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New Multi-Solid Thermodynamic Model for the Prediction of Wax Formation

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Abstract**—**In the previous multi-solid models, φ approach is used for the calculation of fugacity in the liquid phase. For the first time, in the proposed multi-solid thermodynamic model, γ approach has been used for calculation of fugacity in the liquid mixture. Therefore, some activity coefficient models have been studied that the results show that the predictive Wilson model is more appropriate than others. The results demonstrate γ approach using the predictive Wilson model is in more agreement with experimental data than the previous multi-solid models. Also, by this method, generates a new approach for presenting stability analysis in phase equilibrium calculations. Meanwhile, the run time in γ approach is less than the previous methods used φ approach. The results of the new model present 0.75 AAD % (Average Absolute Deviation) from the experimental data which is less than the results error of the previous multi-solid models obviously.

Keywords**—**Multi-solid thermodynamic model, Predictive Wilson model, Wax formation.

I. Introduction

IN the multi-solid model, it is assumed the solid phase (wax) consist of several pure component. The studies show two main models apply the concept of multi-solid model, including Lira-Galeana *et al.* [1] and Nichita *et al.* [2] models. The other multi-solid models are similar to them approximately.

In 1996, Lira-Galeana *et al.* [1] presented an approach based on multi-solid model for the prediction of wax formation. In this model, a correlation was presented for estimating the melting point of pure components including normal paraffinic (C_6-C_{30}), naphthenic (C_6-C_{30} alkylcycloalkanes) and aromatic (C_6-C_{30} alkylbenzenes) hydrocarbons. Also, they suggested a correlation for the estimating of the enthalpy of fusion. They used the Pedersen *et al.* correlation [3] to estimate the specific heat capacity difference between solid and liquid phase. Also, the term of solid-solid phase transition was ignored in the calculation of fugacity ratio of the solid and liquid phase for a pure component. The PR EoS [4, 5] was used for the fugacity calculation in the fluid phases.

In 2001, Nichita *et al.* [2] suggested a multi-solid model. In this model, the melting point temperature of normal alkanes was estimated from the correlation proposed by Won [6]. Also, they applied solid-solid phase transition term for the calculation of the fugacity ratio of solid and liquid phase for a pure component. They suggested correlations for estimating of temperature and enthalpy of solid-solid phase transition. The PR EoS [4, 5] was applied for calculating fugacity in the fluid phases.

In this work, for the first time, a multi-solid model based on γ approach has been presented for the prediction of wax formation phenomena. Some activity coefficient models including the regular solution [3, 6, 7], UNIFAC [8-10], predictive UNIQUAC [11-13] and predictive Wilson [14] models and ideal solution approach have been employed and compared. For validating the proposed model some experimental data have been used which are for 56 equilibrium data points.

II. Experimental Data

In this work, four ternary systems including C_{14}-C_{15}-C_{16} (ternary 1), C_{16}-C_{17}-C_{18} (ternary 2), C_{18}-C_{19}-C_{20} (ternary 3) and C_{19}-C_{20}-C_{21} (ternary 4) have been used [15]. These systems contain 56 mixtures that the amount of WDT (Wax Disappearance Temperature) in Kelvin (K) at atmospheric pressure and compositions of mixtures have been reported in Tables I-IV.

TABLE I
Experimental WDT (K) Data for C_{14}-C_{15}-C_{16} Ternary System 1

Mixture	Composition (molar %)			Exp. WDT (K)
	C_{14}	C_{15}	C_{16}	
1	6	57	37	283.4
2	14	26	63	284.6
3	17	6	77	285.5
4	24	33	43	282.2
5	21	56	23	281.2
6	27	66	7	280.4
7	37	5	58	282.6
8	32	24	44	281.5
9	43	33	24	279.2
10	57	17	26	278.0
11	73	14	13	276.3

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TABLE II
EXPERIMENTAL WDT (K) DATA FOR C_{16}-C_{17}-C_{18} TERNARY SYSTEM 2

Mixture	Composition (molar %)			Exp. WDT (K)
	C_{16}	C_{17}	C_{18}	
1	10	10	80	297.8
2	10	75	15	294.3
3	10	80	10	294.6
4	11	39	50	295.8
5	20	20	60	295.8
6	20	60	20	293.7
7	33	33	34	293.3
8	40	10	50	293.6
9	40	40	20	291.9
10	60	20	20	290.8
11	80	10	10	289.5

TABLE III
EXPERIMENTAL WDT (K) DATA FOR C_{18}-C_{19}-C_{20} TERNARY SYSTEM 3

Mixture	Composition (molar %)			Exp. WDT (K)
	C_{18}	C_{19}	C_{20}	
1	2	2	96	309.0
2	5	5	90	308.5
3	5	90	5	304.7
4	10	10	80	307.5
5	10	40	50	306.3
6	10	55	35	305.9
7	14	73	13	304.4
8	15	15	70	307.3
9	20	20	60	306.3
10	20	60	20	304.9
11	26	26	48	305.5
12	33	33	34	304.3
13	40	10	50	304.6
14	43	43	14	302.8
15	48	15	37	303.8
16	60	20	20	302.3
17	79	11	10	301.4
18	90	5	5	300.6

TABLE IV
EXPERIMENTAL WDT (K) DATA FOR C_{19}-C_{20}-C_{21} TERNARY SYSTEM 4

Mixture	Composition (molar %)			Exp. WDT (K)
	C_{19}	C_{20}	C_{21}	
1	5	5	90	312.9
2	5	89	6	309.4
3	10	40	50	310.9
4	10	80	10	309.5
5	12	10	78	312.1
6	19	50	31	309.9
7	20	21	59	310.9
8	20	60	20	309.3
9	29	28	43	309.7
10	39	10	51	309.5
11	39	50	11	307.8
12	49	20	31	308.1
13	50	40	10	307.4
14	60	20	20	307.3
15	80	10	10	306.0
16	90	5	5	305.5

III. THE MULTI-SOLID MODEL BASED ON γ APPROACH

In the multi-solid model, the number of components precipitate should be obtained by stability analysis condition. The compounds cover this condition precipitates as a pure solid phase. The definition of stability analysis is in the following expression [16]:

$$f_i^Z(P,T,Z_i) - f_i^{S,Pure}(P,T) > 0 \qquad i = 1,...,C \tag{1}$$

where, $f_i^Z(P,T,Z_i)$, is the component fugacity in the mixture at pressure P, temperature T and with mixture composition Z_i. In (1), C, is the number of components. In all correlations in this paper, subscripts S, L and superscript i are referred to the solid and liquid phase and the number of components, respectively.

By the definition of fugacity in γ approach, component fugacity in the mixture, $f_i^Z(P,T,Z_i)$, can be calculated as follows:

$$f_i^Z(P,T,Z_i) = f_i^{L,Pure}(P,T)\gamma_i^L x_i^L \tag{2}$$

thus:

$$f_i^{L,Pure}(P,T)\gamma_i^L x_i^L - f_i^{S,Pure}(P,T) > 0 \tag{3}$$

and

$$\gamma_i^L x_i^L - \frac{f_i^{S,Pure}(P,T)}{f_i^{L,Pure}(P,T)} > 0 \tag{4}$$

In (4), γ_i^L can be calculated using the activity coefficient models. To obtain a suitable activity coefficient model, the regular solution [3, 6, 7], UNIFAC [8-10], predictive UNIQUAC [11-13] and predictive Wilson [14] models have been applied reported in the Appendix. The ideal solution approach ($\gamma_i^L = 1$) has been also considered. The fugacity ratio can be calculated as follows [2]:

$$\frac{f_{pure\ i}^S(P,T)}{f_{pure\ i}^L(P,T)} = \exp[\frac{\Delta H_i^f}{RT_i^f}\left(1-\frac{T_i^f}{T}\right) + \frac{\Delta H_i^{tr}}{RT_i^{tr}}\left(1-\frac{T_i^{tr}}{T}\right) + \frac{1}{RT}\int_T^{T_i^f}\Delta C_{p\,i}^{LS}dT - \frac{1}{R}\int_T^{T_i^f}\frac{\Delta C_{p\,i}^{LS}}{T}dT] \tag{5}$$

The fusion temperature (T_i^f) of normal alkanes is estimated from the following correlation proposed by Won [6].

$$T_i^f = 374.5 + 0.2617MW_i - \frac{20172}{MW_i} \tag{6}$$

For the estimation of solid state transition temperature (T_i^{tr}), Nichita *et al.* proposed the following correlation [2]:

$$T_i^{tr} = 366.39775 + 0.03609 MW_i - \frac{20879}{MW_i} \quad (7)$$

In (6) and (7), T is in K, and MW is the component molecular weight. For the calculation of fusion and the solid-solid transition enthalpy of normal alkanes, Nichita *et al.* suggested the following correlations for $MW_i > 282$ (gr/mol) [2]:

$$\Delta H_i^f = 0.1186 MW_i T_i^f \quad (8)$$

$$\Delta H_i^{tr} = 0.0577 MW_i T_i^{tr} \quad (9)$$

and for $MW_i < 282$ (gr/mol), Nichita *et al.* expressed the total enthalpy (fusion+ solid state transition) by the following correlation [2]:

$$\Delta H_i^t = 0.1777 MW_i T_i^f \quad (10)$$

In (8) to (10), ΔH is in cal/mol. For calculation of heat capacity difference between solid and liquid phase, ΔC_{pi}, the following correlation proposed by Pedersen *et al.* have been applied [3]:

$$\Delta C_{pi} = 0.3033 MW_i - 4.635\times10^{-4} MW_i T \quad (11)$$

that ΔC_{Pi} and T are in cal/mol.K and K, respectively.

For precipitating components the thermodynamic equilibrium can be written as follows [1, 2, 16]:

$$f_i^L(P,T,x_i^L) = f_i^{S,Pure}(P,T) \quad i = 1,...,C_s \quad (12)$$

where, C_s is the number of precipitating components. By using the stability analysis correlation, (4), and material balance for precipitating and non-precipitating components, the mole fraction and composition of solid phase can be obtained. The algorithm and material balance equations have been reported in the literature [16].

IV. Results and Discussion

The results of calculations have been reported in Table V. This table shows the new multi-solid model using predictive Wilson model gives better results in comparison with other activity coefficient models and ideal solution approach and the previous multi-solid models. Figs. 1-4 show the results of calculations with new multi-solid model by using the predictive Wilson model.

TABLE V
THE RESULTS OF CALCULATIONS

Ternary systems	1	2	3	4	Total
No. of data points	11	11	18	16	56
Models			AAD %[a]		
Lira-Galeana *et al.* (1996) [ ]	16.26	13.11	10.85	10.66	12.30
Nichita *et al.* (2001) [ ]	1.17	1.00	1.07	1.63	1.24
New Model $\gamma = 1$	1.17	1.00	1.08	1.63	1.24
New MS model Regular solution	1.17	1.00	1.08	1.63	1.24
New MS model UNIFAC	1.18	1.00	1.08	1.63	1.24
New MS Model P. UNIQUAC	0.91	0.72	0.87	1.37	0.99
New MS model P. Wilson	0.51	0.49	0.66	1.19	0.75

[a] $\%AAD = \frac{100}{n}\sum_i^n \frac{|Cal_i - Exp_i|}{Exp_i}$

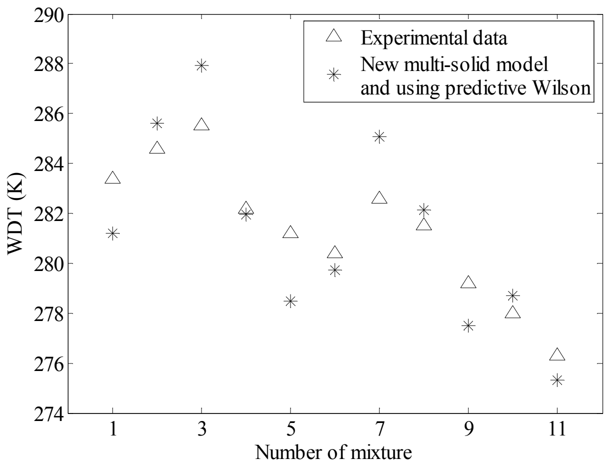

Fig. 1 The results of calculation by new multi-solid model and predictive Wilson model in ternary 1 (C_{14}-C_{15}-C_{16})

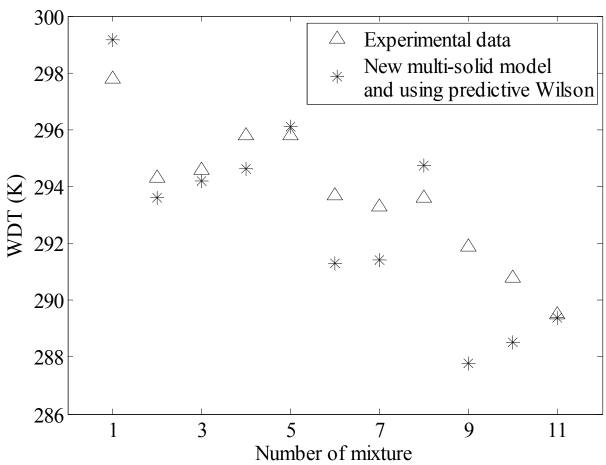

Fig. 2 The results of calculation by new multi-solid model and predictive Wilson model in ternary 2 (C_{16}-C_{17}-C_{18})

Also, Table VI indicates the Nichita *et al.* and new multi-solid models give better results than the Lira-Galeana model strongly. It proves that the consideration of solid-solid transition term is required for the calculation of solid-liquid phase equilibrium based on the concept of multi-solid model for prediction of wax formation phenomena.

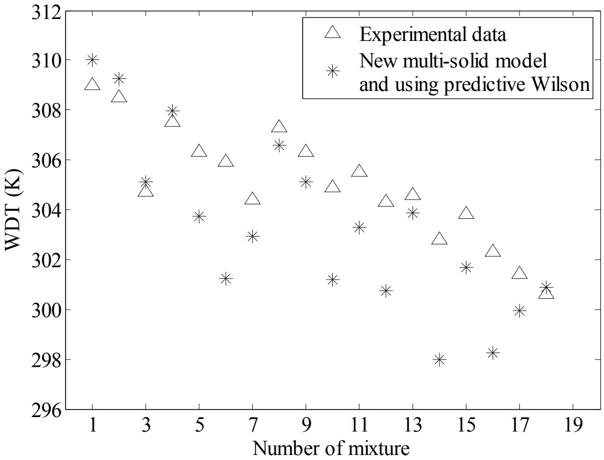

Fig. 3 The results of calculation by new multi-solid model and predictive Wilson model in ternary 3 (C_{18}-C_{19}-C_{20})

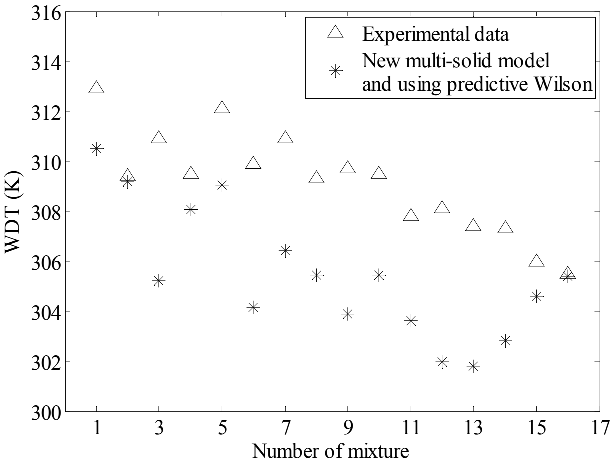

Fig. 4 The results of calculation by new multi-solid model and predictive Wilson model in ternary 4 (C_{19}-C_{20}-C_{21})

V. Conclusion

In the previous multi-solid models for the prediction of wax precipitation phenomena, the equation of state has been used for calculation of fugacity in the liquid phase. For the first time, in this work, activity coefficient models have been applied for the stability analysis and fugacity calculation. The results show that this approach is better than that one uses the equation of state. Also, the run time of new method is less than the previous models.

Appendix

A. Activity Coefficient Models

1) Regular Solution Theory [6, 7]

$$\ln \gamma_i = \frac{V_i(\bar{\delta} - \delta_i)^2}{RT} \quad (1)$$

Where, V, δ and $\bar{\delta}$ are the molar volume, solubility parameter and average solubility parameter, respectively.

$$\bar{\delta} = \sum_i \varphi_i \delta_i \quad (2)$$

As, φ_i^L and φ_i^S are the volume fraction of liquid and solid phases, respectively.

$$\varphi_i^L = \frac{x_i^L V_i^L}{\sum_i x_i^L V_i^L} \quad (3)$$

$$\varphi_i^S = \frac{x_i^S V_i^S}{\sum_i x_i^S V_i^S} \quad (4)$$

In this approach, the liquid and solid molar volumes are assumed to be equal. Therefore,

$$V_i^L = V_i^S = V_i = \frac{MW_i}{d_{i,25}^L} \quad (5)$$

For estimation of the liquid density of each component at 25°c ($d_{i,25}^L$), the following correlation depending on molecular weight is used [3]:

$$d_{i,25}^L = 0.8155 + 0.6272 \times 10^{-4} MW_i - \frac{13.06}{MW_i} \quad (6)$$

Solubility parameters in the liquid and solid phases (δ_i^L and δ_i^S) related to carbon number (C_{ni}) are calculated by (7) and (8) suggested by Pedersen *et al.* [3]:

$$\delta_i^L = 7.41 + 0.5194(\ln C_{ni} - \ln 7) \quad (7)$$

$$\delta_i^S = 8.5 + 5.763(\ln C_{ni} - \ln 7) \quad (8)$$

2) UNIFAC [8]

For mixtures containing alkanes only, the following correlation is used [8]:

$$\ln \gamma_i = \ln\left(\frac{\Phi_i}{x_i}\right) + 1 - \frac{\Phi_i}{x_i} - \frac{Z}{2} q_i \left(\ln\left(\frac{\Phi_i}{\theta_i}\right) + 1 - \frac{\Phi_i}{\theta_i}\right) \quad (9)$$

Z is the coordination number. For orthorhombic molecular structure is set to 6 and θ_i, the area fraction, and φ_i, the segment fraction, are obtained from the following correlations:

$$\theta_i = \frac{x_i q_i}{\sum_j x_j q_j} \quad (10)$$

$$\Phi_i = \frac{x_i r_i}{\sum_j x_j r_j} \tag{11}$$

The values of molecular size parameter, r_i, and molecular external surface parameter, q_i, have been obtained from the Esmaeilzadeh *et al.* correlations [9-10]:

$$r_i = 0.6744 C_{ni} + 0.4534 \tag{12}$$

$$q_i = 0.54 C_{ni} + 0.616 \tag{13}$$

3) Predictive UNIQUAC [11, 12]

$$\ln\gamma_i^S = \ln\left(\frac{\Phi_i}{x_i^S}\right) + 1 - \frac{\Phi_i}{x_i^S} - \frac{Z}{2} q_i \left(\ln\left(\frac{\Phi_i}{\theta_i}\right) + 1 - \frac{\Phi_i}{\theta_i}\right) + q_i - q_i \ln\left(\sum_{j=1}^{n} \theta_j \tau_{ji}\right) - q_i \sum_{j=1}^{n} \frac{\theta_j \tau_{ij}}{\sum_{k=1}^{m} \theta_k \tau_{kj}} \tag{14}$$

$$\tau_{ji} = \exp\left(-\frac{\lambda_{ji} - \lambda_{ii}}{q_i RT}\right) \tag{15}$$

In this equation, the λ_{ji} is the interaction energy. Similar to UNFAC, θ_i and Φ_i are calculated by (10) and (11). The correlations for the r and q values with the n-alkane chain length are [13]:

$$r_i = 0.0148 C_{ni} + 0.00996 \tag{16}$$

$$q_i = 0.0185 C_{ni} + 0.0211 \tag{17}$$

The interaction energy, λ_{ii} is estimated from the heat of sublimation of pure orthorhombic crystal,

$$\lambda_{ii} = -\frac{2}{Z}\left(\Delta H_{sub\,i} - RT\right) \tag{18}$$

with Z being the coordination number. For the orthorhombic crystals, the value of 6 is considered for Z [11, 14]. The interaction energy between two non-identical molecules is given by:

$$\lambda_{ij} = \lambda_{ji} = \lambda_{jj} \tag{19}$$

where j is the n-alkane with the shorter chain of the pair ij.

Heat of sublimation can be calculated by:

$$\Delta H_i^{sub} = \Delta H_i^{vap} + \Delta H_i^{f} + \Delta H_i^{tr} \tag{20}$$

where vaporization enthalpy is assessed using the PERT2 correlation by Morgan and Kobayashi [17]. The critical properties needed in Morgan and Kobayashi correlations can be calculated by Twu correlations [18]. ΔH_{tr}, is calculated by the following correlation:

$$\Delta H_{tr} = \Delta H_{tot} - \Delta H_f \tag{21}$$

$$\Delta H_{tot} = 3.7791 C_n - 12.654 \tag{22}$$

4) Predictive Wilson [14]

$$\ln\gamma_i = 1 - \ln\sum_i x_j \Lambda_{ij} - \sum_k \frac{x_k \Lambda_{ki}}{\sum_j x_j \Lambda_{kj}} \tag{23}$$

$$\Lambda_{ij} = \exp\left[-\frac{\lambda_{ij} - \lambda_{ii}}{RT}\right] \tag{24}$$

Similar to the predictive UNIQUAC approach, λ_{ij} is calculated and the value of 6 is considered for Z.

NOMENCLATURE

Symbols

C	number of component
C_n	carbon number
C_p	specific heat capacity
d	density
f	fugacity
H	enthalpy
i	counter of component
MW	molecular weight
P	pressure
q	molecular external surface parameter
r	molecular size parameter
R	gas universal constant
T	temperature
V	volume
x	mole fraction
Z	coordination number
Z_f	feed composition

Greek letters

Δ	variation
γ	activity coefficient
δ	solubility parameter
$\bar{\delta}$	average solubility parameter
φ	volume fraction
Φ	segment fraction
θ	area fraction
Λ	interaction parameter
λ	interaction energy
τ	characteristic energy parameter

Superscripts

L	liquid
S	solid

Subscripts

C	critical
F	feed
f	fusion
i	component number
j	component number
n	component number
sub	sublimation
tot	total
tr	transition
vap	vaporization

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