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Suggestion of Appropriate Equations of State for Density/Viscosity Estimation in Wax/Catalyst Separation Using Near-Critical Hexane

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Abstract: The target of this study was to investigate the feasibility of wax/catalyst separation process using near-critical hexane, by proposing two reasonable EOS's used for hydrocarbons near-critical density/viscosity estimation. This target was accomplished by studying some theoretical correlations used for hydrocarbons density and viscosity prediction, which have the capability of functioning at near-critical region. According to this investigation, the reasonable VTPR equation-of-state was proposed for density calculation. Furthermore, based on the similarity between P-v-T and T-μ-P relationships, an appropriate MPR EOS-based viscosity model was suggested for viscosity estimation. The advantages of these correlations in terms of accuracy, applicability to both gas/liquid, high pressure/low pressure and having smooth phase transition in the near-critical region were investigated for n-hexane, wax and some hydrocarbon mixtures by making comparison between the calculated properties and the experimental one's. At the last part, wax/catalyst separation tests using near-critical hexane were carried out in conjunction with physical properties calculations by the suggested EOS's. The main objective was understanding the consistency of density/viscosity values with separation efficiencies amounts in quality and in fact the process analysis. Test results showed that maximum separation efficiency occurred in applying supercritical hexane state. In addition, based on the proposed EOS's estimation, the lowest densities and viscosities values were gained at this phase, that these two outcomes of this investigation were also in line with the Stoke's theorem.

Key words: Separation, physical properties, equations of state, near-critical

INTRODUCTION

Today, slurry-bubble-column reactors are more preferable for the Fischer-Tropsch synthesis (FT) in Gas to Liquid (GTL) process. Beside all advantages of these reactors such as elimination hot spots within the catalyst bed, limiting the coke buildup on catalyst particles and both addition and withdrawal of catalyst during operation, the separation of Fischer-Tropsch catalyst from wax products is an important challenge when the synthesis is conducted in a slurry-phase reactor (Lunsford, 2000).

Since 1950, various solid/liquid separation techniques have been tested for F-T catalyst/wax separation (Zhou, 1991) including settling tanks, filtration, centrifugation, thermal cracking and HDMS, However all these methods had their own problems such as poor mechanical history, high capital and operating costs, intermitted operation, difficulties in recovered catalyst recycle and low rate of catalyst separation. All these problems stem from very high density and viscosity of wax media in reactor operating condition.

Separation of F-T wax/catalyst using near-critical fluid extraction method (SFE) was first proposed in 2001 (Roberts and Kilpatrick, 2001). In this method, a solvent which its critical temperature is within the operating temperature range of slurry reactor (200-300°C) is of primary interest for use as a supercritical solvent. A reasonable choice is hexane (Tc = 234.4°C; Pc = 30.25 bar).

Such organic solvent dissolves wax hydrocarbons, so that the density and viscosity of media decreases until it leads to easily catalyst/wax separation by all mentioned methods (Zhou, 1991). In other words, the main reason for applying the near-critical fluid is its special and excellent physical properties such as density and viscosity, caused in increasing the rate of solid precipitation.

In addition, it should be noted that wax/catalyst separation modeling and any other near-critical processes, inevitably require the accurate values of physical properties particularly, densities and viscosities in critical region, while there was always shortage of such empirical properties within the mentioned region because of their measurement difficulties. Therefore, achieving accurate

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thermodynamic equations is so considerable to predict such properties for the process analysis from the aspect quality and then its modeling.

In this way, improved equations of state applicant shall be more reasonable for their simplicity, accuracy and the capability of adjustment for the desire components and operating conditions.

The target of this work is to search and study some appropriate EOS's and then investigate and compare their ability and accuracy for near-critical hydrocarbons density/viscosity prediction, so that such correlations shall be used for describing the feasibility of the wax/catalyst separation process using near-critical hexane.

Such correlations would be also used for the exact modeling of the stated process in future, if the accurate correlation is derived for fine solid settling in high density/viscosity fluids.

EOS-BASED VISCOSITY CORRELATION

Modified PR-viscosity correlation for pure substances: The viscosity data of hydrocarbon fluids are among the basic data required in petroleum/natural gas production, processing and transportation. Although numerous viscosity graphs and correlations (empirical or semi-empirical) for hydrocarbon liquids and gases are available in the literature, there are three main drawbacks in their application (Guo *et al.*, 1997):

- Application range and accuracy are limited.
- As the viscosity of liquid phase and gas phase are calculated by different graphs/correlations, a smooth transition cannot be achieved in near-critical and supercritical region.
- Density, in general, involved in evaluating the fluid viscosity, hence separate density correlation is required.

The major advantages of developing a viscosity model based on an equation of state (EOS) are (Guo *et al.*, 1997, 2001).

- Viscosity of all gas, liquid and supercritical phases can be described by a single model, achieving a smooth transition of liquid/gas in the near-critical region.
- Both high/low pressure and temperature data can be correlated and density is not involved in evaluating the fluid viscosity.

One of the most reasonable EOS's for the viscosity calculation is the two-parameter Peng Robinson (PR) EOS.

According to the similarity between the P-v-T and T-μ-P relationships, Guo *et al.* (1997) proposed an equation for calculating hydrocarbons viscosity and their mixtures, but the calculation results indicated that the generalized model was not capable to describe the viscosity with acceptable accuracy over a wide range of temperatures and pressures, particularly in near-critical region. Therefore, in 2001 (Guo *et al.*, 2001), they proposed Modified PR (MPR) EOS viscosity correlation.

By transforming the PR EOS by the mentioned similarity application, the following MPR viscosity equation was achieved (Guo *et al.*, 2001):

$$T = \frac{rP}{\mu - b'} - \frac{a}{\mu(\mu + b) + b(\mu - b)} \quad (1)$$

As above equation shows, the positions of T and P in the PR EOS are interchanged; v is replaced by μ and gas constant R is replaced by r (defined subsequently), where (Guo *et al.*, 1997, 2001):

$$a = 0.45724 \frac{r_c^2 P_c^2}{T_c} \quad (2)$$

$$b = 0.07780 \frac{r_c P_c}{T_c} \quad (3)$$

$$r = r_c \tau(T_r, P_r) \quad (4)$$

$$r_c = \frac{\mu_c Y_c}{P_c Z_c} \quad (5)$$

$$\mu_c = 7.7 T_c^{-1/6} M^{1/2} P_c^{2/3} \quad (6)$$

$$\tau(T_r, P_r) = \{1 + Q_1[(P_r T_r)^{0.5} - 1]\}^{-2} \quad (7)$$

$$b' = b\phi(T_r, P_r) \quad (8)$$

$$\phi(T_r, P_r) = \exp[Q_2(\sqrt{T_r} - 1) + Q_3(\sqrt{P_r} - 1)] \quad (9)$$

τ and ϕ are determined from Eq. (1) based on the viscosity data of hydrocarbons.

For hydrocarbons C_5 to C_{30}, carbon dioxide and nitrogen, the three adjustable coefficients, Q_1, Q_2 and Q_3, are generalized in terms of acentric factor as follows (Guo *et al.*, 1997, 2001):

When $\omega < 0.3$:

$$Q_1 = 0.829599 + 0.350857\omega - 0.747680\omega^2 \qquad (10)$$

$$Q_2 = 1.94546 - 3.19777\omega - 2.80193\omega^2 \qquad (11)$$

$$Q_3 = 0.299757 - 2.20855\omega - 6.64959\omega^2 \qquad (12)$$

When $\omega < 0.3$:

$$Q_1 = 0.956763 + 0.192829\omega - 0.303189\omega^2 \qquad (13)$$

$$Q_2 = -0.258789 - 37.1071\omega + 20.5510\omega^2 \qquad (14)$$

$$Q_3 = 5.16307 - 12.8207\omega + 11.0109\omega^2 \qquad (15)$$

Allowable range for applying the stated correlation is approximately 0.6% below the critical pressure/temperature and 6.5% above it, for carbon dioxide and hydrocarbons lighter than n-decane, and 2.5% around the critical point for other alkanes up to C_{32}.

Extension to hydrocarbon mixtures: The stated EOS-based viscosity model was extended to mixtures by applying the following conventional mixing rules (Guo *et al.*,1997, 2001):

$$T = \frac{r_m p}{\mu_m - b_m} - \frac{a_m}{\mu_m(\mu_m + b_m) + b_m(\mu_m - b_m)} \qquad (16)$$

$$a_m = \sum_i x_i a_i \qquad (17)$$

$$b_m = \sum_i^i \sum_i x_i x_j \sqrt{b_i b_j}(1 - k_{ij}) \qquad (18)$$

$$r_m = \sum_i x_i r_i \qquad (19)$$

Where, subscript m denotes mixture. Where else, it is to be noted that the binary interaction coefficient, k_{ij}, is introduced to parameter b'_m in MPR model, instead of a_m (Guo *et al.*, 1997).

Selecting correct viscosity root: Equation (1) is cubic in μ so, Standard subroutine can be used to solve the viscosity root. If three viscosity roots were obtained at the specified temperature and pressure, the correct viscosity root is chosen as follows (Guo *et al.*, 1997, 2001):

- In the liquid region (when the pressure is greater than the saturated vapor pressure at prevailing temperature), maximum real root should be chosen.
- In the sub-critical gas region (when the pressure is lower than the saturated vapor pressure at prevailing temperature) the smallest real root greater than b', should be selected, as b' represents the asymptotic value of ideal gas viscosity at infinite temperature.

- In the supercritical region (T>T$_c$), the maximum real root must be chosen.

The saturated vapor pressures (P^s) should be calculated using the Antoine equation for P^s<1500 mm Hg, and from the Lee-Kesler equation for P^s>1500 mm Hg.

Test results on n-hexane and a hydrocarbon mixture: In this section, The proposed generalized MPR viscosity equation, Eq. (1), was applied to calculate the viscosity of a typical hydrocarbon mixture at high pressures and also to pure the normal hexane, presented as a solvent in wax/catalyst separation at its near-critical and supercritical condition.

A parallel comparison was also carried out with original PR-correlation and SRK EOS.

The calculation results are illustrated in Fig. 1 and 2 together with the experimental data (Stephen and Lucas, 1979; Sergio *et al.*, 2001).

Comparison between the predicted viscosities and their experimental data for hexane and the stated hydrocarbon mixture showed that the original PR and SRK EOS's were failed to describe most of the data tested, especially at high temperatures and pressures; however, the proposed MPR correlation produced the accurate results, even in the supercritical region for n-hexane and high pressures for the hydrocarbon mixture.
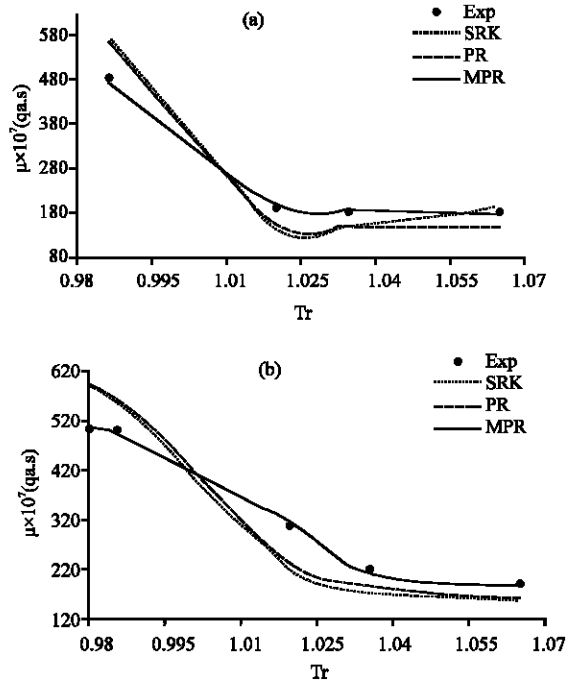

Fig. 1: Viscosity of n-hexane in near-critical and supercritical region:(a) $P_r = 1.00$: (b) $P_r = 1.16$ Exp. data (Stephen and Lucas, 1979)
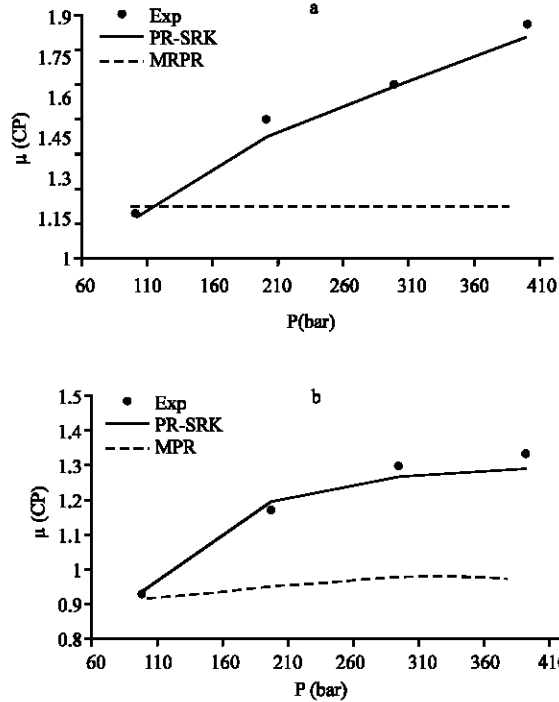
Fig. 2: Viscosity of %31.44 n-$C_{10}H_{22}$+%26.26 n-$C_{12}H_{26}$+ %19.75 n-$C_{16}H_{34}$ (a) T = 313.15k : (b) T= 333.15K. Exp. data (Sergio *et al.*, 2001)

It should be mentioned that the calculation accuracy could be further improved if the correlation was tuned by using non-zero k_{ij} values.

DENSITY CORRELATION BASED ON VOLUME TRANSLATION METHOD

Correlation for pure substances: Cubic equations of state are widely applied in chemical industry because of their simplicity and capabilities. However, they have inherent limitation that the critical compressibility factor is a constant for all fluids. As a result, the predicted volumetric properties in the vicinity of the critical region differ considerably from their experimental values.

Many efforts have been made to overcome these shortcomings in cubic EOS's with additional parameters introduced with many three-parameter CEOS's like SRK and PR to improve the critical compressibility factor.

The application of volume translation method was proposed in 2001. In 2005, it was used for PR EOS improvement (Lin and Duan, 2005) to modify the densities. It was shown that the volume translation method could be successfully applied to PR as VTPR EOS to improve density calculations of hydrocarbon molecules, especially in supercritical region.

The VTPR EOS can be written as (Lin and Duan, 2005):

$$P = \frac{RT}{v+c-b} - \frac{a}{(v+c)^2 + 2b(v+c) - b^2} \tag{20}$$

Where c is the volume translation parameter and can be calculated from experimental values (Lin and Duan, 2005):

$$^v VTPR = {}^v exp = {}^v PR^{-c} \tag{21}$$

$$a = 0.45724 \frac{r_c^2 P_c^2}{T_c} \alpha(T) \tag{22}$$

$$b = 0.07780 \frac{r_c P_c}{T_c} \tag{23}$$

$$\alpha(T) = [1 + m(1 - T_r^{0.5})]^2 \tag{24}$$

When $\omega \leq 0.49$:

$$m = 0.37464 + 1.54226 \, \omega - 0.26992 \, \omega^2 \tag{25}$$

When $\omega \geq 0.49$:

$$m = 0.37464 + 1.54226 \, \omega - 0.26992 \, \omega^2 \tag{26}$$

Because a constant volume translation, c, could not satisfy the critical region, a temperature-dependent parameter was considered as follows (Lin and Duan, 2005):

$$C(T) = C_c f(T_r) \tag{27}$$

$$c_c = (v_{PR})_c - (v_{exp})_c = (0.3074 - Z_c) \frac{RT_c}{P_c} \tag{28}$$

At the reduced temperature $T_r = 1.0$, the *f*-function in Eq. (27) should be unity because the volume translation must be equal to the critical volume translation in Eq. (28). Therefore (Lin and Duan, 2005):

$$f(Tr) = \beta + (1 - \beta) \exp(\gamma|1 - Tr|) \tag{29}$$

$$\beta = -2.8431 \exp[-64.2184(0.3074 - Z_c)] + 0.1735 \tag{30}$$

$$\gamma = -99.2558 + 301.6201 Z_c \tag{31}$$

β and γ are the fitted parameters.

The functional operating condition range to the related correlation is about 0.5% below the critical point

Table 1: Densities of 9/04% n-C_4H_{15+}73/38% n-C_7H_{15+} 17/38% n-$C_{16}H_{34}$ calculated by VTPR, ISRK, PR, SRK EOSs. Exp. data (Fenghour and Trusler, 2001) AAD (%)

VTPR	ISRK	PR	SRK	ρ_{exp}(mol 3 m)	P(Mpa)	T(K)
0.102	2.047	7.539	16.836	4802.7	0./773	447.90
0.849	2.584	3.756	5.682	4801./90	1.800	451.07
0.874	3.786	4.510	7.560	4800.7	3.286	455.65
1.756	5./316	6.710	6.78	4799.4	4.783	460.25
1.99	6.081	7.560	8.560	4798.2	6.274	464.86
2.050	7.358	9.562	10.650	4797.0	7.774	469.47
1.270	4.528	6.606	9.374		Overall AAD (%)	

and 7% above it for nitrogen, benzene, carbon dioxide and hydrocarbons lighter than n-nonane and 2% around the critical point for other alkanes up to C_{30}.

Extension the model to mixtures: The generalized VTPR EOS was also used for the calculating densities of hydrocarbon mixtures applying the conventional van der waals mixing rules (Lin and Duan, 2005):

$$a_m = \sum_{i=1} \sum_{j=1} x_i x_j a_{ij} \qquad (33)$$

$$a_{ij} = \sqrt{a_i a_j}(1 - K_{ij}) \qquad (34)$$

$$b_m = \sum_{i=1}^{n} x_i b_i \qquad (35)$$

$$C_m = \sum_{i=1} x_i c_i \qquad (36)$$

Where x_i is the mol fraction of component i, a_i and a_j are the cohesive energy, b_i the volumetric parameter of component i and k_{ij} is the binary interaction coefficient.

Calculation results for n-hexane and a hydrocarbon mixture: The proposed VTPR EOS was applied to predict the densities of n-hexane near its critical region. Similar calculations were also done for a typical hydrocarbon mixture at high pressures and temperatures. The results were compared with the original PR and SRK and with ISRK (Wei-Rong and Lempe, 1997) EOS's. These results are summarized in Fig. 3 and Table 1, respectively.

In Fig. 2 and Table 1, it is observed that the volume translation method shifted the densities rather closer to the experimental values in comparison with the results calculated by original SRK and PR EOS's and also ISRK equation. In addition, the mentioned method improved the estimations more significantly at high pressure, temperature and supercritical region. As results in Table 1 indicate, the overall AAD's of VTPR, ISRK, PR and SRK correlations are, respectively 1.27, 4.528, 6.6.6 and 9.374%.

Similar results are obtained for dioxide carbon, other light/heavy hydrocarbons and reservoir fluids.

TEST RESULTS FOR WAX/CATALYST SEPARATION

Estimating wax physical properties by suggested correlations: The different EOS's were compared to predict viscosities and densities of linear hydrocarbons at high pressures/temperatures. Among those equations, MPR and VTPR EOS's had reasonable accuracy to predict the mentioned properties, respectively.

In order to investigate the appropriateness of stated EOS's for describing the wax/catalyst separation process, some experiments were also carried out for testing wax density and viscosity accordance with the proposed equations.

The wax consisted of linear paraffins ranging from C_6 to C_{28}, with the melting point of 56°C produced in SBC reactors. The experiments were accomplished in several temperatures at atmospheric pressure. The results are given in Table 2.

The results showed that VTPR and MPR EOS's were in consisted with the empirical results and they had acceptable accuracy to predict wax viscosities and densities, respectively.

This result together with the results gained by previous sections also showed that the proposed EOSs could be applied to predict wax/hexane mixture densities and viscosities for a wide range of pressures and temperatures.

Experimental apparatus and method: The main objective of this study was suggesting correlations to describe the wax/catalyst separation results. The wax/catalyst separation facility used in this work was a batch setup, designed at Research Institute of Petroleum Industry Iran, during autumn 2005, and the experiments were conducted in early 2006. The related setup is represented in Fig. 4.

The setup was consisted of three main headers, including nitrogen header as system purging, hexane

Table 2: Wax density and viscosity Exp. data (Amiri and Khakdaman, 2005)

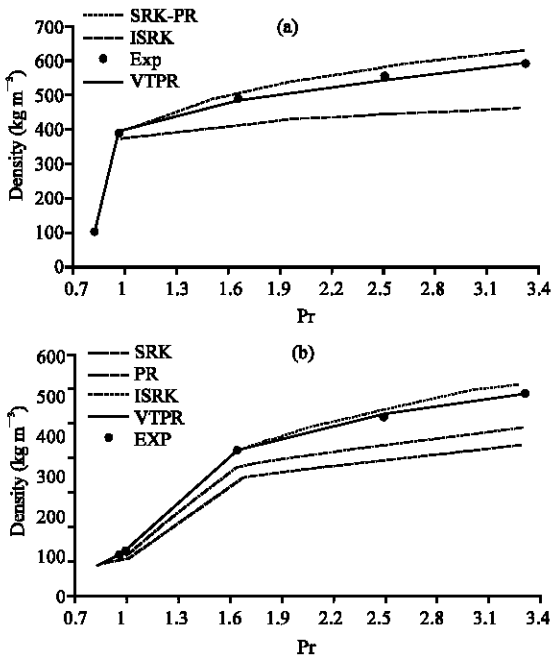| $\rho(VTPR)$ (kg m^{-3})	ρ_{exp} (kg m^{-3})	MPR))υ (cSt)	$_{exp}\upsilon$ (cSt)	T(K)
890	910	4.998	5.2	80
876.25	884.5	3.56	3.7	100
861.01	876	2.761	2.82	120
859.3	868.2	1.61	1.67	140

Fig. 3: Density of n-hexane in near-critical and supercritical region:(a) T = 523.15 : (b) T = 498.15 Exp. data (Abdulagatov *et al.*, 2001)
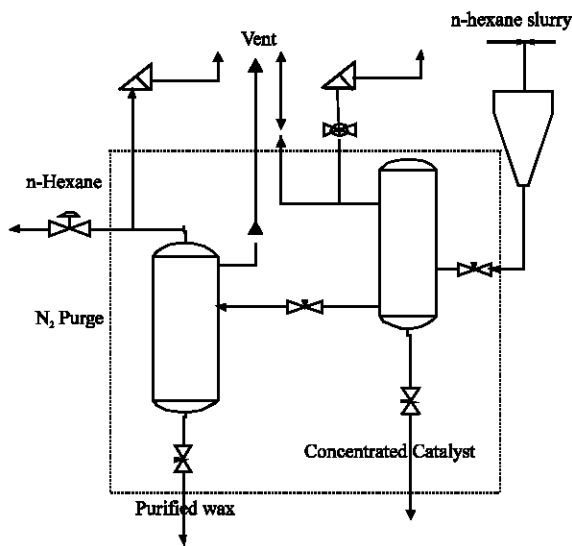

Fig. 4: Wax/catalyst separation apparatuses

eader as solvent and the ventilation header. A vessel where slurry (wax+catalyst) flow was delivered and mixed with the 50°C n-hexane was used as a feeder tank.

The main section, in which the operating conditions were adjusted to the near-critical hexane pressure and temperature, was a high pressure and temperature vessel with height to the diameter ratio of 10 and the inner volume of 400 cc. It was just a simple settler also called contactor, in which the catalyst particles with 3 μm mean diameter were separated from the viscous slurry, by near-critical hexane.

All experiments were conducted in the same retention time of 5 min. At the end of each test, the flow including hexane/wax mixture and any unsettled catalysts was conducted into the flash drum, where hexane recovery takes place.

In the other hand, any precipitated catalysts were ashed at 750°C for 2 h, so that the separation efficiency was gained through the weighting the remained catalyst in the ashed sample.

RESULTS AND DISCUSSION

The feasibility and the manner of separation test results was studied through estimating densities and viscosities for wax-hexane mixture using the proposed EOS's. By this way, not only test results could be used to insure the stated EOS's for near-critical processes, but also by relying on such equations, through the investigations and outcomes gained in previous sections of this work, the feasibility of wax/catalyst separation could also be studied.

The operating conditions for the experiments and physical properties calculations were adjusted to the near-critical hexane pressure and temperature, including liquid, gas and supercritical phase. All three states of hexane were also investigated for three points at 1.05, 1.1 and 1.15 % around its critical point.

The effect of amount of applied hexane, either on physical properties or on separation efficiency was taken in to consideration in all experiments by the hexane to wax ratio parameter. This investigation was carried out for three various hexane to wax ratio as 0, 2 and 4. Wax/catalyst separation test results in addition with density and viscosity calculation are listed in Table 3.

It could be interpreted from Table 3 that, in near-critical hexane operating condition, the highest separation efficiency values and lowest amounts of densities/viscosities for hexane/wax mixture, respectively belonged to the supercritical, gas and liquid hexane use. In addition, moving to the higher pressures and

Table 3: Wax/catalyst separation results and wax+ hexane densities/viscosities estimation Exp. data (Amiri and Khakdaman, 2005)

Exparation efficiency (%)	Viscosity (cP)	Density (kg m^{-3})	Hexane to wax ratio	P (bar)	T (C)	Hexane state
2.83	1.216	742.43	0	31.76	246.12	Supercritical
75.32	0.100	253.74	2			
79.02	0.080	182.27	3			
33.11	1.072	726.49	0	33/275	257.84	
86.92	0.015	161.73	2			
88.51	0.01	122.41	3			
35.00	0.951	675/285	0	34.79	269.56	
90.71	0.005	116.58	2			
93.28	0.0004	98.999	3			
32.87	1.215	742.62	0	28.73	246.12	Gas
58.32	0.16	449.67	2			
58.40	0.159	449.601	3			
33.02	1.0717	734.89	0	27.225	257.84	
84.72	0.02	296.27	2			
84.80	0.0198	296.30	3			
34.09	0.950	727.12	0	25.71	269.56	
82.52	0.0092	228.03	2			
82.58	0.0091	228.01	3			
29.44	1.5985	758.00	0	28.73	222.68	Liquid
67.93	0.1366	505.16	2			
72.01	0.1097	474.00	3			
28.31	1.8557	765.65	0	27.225	210.96	
59.73	0.1538	525.52	2			
69.97	0.12336	497.48	3			
27.50	2.1756	773.27	0	25.71	199.24	
53.60	0.17027	543.28	2			
67.52	0.1364	517.18	3			

temperatures in each investigated phases, the values of the physical properties approached to lower one's together with growing the separation efficiencies amounts. Besides, it could be defined that by increase of hexane to wax ratio, the densities and viscosities were brought to the lower values. The separation efficiencies also approached to the higher amounts, simultaneously. Similar behavior was observed when using the hexane liquid state, whereas, during the gaseous hexane application, the values of viscosity and density stayed constant together with the efficiency values. Such results are obvious in Table 3.

This outcome could be interpreted by knowing the fact of partial solubility of gaseous hexane in liquid phase (wax) opponent to its complete solubility of other hexane states, such as liquid and the supercritical. It should be noted that, still there is no accurate equation derived for estimating solubility of near-critical hexane in viscous wax media. However, the effect of such solubility on separation efficiency results were studied and shown through calculation of wax-hexane mixture's density/viscosity by proposed EOS's.

All the results either gained by experiments or by theoretical EOS's calculations, should be compared to the general Stoke's law used in sedimentation. according to the Stoke's law, settling rate of a solid (catalyst) in a fluid is proportional to the density difference between the solid and fluid and is inversely related to the fluid viscosity.

Regarding this theorem, all viscosities and densities values in Table 3 based on the proposed EOS's estimations, were in consisted with the wax/catalyst separation efficiencies.

All mentioned earlier, could prove the feasibility and the manner of the catalyst/wax separation results using near-critical hexane and the accuracy of proposed EOS's in conjunction with Stoke's law certification. By the results obtained in this investigation, it seems that, if accurate theoretical correlations are derived first, for fine solids settling in viscous media second, for the solubility of near-critical hydrocarbons in viscous wax; then wax/catalyst separation modeling would be simply conducted by substituting exact values of densities and viscosities in such correlations using VTPR and MPR EOS's.

CONCLUSIONS

- The volume translation PR formula (VTPR) and the MPR EOS-based analog correlation were proposed to improve the accuracy of predicted densities and viscosities for near-critical pure hydrocarbons and their mixtures.
- The generalized correlations provided satisfactory interpretation for wax/catalyst separation results in near-critical hexane operating condition, regarding the accurate values of hexane and wax densities and viscosities estimation.

- According to the wax/catalyst experimental tests and density/viscosity estimation by proposed EOS's, the highest separation efficiencies were related to the case using hexane in supercritical state. The values of densities and viscosities, respectively calculated by the VTPR and MPR EOS's, certificated such result, as the lowest estimated values were associated with the supercritical state. The outcomes of this research were also in line with the Stoke's theorem.
- Wax/catalyst separation modeling can be simply conducted by substituting VTPR and MPR model for density/viscosity calculations, in accurate correlations, which would be derived for related sedimentation

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