ТЕОРЕТИЧЕСКИЕ ОСНОВЫ ХИМИЧЕСКОЙ ТЕХНОЛОГИИ THEORETICAL BASES OF CHEMICAL TECHNOLOGY

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Drawing PT-phase envelopes and calculating critical points for multicomponent systems using flash calculations

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Objectives. This study aims to draw PT-phase envelopes and calculate the critical points for multicomponent systems using flash calculations.

Methods. Flash calculations with an equation of state and a mixing rule were used to construct phase envelopes for multicomponent systems. In general, the methodology uses the Soave–Redlich–Kwong equation of state and Van der Waals mixing rules; and the Peng–Robinson equation of state with Wong–Sandler mixing rules and the non-random two-liquid activity coefficient model. **Results.** The method was applied to the following mixtures: ethane (1)–butane (2) (four different compositions); ethane (1)–propane (2) (four different compositions); butane (1)–carbon dioxide (2) (three different compositions); C2C3C4C5C6 (one composition); isobutane–methanol–methyl tertbutyl ether–1-butene (one composition); and propylene–water–isopropyl alcohol–diisopropyl ether (one composition).

Conclusions. Our results agreed to a large extent with the experimental data available in the literature. For mixtures that contained CO_2 , the best results were obtained using the Peng–Robinson equation of state and the Wong–Sandler mixing rules. Our methodology, based on flash calculations, equations of state, and mixing rules, may be viewed as a shortcut procedure for drawing phase envelopes and estimating critical points of multicomponent systems.

Keywords: flash calculations, critical points, phase envelopes.

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Построение РТ-фазовых диаграмм и расчет критических точек для многокомпонентных систем с использованием флэш-вычислений

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Цели. Построение РТ-фазовых диаграмм и расчет критических точек для многокомпонентных систем с использованием флэш-вычислений.

Методы. Для построения фазовых диаграмм многокомпонентных систем использовали флэш-вычисления на основе уравнения состояния и правила смешения. В общем случае методология использует уравнение состояния Соаве–Редлиха–Квонга и правило смешения Ван дер Ваальса; уравнение состояния Пенга–Робинсона и правило смешения Вонга–Сэндлера, а также неслучайная двужидкостная модель активных коэффициентов.

Результаты. Метод был применен к следующим смесям: этан (1)-бутан (2) (четыре разных состава); этан (1)-пропан (2) (четыре разных состава); бутан (1)-диоксид углерода (2) (три разных состава); C2C3C4C5C6 (один состав); изобутан-метанол-метил-трет-бутиловый эфир-1-бутен (один состав); и пропилен-вода-изопропиловый спирт-диизопропиловый эфир (ДИПЭ) (один состав).

Выводы. Согласно нашим результатам, метод флэш-вычислений, базирующийся на уравнении состояния и правилах смешения, используемый для построения фазовых диаграмм, на основе которых проводится оценка критических точек для многокомпонентных смесей, хорошо согласуется с экспериментальными данными, имеющимися в литературе. Для смесей, содержащих СО₂, лучшие результаты получены с использованием уравнения состояния Пенга–Робинсона и правила смешения Вонга–Сэндлера.

Ключевые слова: флэш-вычисления, критические точки, фазовые диаграммы.

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INTRODUCTION

The knowledge of phase envelopes and critical points is valuable for the calculation of phase equilibria and solving various problems in chemical engineering. Also, from a modeling point of view, knowledge of critical data is of paramount importance, as it provides information about real fluids, as well as characterizes phase change boundaries in mixtures with the help of phase diagrams. Through the combination of excess properties, phase equilibrium data, and critical data, it is possible to encompass the major thermodynamic aspects of multicomponent mixtures. In this paper, we present a methodology of constructing phase envelopes of multicomponent mixtures, from which the critical points follow. In the available literature, there is a variety of methods (both theoretical and experimental) to construct and estimate critical points.

A methodology has been proposed [1] to calculate critical points of multicomponent mixtures using a modification of the Gibbs plane tangent. A group of researchers [2] solved the Heidemann and Khalil formulation [3] using a Newton method with defined intervals to calculate critical points of binary and ternary mixtures.

Calculations of the critical points of multicomponent mixtures have been reported [4–6], where the optimization problem was solved in such a way that the function to minimize is the Gibbs plane tangent criterion. Other researchers [7] have also solved the Heidemann and Khalil formulation with a simulated annealing algorithm [8, 9] and determined the critical properties of some multicomponent mixtures. Besides, predictive equations of state have been used [10] to construct natural gas phase envelopes and to calculate its critical points. A general algorithm has been suggested [11] for the calculation of dew and bubble points for multicomponent mixtures, representing a continuation method that can be used to draw phase envelopes and estimate critical points. The Soave–Redlich–Kwong equation of state (SRK EoS) and its derivatives have been used [12] to construct the phase envelope of binary mixtures, with dew and bubble point calculations.

Another study [13] reports the use of the perturbed-chain statistical associating fluid theory (PC-SAFT) equation of state to calculate critical points of multicomponent mixtures containing hydrocarbons and non-hydrocarbon components. Furthermore, the simplified critical-point criteria [14] have been presented for some multicomponent systems, based on a thermodynamic model where the Helmholtz energy depends on the composition average model parameters. There is also a report [15] on a method for calculation of critical points for refrigerant mixtures, using mixture models based on Helmholtz energy equations of state. The Peng-Robinson (PR), SAFT, and PC-SAFT equations of state have been used [16] for calculating critical points of hydrocarbon mixtures. The development of a new flow apparatus is reported [17] for the experimental determination of critical points of pure components and binary mixtures. Moreover, a dynamic-synthetic apparatus [18] can be used to determine the critical properties of pure and multicomponent mixtures.

From a theoretical point of view, the aforementioned methods for drawing phase envelopes and calculating critical points of multicomponent mixtures require sophisticated mathematical procedures and complex algorithms. It is possible to do the same task for some mixtures, using the flash evaporation that is a well-known operation of separation in chemical engineering [19, 20]. The simple mathematical model of this operation permits several calculations, such as bubble and dew points, molar fractions in the phases (vapor and liquid) for constructing vapor–liquid equilibrium diagrams for mixtures, evaporated fraction, isotherms and vapor pressures versus temperature for pure substances.

In this report, we used flash calculations with an equation of state and a mixing rule to construct phase envelopes for multicomponent systems, from which the critical points of mixtures are estimated. This methodology was applied to some binary and multicomponent mixtures reported in the literature, and our results are in good agreement with the reported data. In general, the methodology uses the SRK EoS and Van der Waals (VdW) mixing rules; but for mixtures that contain CO₂, the best results were obtained with the PR EoS and Wong–Sandler mixing rules (WS MR). The presented methodology might be seen as a shortcut procedure when an easy method for drawing phase envelopes and estimating critical points of a multicomponent system is needed.

MATERIALS AND METHODS

Suppose that a stream F is fed to a flash evaporator with a global composition $z = [z_1, z_2, ..., z_n]$ (molar fraction) of n components. The pressure P and temperature T are constant in the evaporator. Depending on the nature of the components and the feed composition, there is a vapor phase and one or two liquid phases. The first situation is shown in Fig. 1. The outlet streams, V and L, are in equilibrium at P and T.



Fig. 1. Flash evaporator scheme.

Applying the material balance for the *i*-component, it follows that

$$Fz_i = Lx_i + Vy_i, i = 1, 2, \dots, n$$
 (1)

Since the outlet streams are in equilibrium, then

$$y_i = K_i x_i, i = 1, 2, \dots, n$$
 (2)

A total material balance gives

$$F = L + V . \tag{3}$$

Let α be

$$\alpha = \frac{V}{F}.$$
(4)

From equations (3) and (4) we obtain

$$z_{i} = (1 - \alpha)x_{i} + \alpha K_{i}x_{i}, i = 1, 2, \dots, n.$$
(5)

Plugging (5) into (1) gives

$$z_{i} = (1 - \alpha)x_{i} + \alpha y_{i}, i = 1, 2, \dots, n.$$
(6)

Using (2), an equivalent equation is

$$z_{i} = (1 - \alpha)x_{i} + \alpha K_{i}x_{i}, i = 1, 2, \dots, n.$$
(7)

The molar fraction condition gives the following equations in both phases:

$$\sum_{i=1}^{n} x_i = 1, \ \sum_{i=1}^{n} y_i = 1.$$
(8)

From (8)

$$\sum_{i=1}^{n} x_i - \sum_{i=1}^{n} y_i = 0.$$
(9)

To draw the phase envelope for a multicomponent mixture, the following procedure is implemented. From equation (4), if $\alpha = 0$, there is no vapor phase as V = 0 and only the liquid phase exists. Thus, bubble point calculation is made and a bubble point curve is obtained. If $\alpha = 1$, there is no liquid phase as L = 0 and only the vapor phase exists. Then, a dew point calculation is made and a dew point curve is obtained. By drawing the two curves in the same coordinates (PT-system), we observe their intersection in the critical point of the mixture and the PT-phase envelope is obtained.

Next, the construction of a bubble point curve is explained. In this case, $\alpha = 0$ and the only known data is $z = [z_1, z_2, ..., z_n]$ that is held constant. It is necessary to calculate the equilibrium compositions from equation (2), that depends on K_i and this is unknown, but it is also possible to use an EoS such as SRK or PR (with the appropriate mixing rules), that involves the equilibrium compositions that is the $\phi - \phi$ method [21]:

$$K_i = \phi_{\rm liq} \,/\, \phi_{\rm vap} \,. \tag{10}$$

When the WS MR are used in PR EoS [22], the excess Helmholtz free energy, at infinite pressure, is equal to the excess Gibbs free energy, and the non-random two-liquid (NRTL) activity coefficient model is adopted for the calculation of required parameters for WS MR [23].

Combining equations (6), (7), (9), (10) (from EoS), a set of nonlinear equations containing temperature, pressure, equilibrium compositions, and constants as unknowns is obtained and needs to be solved. For the solution of the final nonlinear equation system, a *Matlab* code that incorporates its built-in function *fsolve* was written, and it was used for drawing the dew point curve for which $\alpha = 1$. For obtaining the points in both curves, is varied and is calculated along with the equilibrium compositions and corresponding constants. The initial equilibrium compositions and temperature must be guesstimated for testing convergence. This is necessary to avoid unreal results.

RESULTS AND DISCUSSION

In this section, the results obtained by applying the exposed methodology to several mixtures are shown. The relative error ε_{μ} is calculated as follows:

$$\varepsilon_r = \left| \frac{\text{Exp. value} - \text{Calc. value}}{\text{Exp. value}} \right| \times 100.$$
(11)

Ethane (1)-n-butane (2) system

This system was studied in four different compositions. For drawing the phase envelopes for this system, SRK EoS with VdW mixing rule and a binary interaction parameter $k_{ij} = 0.0$ were used. Figure 2 shows the graphical results for this system.

Table 1 summarizes the results for critical points of the studied system and from this, we conclude that the calculated data are in good agreement with the experimental data.

Ethane (1)-propane (2) system

This system was studied in four different compositions. For drawing the phase envelopes for this system, SRK EoS with VdW mixing rule and a binary interaction parameter $k_{ij} = 0.0$ were used. Figure 3 shows the graphical results for this system.

Table 2 summarizes the results for critical points of the studied system, and from this we conclude that the calculated data are in good agreement with the experimental ones.

Multicomponent systems

Three multicomponent systems were studied. For drawing the phase envelopes of such systems, SRK EoS with VdW mixing rule and a binary interaction parameter $k_{ij} = 0.0$ were employed. Figure 4 shows the graphical results of the studied systems. The results reported in [11] are in a graphical form, too. The critical points of the mixtures were read from such graphics. The comparison between both results is shown in Table 3. Despite 10.63% in critical pressure, the results obtained by the presented methodology are in good agreement with the literature.

n-Butane (1)-carbon dioxide (2) system

This system was studied in three different compositions. The binary interaction parameter used in all mixing rules for PR and SRK was 0.133. The following mixing rules (MR) were used: a) VdW; b) WS; c) VdW; d) WS; e) VdW; and f) WS. Figure 5 shows the graphical results for this system. As follows from Fig. 5, the best results were obtained with PR EoS and the NRTL activity model.

Table 4 shows the results for critical points of the studied system, and from this, we conclude that the calculated data are in good agreement with the experimental data, when using PR EoS and the NRTL activity model.



Fig. 2. Ethane (1)–*n*-butane (2) mixtures. Phase envelope using SRK EoS and VdW MR. **a)** $z = [0.5605 \ 0.4395]$; **b)** $z = [0.4402 \ 0.5598]$; **c)** $z = [0.1496 \ 0.8504]$; **d)** $z = [0.2990 \ 0.7010]$.



Fig. 3. Ethane (1)–propane (2) mixtures. Phase envelope using SRK EoS and VdW MR. **a)** $z = [0.1202 \ 0.8798]$; **b)** $z = [0.3598 \ 0.6402]$; **c)** $z = [0.8205 \ 0.1795]$; **d)** $z = [0.8997 \ 0.1003]$.

Table 1. Ethane (1)-*n*-butane (2) mixtures; comparison with experimental results [5]

z_1	$T_{\rm c}({\rm K})$ (calc.)	$P_{\rm c}({\rm kPa})$ (calc.)	<i>T</i> _c (K) (exp.)	$P_{\rm c}({\rm kPa})$ (exp.)	$\varepsilon_{\rm r} T_{\rm c}$	$\varepsilon_{\rm r} P_{\rm c}$
0.5605	379.5	5597	377.54	5598	0.52	0.02
0.4402	392.2	5266	390.67	5266	0.39	0.00
0.1496	415.8	4285	415.72	4285	0.02	0.00
0.2990	404.8	4810	403.82	4810	0.24	0.00

Table 2. Ethane (1)-propane (2) mixtures; comparison with experimental results [5]

z_1	$T_{\rm c}({\rm K})$ (calc.)	$P_{\rm c}({\rm kPa})$ (calc.)	<i>T</i> _c (K) (exp.)	$P_{\rm c}({\rm kPa})$ (exp.)	$\varepsilon_{\rm r} T_{\rm c}$	$\varepsilon_{\rm r} P_{\rm c}$
0.1202	364.6	4447	363.96	4447	0.180	0.000
0.3598	352.4	4798	352.45	4798	0.014	0.000
0.8205	321.4	5065	321.38	5065	0.060	0.000
0.8997	314.7	5017	314.10	5017	0.190	0.000



Fig. 4. Multicomponent mixtures. Phase envelopes using SRK EoS and VdW MR. a) C2C3C4C5C6 mixture, $z = [0.39842 \ 0.29313 \ 0.20006 \ 0.07143 \ 0.03696];$ b) isobutane–methanol–MTBE–1-butene mixture, $z = [0.25 \ 0.25 \ 0.25 \ 0.25];$ c) propylene–water–isopropyl alcohol (IPA)–diisopropyl ether (DIPE) mixture, $z = [0.25 \ 0.25 \ 0.25 \ 0.25].$

System	T _c (K) (calc.)	P _c (bar) (calc.)	<i>T</i> _c (K) (from graphics)	P _c (kPa) (from graphics)	$\varepsilon_{\rm r} T_{\rm c}$	$\varepsilon_{\rm r} P_{\rm c}$
a)	390.71	57	390	56	0.18	1.79
b)	462.52	46.45	459	46	0.77	0.98
c)	483.07	57.2	64	493	2.01	10.63

Table 3. Multicomponent mixtures; comparison with reported data [11]



Fig. 5. *n*-Butane (1)–carbon dioxide (2) mixtures. Phase envelope using PR EoS.
a) VdW MR, z = [0.1694 0.8306]; b) WS MR, z = [0.1694 0.8306];
c) VdW MR, z = [0.3334 0.6666]; d) WS MR, z = [0.3334 0.6666];
e) VdW MR, z = [0.4984 0.5016]; f) WS MR, z = [0.4984 0.5016].

z _i	$T_{\rm c}({\rm K})$ (calc.)	$P_{\rm c}({\rm kPa})$ (calc.)	<i>T</i> _c (K) (exp.)	$P_{\rm c}({\rm kPa})({\rm exp.})$	$\varepsilon_{\rm r} T_{\rm c}$	$\varepsilon_{\rm r} P_{\rm c}$
0.1694	327.94	7815.99	325.9	7908	0.6300	1.1600
0.3334	351.67	8169.68	351.7	8170	0.0085	0.0039
0.4984	377.27	7535.68	377.2	7536	0.0190	0.0042

 Table 4. n-Butane (1)-carbon dioxide (2) mixtures; comparison with experimental results [5]

Table 4 shows the results for critical points of the studied system, and from this, we conclude that the calculated data are in good agreement with the experimental data, when using PR EoS and the NRTL activity model.

CONCLUSIONS

This work presents a methodology (based on flash evaporation) for drawing phase envelopes, from which critical points of multicomponent mixtures can be estimated. The SRK EoS with VdW MR, SRK EoS with WS MR, and NRTL activity coefficient model were used. For the studied mixtures whose experimental critical data are available in the literature, our results were in good agreement with the experimental data. The methodology was capable of reproducing phase envelopes for multicomponent mixtures reported in [11], where a method which included the solution of a set ofordinary differential equations was used for drawing the phase envelopes. For the *n*-butane (1)-carbon dioxide (2) mixtures, both SRK and PR EoS were used, and the best results were obtained with PR EoS (WS MR and NRTL activity coefficient model). It is important to note that when the presented methodology was applied to the systems ethane (1)–n-butane (2), ethane (1)–propane (2), and *n*-butane (1)-carbon dioxide (2), the results were basically the same as when global optimization for obtaining critical points was applied to these systems, as reported in [5]. The presented methodology may be viewed as a shortcut procedure when an easy method for drawing the phase envelopes and estimating critical points of a multicomponent system is needed.

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