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Bubble and Dew Points of Carbon Dioxide + a Five-Component Synthetic Mixture: Experimental Data and Modeling with the PPR78 Model

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In this work, a high-pressure visual cell is used to measure bubble and dew pressures and temperatures of a five-component synthetic mixture in the presence of CO_2. The synthetic mixture was gravimetrically prepared and contains octane, hexadecane, methylcyclohexane, *cis*-decalin, and methylbenzene (toluene). Seven quantities of CO_2 were injected in the synthetic mixture, and for each amount of CO_2 bubble and dew pressures were measured at temperatures ranging from (292.95 to 373.35) K. The corresponding pressures vary between (20 and 165) bar. A total of 61 experimental bubble and dew pressures are reported. The experimental data are compared with results obtained using the PPR78 model in which the temperature-dependent binary interaction parameters are predicted by a group contribution method. Results clearly indicate that the PPR78 model is a good predictive model because the average absolute deviation is about 5.5 bar between experimental and calculated pressures. Results are presented at constant composition in (P,T) diagrams. They are also presented at constant temperature in (P, x_{CO_2}) diagrams by considering the CO_2 and the synthetic mixture as a pseudobinary mixture.

Introduction

Phase equilibrium data of multicomponent mixtures are relatively scarce in the open literature. However, such data and their correlation are essential for the design and the improvement of many industrial operations like reservoir fluid exploitation, refining, distillation, or transport. For example, the gas injection technique is now routinely employed to enhance oil recovery. However, to simulate such experiments, one needs models capable of accurately predicting the equilibrium properties, that is, the influence of the presence, sometimes in large amounts, of injection gas like CO_2 on the oil composition and properties.[1,2] Because of the complexity of reservoir fluid composition, a good alternative is to study synthetic mixtures with a relatively small number of components.[3,4] The problem of the mixture chemical characterization is thus eliminated. Experimental data obtained on these kind of mixtures are useful to compare equation of state (EOS), predictive models, or to fit model parameters because there is no approximation linked to the fluid composition.

The objective of the present work is two-fold: first to obtain experimental data on a hydrocarbon mixture in which CO_2 is injected, and second to test the ability of the PPR78 model,[5−8] recently developed by Jaubert et al., to correlate these data. The experiments performed in this study (CO_2 injection in a hydrocarbon mixture) are similar to what is called a swelling test[9,10] in the petroleum industry. Nevertheless, the swollen volumes were not determined and only saturation pressures were measured and calculated. In this work, the studied synthetic mixture contains two paraffins (octane and hexadecane), two naphthenes (methylcyclohexane and *cis*-decalin), and an aromatic compound (methylbenzene (toluene)). The experiments were conducted in a high-pressure cell with a sapphire window allowing us direct observation of the phase transition.

Table 1. Suppliers and Purities of the Components Used in This Study

component	supplier	purity/%
carbon dioxide	Messer France	>99.9
octane	Fluka	>99.5
hexadecane	Aldrich	>99.0
methylcyclohexane	Aldrich	>99.0
cis-decalin	Aldrich	>99.0
methylbenzene	Aldrich	>99.5

Experimental Section

Materials. Suppliers and purities of the six pure components used in this study are given in Table 1. All the chemicals had a minimum purity of 99 % and were used without any further purification.

Apparatus and Procedure. Bubble and dew pressures were measured using the apparatus schematically described in Figure 1.

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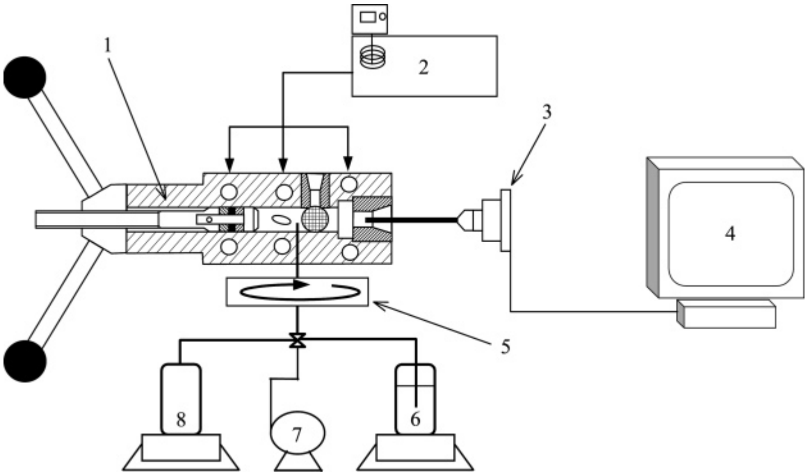

Figure 1. Schematic description of the high-pressure cell used in this study: 1, high-pressure variable volume cell; 2, thermostated bath; 3, endoscope and video camera; 4, TV connected to the camera; 5, magnetic stirrer; 6, liquid weighing; 7, vacuum pump; 8, weighing of the CO_2 tank.

Table 2. Composition of the Five-Component Synthetic Mixture

component	mole fraction
octane	0.4000
hexadecane	0.0500
methylcyclohexane	0.3000
cis-decalin	0.0500
methylbenzene	0.2000

More details concerning the equipment used can be found elsewhere.[11,12] The technique used to carry out vapor−liquid equilibrium measurements was based on a synthetic method that avoids sampling and analyses of both phases. The experimental apparatus was essentially made up of a variable volume high-pressure cell (Top Industrie S.A.). One end of the cell was closed by a movable piston and the other end by a sapphire window allowing a visual observation of the equilibrium cell. A second sapphire window was fixed on the cylinder wall to illuminate the fluid with an optical fiber. A video acquisition system, consisting of an endoscope and a video camera, was placed behind the sapphire window and connected to a screen for observation inside the measuring cell. The mixture was permanently homogenized thanks to a small magnetic bar and an external magnetic stirrer. The cell volume ranged from (8 to 30) cm^3 and the internal temperature was kept constant by circulating a thermo-regulated heat-carrier fluid through three lines in the cell. The temperature was precisely measured with a thermometer (AOIP brand, model PN5207, accuracy of 0.1 K) connected to a calibrated platinum resistance inserted in the cell, while the pressure was measured by a piezoresistive silicon pressure transducer (Kulite, model HEM375, working in the full scale range of 1 to 1000 bar) directly placed inside the cell to minimize dead volumes. Calibration of the pressure transducer was done in the temperature range (263.15 to 383.15) K by means of a dead-weight gauge (Budenberg) with an accuracy of 0.02 %.

The synthetic mixture was gravimetrically prepared outside the cell under atmospheric pressure and ambient temperature because the five hydrocarbons were liquid at ambient temperature. A precision balance (Ohaus) with an accuracy of 0.0001 g was used to measure the mass of each liquid hydrocarbon added. The mixture was stirred during the successive additions of the components. The composition of the prepared synthetic mixture is given in Table 2.

The equilibrium cell was first loaded with a known amount of liquid synthetic mixture by vacuum extraction. The exact mass of synthetic mixture introduced in the cell was determined using the precision balance previously mentioned. To avoid the calculation of the quantity of liquid that could remain in the injection tube linking the balance to the three-way valve (see Figure 1), this small tube was filled with liquid before the beginning of an experiment. CO_2 was then added under pressure. For this purpose, the gas was initially loaded in an aluminum reservoir tank fixed on the plate of a high mass/high precision balance (Sartorius). The maximum weighing capacity of this balance was 2000 g, and its accuracy was 0.001 g. The CO_2 reservoir tank was connected to the cell by a flexible high-pressure capillary. The exact mass of CO_2 injected in the cell was thus determined by weighing the reservoir tank during the filling. After these filling operations, precise mole fractions of the compounds contained in the cell (i.e., CO_2 + synthetic mixture) could be calculated.

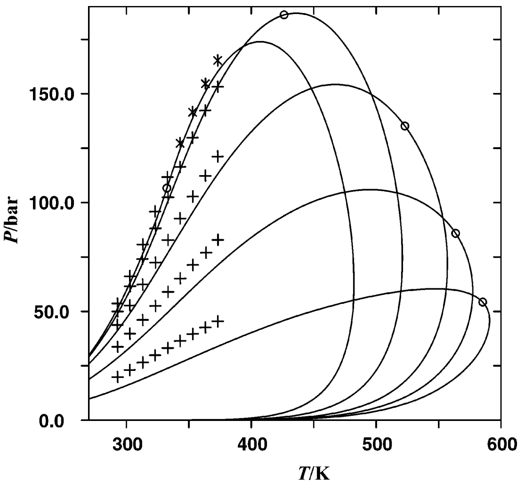

Figure 2. (*P*,*T*) phase envelopes for various quantities of CO_2 injected in the synthetic mixture. CO_2 mole fractions are: 0.2036 (smallest envelope); 0.4021; 0.6008; 0.8000; and 0.9101. +, experimental bubble points; ∗, experimental dew points; solid lines, phase envelopes predicted with the PPR78 model; O, calculated mixture critical point.

When the desired temperature cell was reached, the pressure was slowly increased until the system became homogeneous. Because of supersaturation effects, the phase boundaries were evaluated by determining the disappearance conditions of one phase instead of its appearance, meaning that measurements were carried out by observing the disappearance of the vapor phase (for a bubble point) or the liquid phase (for a dew point) at constant temperature. Reproducibility of the pressure disappearance measurements was 0.2 bar.

Table 3. Experimental Bubble and Dew Pressures for the CO_2 + Synthetic Mixture System at Seven Different CO_2 Mole Fractions[a]

$x_{CO_2} = 0.2036$		$x_{CO_2} = 0.4021$		$x_{CO_2} = 0.6008$		$x_{CO_2} = 0.8000$		$x_{CO_2} = 0.8723$		$x_{CO_2} = 0.9101$		$x_{CO_2} = 0.9601$	
T/K	*P*/bar	*T*/K	*P*/bar	*T*/K	*P*/bar	*T*/K	*P*/bar	*T*/K	*P*/bar	*T*/K	*P*/bar	*T*/K	*P*/bar
293.05	19.7	293.05	33.6	292.95	43.7	293.05	49.9	292.95	51.9	293.05	53.5	292.95	55.1
302.85	22.9	302.95	39.6	302.95	52.6	303.05	61.4	302.85	63.9	302.95	66.0	302.95	68.7
313.15	26.4	313.05	45.9	313.05	62.2	312.95	73.9	312.85	77.5	313.15	80.6	313.15	84.0
323.05	29.7	323.25	52.4	323.25	72.3	323.35	88.0	323.25	92.8	323.05	95.7	322.95	98.5
333.15	33.1	333.35	58.8	333.35	82.6	333.35	102.2	332.95	107.7	332.95	111.5	333.25	**115.3**
343.05	36.3	343.15	64.9	343.05	92.6	343.05	116.1	342.95	122.4	343.05	**127.0**	343.05	**130.4**
353.15	39.5	353.25	71.2	353.15	102.6	352.95	129.6	353.25	136.5	353.05	**141.3**	352.95	**142.9**
363.25	42.5	362.85	76.8	363.25	112.2	363.15	142.1			363.35	**154.4**	363.05	**153.4**
373.25	45.3	373.35	82.7	373.15	121.0	373.15	153.0			373.15	**165.1**	373.05	**161.2**

[a] Bold data are dew pressures; the rest are bubble pressures.

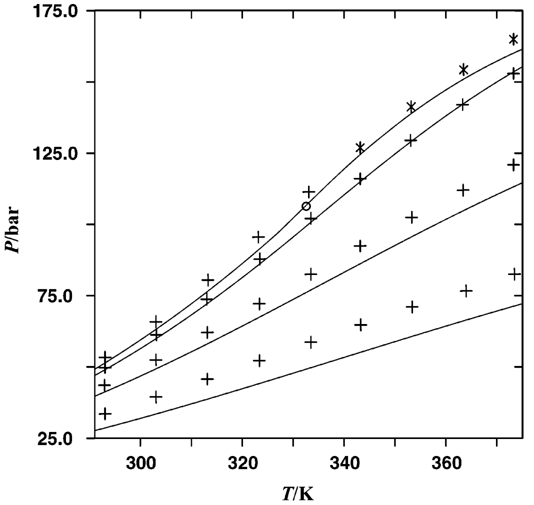

Figure 3. Enlargement of Figure 2 around the measured bubble or dew points for four amounts of CO_2 injected in the synthetic mixture. CO_2 mole fractions are: 0.4021 (lowest points and lowest curve); 0.6008; 0.8000; and 0.9101. +, experimental bubble points; $*$, experimental dew points; solid lines, phase envelopes predicted with the PPR78 model; O, calculated mixture critical point.

Results and Modeling

A total of 61 experimental points were measured: 52 bubble pressures and 9 dew pressures. The CO_2 + synthetic mixture system was studied at 7 different mole fractions of CO_2 ranging from (0.2036 to 0.9601). The results are given in Table 3 and are partially shown in Figures 2 and 3 with calculated phase envelopes.

The PPR78 model was used to predict the experimentally determined pressures. The PPR78 model relies on the Peng–Robinson equation of state (PR-EOS) as published by Peng and Robinson in 1978.[13] For a pure component, the PR78 EOS is

$$P = \frac{RT}{V - b_i} - \frac{a_i(T)}{V(V + b_i) + b_i(V - b_i)} \tag{1}$$

with

$$\begin{cases} R = 8.314472\ \text{J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1} \\ b_i = 0.0777960739\,\dfrac{RT_{c,i}}{P_{c,i}} \\ a_i = 0.457235529\,\dfrac{R^2T_{c,i}^2}{P_{c,i}}\left[1 + m_i\left(1 - \sqrt{\dfrac{T}{T_{c,i}}}\right)\right]^2 \\ \text{if } \omega_i \le 0.491 \quad m_i = 0.37464 + 1.54226\omega_i - 0.26992\omega_i^2 \\ \text{if } \omega_i > 0.491 \quad m_i = 0.379642 + 1.48503\omega_i - 0.164423\omega_i^2 + 0.016666\omega_i^3 \end{cases} \tag{2}$$

P is the pressure, *R* is the ideal gas constant, *T* is the temperature, *a* and *b* are EOS parameters, *V* is the molar volume, T_c is the critical temperature, P_c is the critical pressure, and ω is the acentric factor.

To apply such an EOS to mixtures, mixing rules for *a* and *b* parameters need to be defined. The PPR78 model uses classical mixing rules:

$$\begin{cases} a = \sum_{i=1}^{N}\sum_{j=1}^{N} z_i z_j \sqrt{a_i a_j}\,(1 - k_{ij}(T)) \\ b = \sum_{i=1}^{N} z_i b_i \end{cases} \tag{3}$$

z_k represents the mole fraction of component "*k*" in a mixture, and *N* is the number of components in the mixture. In eq 3, the summations are over all chemical species. $k_{ij}(T)$, whose choice is difficult even for the simplest systems, is the so-called binary interaction parameter characterizing molecular interactions between molecules "*i*" and "*j*". When *i* equals *j*, k_{ij} is zero. In the PPR78 model (predictive, 1978 PR-EOS), k_{ij}, which depends on temperature, is calculated by a group contribution method[5−8] through the following expression:

$$k_{ij}(T) = \frac{-\dfrac{1}{2}\sum_{k=1}^{N_g}\sum_{l=1}^{N_g}(\alpha_{ik} - \alpha_{jk})(\alpha_{il} - \alpha_{jl})A_{kl}\cdot\left(\dfrac{298.15}{T}\right)^{(B_{kl}/A_{kl} - 1)} - \left(\dfrac{\sqrt{a_i(T)}}{b_i} - \dfrac{\sqrt{a_j(T)}}{b_j}\right)^2}{2\,\dfrac{\sqrt{a_i(T)\cdot a_j(T)}}{b_i\cdot b_j}} \tag{4}$$

In eq 4, a_i and b_i are calculated by eq 2. N_g is the number of different groups defined by the method (for the time being, 12 groups are defined and $N_g = 12$). α_{ik} is the fraction of molecule *i* occupied by group *k* (occurrence of group *k* in molecule *i* divided by the total number of groups present in molecule *i*). $A_{kl} = A_{lk}$ and $B_{kl} = B_{lk}$ (where *k* and *l* are two different groups) are constant parameters[5−8] ($A_{kk} = B_{kk} = 0$). As can be seen, to calculate the k_{ij} parameter between two molecules *i* and *j* at a selected temperature, it is necessary to know the critical temperature of both components ($T_{c,i}$, $T_{c,j}$), the critical pressure of both components ($P_{c,i}$, $P_{c,j}$), the acentric factor of each component (ω_i, ω_j), and the decomposition of each molecule into elementary groups (α_{ik}, α_{jk}). Table 4 presents the pure fluid physical properties (T_c, P_c, and ω) of the six components used in this study. They originate from Poling at al.[14]

Table 4. Critical Temperature (T_c), Critical Pressure (P_c), and Acentric Factor (ω) of the Pure Components Used in This Study[14]

component	T_c/K	P_c/bar	ω
carbon dioxide	304.12	73.74	0.225
octane	568.70	24.90	0.399
hexadecane	723.00	14.00	0.718
methylcyclohexane	572.19	34.71	0.235
cis-decalin	703.60	32.00	0.276
methylbenzene	591.75	41.08	0.264

Today, 12 groups are defined in the PPR78 model. Groups 1 to 6 are relative to alkanes, groups 7 to 9 are relative to aromatic compounds, and groups 10 and 11 are naphthenic groups. Recently, a new group was added: CO_2 (group 12). Today, it is thus possible to estimate at any temperature the k_{ij} between two components in any mixture containing paraffins, naphthenes, aromatics, and CO_2. More details and explanations concerning the PPR78 model (decomposition of molecules into groups, group contribution parameters, temperature dependence of k_{ij}) can be found elsewhere.[5−8] In Figures 2 and 3, phase envelopes are calculated by the PPR78 model, meaning that 15 k_{ij} are predicted using eq 4 at a given temperature. The average absolute deviation is

$$\Delta P = \frac{\sum_{i=1}^{n} |P_{\text{exp}} - P_{\text{cal}}|}{n} \approx 5.5\ \text{bar} \tag{5}$$

while the average relative deviation is

$$\Delta P = \frac{\sum_{i=1}^{n} \left| \frac{P_{\text{exp}} - P_{\text{cal}}}{P_{\text{exp}}} \right|}{n} \approx 9.2\ \% \tag{6}$$

where n is the number of experimental data, and P_{exp} and P_{cal} are experimental and calculated saturation pressures.

The accuracy of the PPR78 model is satisfactory because an average deviation of $\Delta P \approx 9.2\ \%$ (i.e., 5.5 bar) is observed between calculated and experimental bubble or dew pressures. The highest deviation reaches 11.2 %. To complete the investigation of the studied system, isothermal (P, x_{CO_2}) curves were derived. However, as phase transition measurements were not carried out at fixed temperatures, these curves cannot be obtained directly from the original data given in Table 3. Interpolated values at fixed temperatures were obtained by smoothing the original data using low-degree polynomial fits. The interpolated values are given in Table 5 and are partially shown in Figures 4 and 5 with seven isothermal curves predicted by the PPR78 model in the temperature range (293.15 to 373.15) K. In such curves, the injection gas (CO_2) and the synthetic mixture are considered as a pseudobinary mixture. The (P, x_{CO_2}) curves thus obtained, usually called swelling diagrams, have the same appearance as those obtained for a real binary mixture, although several differences must be noted. For example, the critical point is not at the top of the isothermal curve. Figures 4 and 5 show that the critical point is located for the studied system slightly on the left of the pressure maximum. These figures confirm that predictions obtained with the PPR78 model are accurate even if some deviations appear when the amount

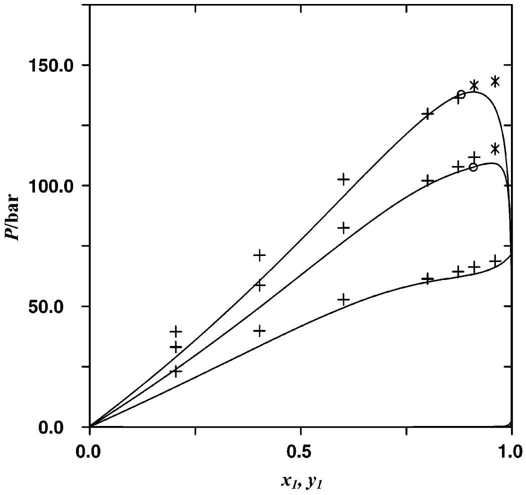

Figure 4. Bubble and dew pressures versus CO_2 mole fraction for the pseudobinary system CO_2 (1) + synthetic mixture (2) at three different temperatures: T_1 = 303.15 K, T_2 = 333.15 K, and T_3 = 353.15 K. +, interpolated experimental bubble points; ∗, interpolated experimental dew points; solid lines, bubble and dew curves predicted with the PPR78 model; O, calculated mixture critical point.

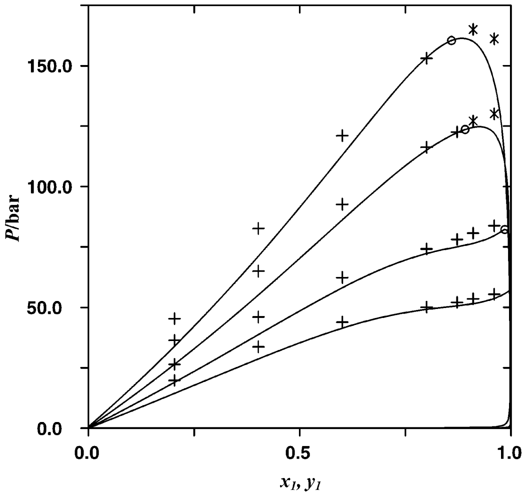

Figure 5. Bubble and dew pressures versus CO_2 mole fraction for the pseudobinary system CO_2 (1) + synthetic mixture (2) at four different temperatures: T_1 = 293.15 K, T_2 = 313.15 K, T_3 = 343.15 K, and T_4 = 373.15 K. +, interpolated experimental bubble points; ∗, interpolated experimental dew points; solid lines, bubble and dew curves predicted with the PPR78 model; O, calculated mixture critical point.

Table 5. Smoothed (P, x_{CO_2}) Data for the CO_2 + Synthetic Mixture System at Nine Different Temperatures Calculated from Table 3[a]

	P/bar								
x_{CO_2}	293.15 K	303.15 K	313.15 K	323.15 K	333.15 K	343.15 K	353.15 K	363.15 K	373.15 K
0.2036	19.8	23.1	26.4	29.8	33.1	36.3	39.5	42.5	45.3
0.4021	33.7	39.7	46.0	52.3	58.7	65.0	71.1	77.0	82.6
0.6008	43.9	52.8	62.3	72.3	82.4	92.6	102.6	112.1	121.0
0.8000	50.1	61.4	74.1	87.9	102.1	116.2	129.7	142.2	153.0
0.8723	52.1	64.3	78.1	92.8	107.8	122.5	136.3		
0.9101	53.5	66.3	80.7	96.0	111.7	**127.1**	**141.5**	**154.3**	**165.0**
0.9601	55.4	68.8	83.7	99.5	**115.2**	**130.0**	**143.2**	**153.8**	**161.1**

[a] Bold data are dew pressures; the rest are bubble pressures

of injected CO_2 is small. Prediction seems to be quite good in the vicinity of the critical point and at the top of the isothermal curves. This is important because the top of the curve represents the first contact minimum miscibility pressure[15−18] (FCMMP). At a pressure higher or equal to the FCMMP, all possible mixtures of the two fluids (CO_2 + synthetic mixture) are single phase fluids at the considered temperature.

Conclusion

New high-pressure saturation points for a system containing CO_2 and five hydrocarbons (octane, hexadecane, methylcyclohexane, *cis*-decalin, and methylbenzene) have been measured in a visual cell. Fifty-two bubble and nine dew pressures were measured in the temperature range (292.95 to 373.25) K. In addition, the experimental data have been used to assess the performance of the PPR78 model recently described in the literature. Saturation pressures predicted by this model were found to deviate up to 5.5 bar (9.2 %). Taking into account the relative asymmetry of the studied system and the fact that no parameters were fitted, it can be concluded that the obtained results are satisfactory.

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