advantages of new scaling equation is prediction of asphaltene instability under gas injection without involving asphaltene properties. When simple PVT data including molecular weight of oil and injected gas sample, mixture bubble point pressure and asphaltene content of oil sample are available, the scaling equation would be applicable.

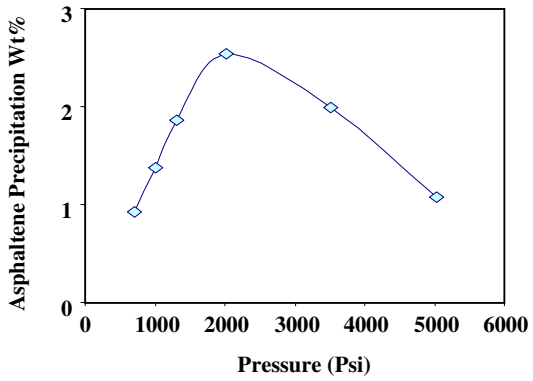

Fig. 6: Asphaltene precipitation curve for natural depletion.

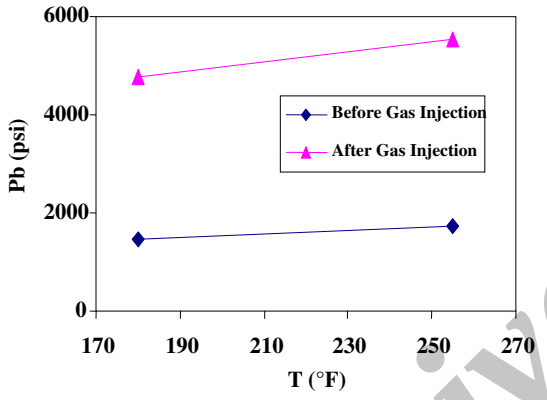

Fig. 7: Variation of bubble point pressure with temperature and gas injection

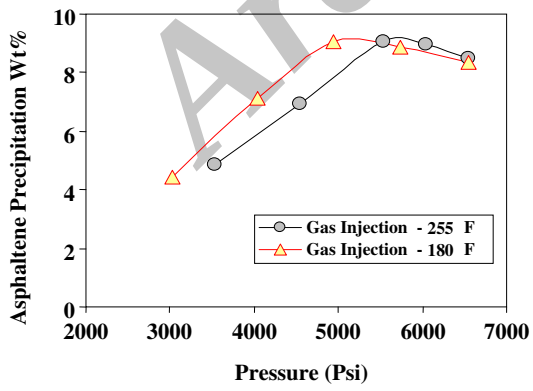

Fig. 8: Asphaltene precipitation curve for gas injection at different temperatures (180 and 250 °F).

Table 4: Results of Asphaltene Precipitation tests.

	Pressure (psi)	Precipitated Asphaltene (wt%)
Natural Depletion	5021	1.07
	3516	1.99
	2016	2.54
	1315	1.86
	1015	1.38
	715	0.93
Methane Injection – 255 °F	6543	8.49
	6043	8.95
	5537	9.04
	4537	6.93
	3531	4.88
Methane Injection – 180 °F	6543	8.36
	5743	8.85
	4937	9.05
	4036	7.1
	3028	4.44

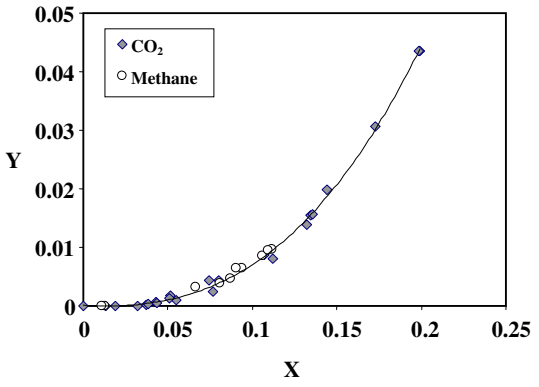

Fig. 9: Collapse of methane injection data on general scaling curve.

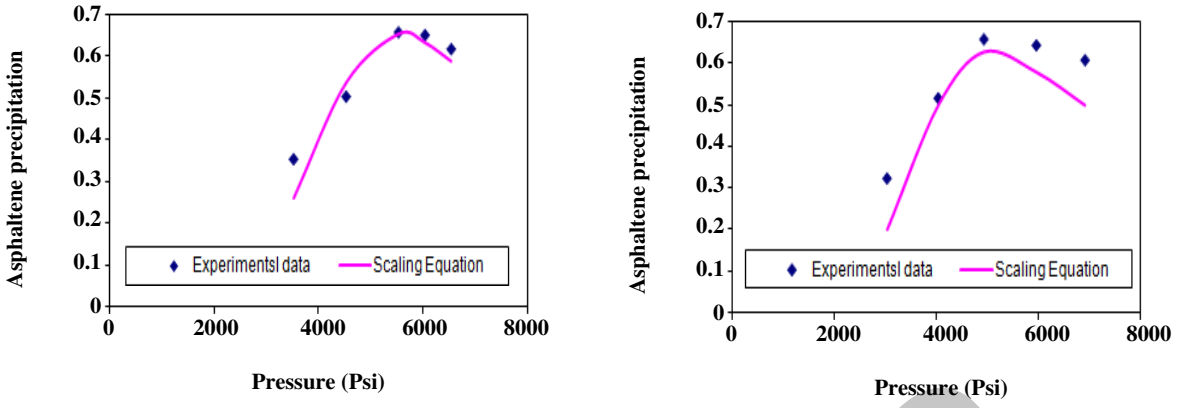

Fig. 10: Comparison of scaling model prediction vs. experimental data, a) Methane Injection (T=255 F), b) Methane Injection (T=180 F).

As mentioned before, in thermodynamic–based models asphaltene is considered as a pseudo-component and its properties are tuned to match experimental data. However, this regression is valid only for static test conditions. The regressed model does not comprise any change in thermodynamic conditions (e.g. gas concentration and temperature). At reservoir conditions, injected gas concentration is different from point to point and applying original regressed model to different grid blocks would be misleading. Scaling equation would be more reliable because in this approach a unique X is estimated for each grid block depending on its properties and the corresponding asphaltene precipitation would be calculated from the 3rd order tuned polynomial.

Sensitivity analysis was performed to investigate universality of model parameters. Z' = 2 was found to be universal in agreement with original scaling equation. Different Z' values resulted in dispersion of XY plot.

Rassamdana et al. [21] and *Hu et al.* [28] suggested range of 0.1 to 0.5 for Z exponent. The optimum Z value was found to be Z=0.11 in this work. Data points will scatter around the general curve if other values are used. Since h is related to the pressure function with important impact on precipitation, it should be tuned more accurately. The best value for h is suggested 7. Higher h values move the points toward the horizontal axis which is not the favour and lower values again disperse data.

Different values between -0.05 and 0.05 were examined for the temperature exponent. The best fit obtained for n=-0.04, although excluding temperature term (n=0) did not produce large regression error. This is in agreement with our experiment results and literature observations [32] that direct effect of temperature is negligible compared to other parameters and are different above and below bubble point pressure.

CONCLUSIONS

- Experimental results showed that methane injection has significant effect on asphaltene precipitation and investigation of asphaltene stability is a key point in success of gas injection projects.

- Effect of temperature on asphaltene precipitation is less severe compared to other parameters (e.g. pressure and injected gas concentration).

- A new scaling equation was developed to predict asphaltene precipitation due to gas injection. This simple model describes asphaltene instability with preliminary PVT data.

- The scaling equation is more reliable for dynamic reservoir conditions because its parameters are redefined if any change in thermodynamic conditions occurs.

- Collapse of data is often possible by defining dimensionless parameters. Although X and Y are not dimensionless, excellent collapse of data from different oil and gas samples for various temperature and pressures is reached.

- Equation parameters are defined so that all exponents are universal for gas injection. This general curve which is constructed from extensive experimental data can be applied for other samples. However, more accurate results would be gained if unique 3rd order polynomial is constructed for each sample.

- More experimental data on nitrogen and hydrocarbon gas injection would improve equation exponents for these gases.

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