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Isobaric vapour–liquid equilibrium measurements and extractive distillation process for the azeotrope of (*N*,*N*-dimethylisopropylamine + acetone)

Yixin Ma [a], Jun Gao [a], Min Li [b], Zhaoyou Zhu [b], Yinglong Wang [b],*

[a] *College of Chemical and Environmental Engineering, Shandong University of Science and Technology, Qingdao 266590, China*
[b] *College of Chemical Engineering, Qingdao University of Science and Technology, Qingdao 266042, China*

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ABSTRACT

In this work, *N*,*N*-dimethylacetamide (DMAC) and furfural were selected as solvents to separate the mixture of (*N*,*N*-dimethylisopropylamine (DMIP) + acetone) by extractive distillation. The isobaric vapour–liquid equilibrium (VLE) data for the binary systems of (DMIP + acetone), (DMIP + DMAC), (DMIP + furfural) and (acetone + furfural) were measured at 101.3 kPa by a modified Rose vapour recirculating type equilibrium still. Two thermodynamic consistency tests of Herington and Van Ness were employed to check the experimental data for all the binary systems, respectively. The measured VLE data were correlated by NRTL, UNIQUAC and Wilson models. The calculated results show that the UNIQUAC model is best-fitted to correlate the measured VLE data for the four binary systems. Furthermore, extractive distillation process for separating the azeotrope of DMIP and acetone was studied in this paper.

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1. Introduction

N,*N*-Dimethylisopropylamine (DMIP) [1,2] is a kind of tertiary amine, in which nitrogen atoms play the role of ionizing chromophore, and the aliphatic substituents provide interesting structural and chemical dynamics. DMIP is often used as chemical reagents, fine chemicals, pharmaceutical intermediates and material intermediates. DMIP can be synthesized by dimethylamine and acetone as the main raw material in industry, after the synthesis, an DMIP aqueous solution containing unreacted acetone is generated. To recover acetone and obtain high purity DMIP, it is necessary to separate DMIP from its aqueous solution. The boiling points of acetone and DMIP are 329.15 K and 338.65 K, respectively. With ordinary distillation, it is difficult to separate the mixture of DMIP and acetone efficiently. Special distillation methods, such as pressure-swing distillation [3–5], azeotropic distillation [6–8], and extractive distillation [9–11], are needed to separate this ternary azeotropic mixture.

Extractive distillation is one of the most economical ways to separate close-boiling and azeotropic mixtures in the chemical and petroleum industries. For extractive distillation, the key to select solvents is to compare the relative volatilization degree of the solvent after the addition of different solvents [12]. *N*,*N*-Dimethylacetamide (DMAC) and furfural, which were all widely used to separate close-boiling or azeotrope mixtures, were selected as the solvent to separate the mixture of DMIP and acetone based on the criteria of solvent selection for extractive distillation. To the simulation and optimization of separation process, the binary interaction parameters regressed by experimental data are necessary [13–16]. In the previous works, the isothermal VLE behaviour of DMIP and water mixtures has been investigated by Ahmed et al. [17] and Chun et al. [18] at different temperatures. Carli et al. [19] studied the vapour–liquid data for water + DMAC. Ni et al. [20] reported the isobaric data for the binary system of (water + furfural) at 101 kPa. Al-Sahhaf and Jabbar [21] reported the isobaric VLE data for (acetone + water) at 0.02–0.103 MPa. Deizenrot et al. [22] measured the VLE data for (acetone + DMAC) at 101 kPa. Myles et al. [23] reported the isobaric VLE data for (acetone + furfural) at 101 kPa. For the binary systems of DMIP + acetone, DMIP + DMAC and DMIP + furfural, no experimental data were retrieved from the Dortmund Data Bank (DDB) [24] and the NIST database [25].

In the study of the extractive distillation process with furfural as solvent, the phase behaviour for the system of acetone + furfural described by different Aspen Plus models are different. Although the experimental data are reported in the literature [23], the phase behaviour of the full concentration range cannot be described. So the isobaric VLE data for (DMIP + acetone), (DMIP + DMAC),

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* Corresponding author.
E-mail address: yinglongw@126.com (Y. Wang).

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(DMIP + furfural) and (acetone + furfural) binary systems were measured with a modified Rose still [26,27] at 101.3 kPa. In addition, two thermodynamic consistency tests of Herington [28] and van Ness [29] were adopted to check the consistency of the measurement of VLE data. Furthermore, the measured VLE data were correlated by three models of the NRTL [30], UNIQUAC [31], and Wilson [32], and the model interaction parameters of the binary systems were obtained. Meanwhile, two extractive distillation processes were presented to separate the mixture of DMIP + acetone using DMAC and furfural as solvents.

2. Experimental

2.1. Chemicals

Analytical reagents, acetone, DMIP, DMAC and furfural, were used without any further purification in this work. The water content in acetone, DMIP, DMAC and furfural was 0.3%, 0.09%, 0.05%, 0.01%, respectively. The purity of the chemicals was checked and confirmed using gas chromatography (GC-14C, Shimadzu). A precision mercury thermometer with an uncertainty of ±0.1 K was used to measure the boiling temperature (T_b) at 101.3 kPa for the chemicals. The measured boiling temperatures were compared with the values obtained from the NIST database [25]. The maximum boiling point deviations for acetone, DMIP, DMAC, and furfural are 0.28 K, 0.48 K, 0.01 K and 0.37 K, respectively. Experimental data show good agreement with the NIST database [25]. The detailed information of the chemicals are listed in Table 1.

2.2. Apparatus and procedure

In this work, the VLE measurements were carried out by a modified Rose type recirculating equilibrium still. The detailed description of the experimental device was reported in our previous work [33,34]. A precision mercury thermometer (Tianjin Glass Instrument Factory) was used to measure the equilibrium temperature with the accuracy of ±0.1 K. A U-shaped differential manometer (Nanjing Hengyuan Automatic Gauge Co., Ltd.) was used to maintain the pressure of the equilibrium still 101.3 kPa with the accuracy of ±0.1 kPa. In recirculating still, the liquid phase and the condensed vapour phase were recirculated continuously to make the liquid phase fully contact with the vapour phase and establish the balance as soon as possible. The equilibrium state was reached after the system temperature was kept at a constant value for at least 50 min. After that, the samples of the vapour phase and liquid phase were taken out at the same time by the syringe, respectively, and analysed by GC.

2.3. Analysis

The compositions of the liquid phase and vapour phase samples were analysed by GC (GC-14C, Shimadzu) with a capillary column (DB-5, 0.25 μm × 0.25 mm × 30 m) and a hydrogen flame ionization detector (FID). The detector was connected to N2000 gas chromatography workstation. The compositions of all samples were obtained by the N2000 workstation software (Zhejiang University). Nitrogen with the purity of 0.9999 was used as the carrier gas with a constant flow rate of 30 $\text{mL}\cdot\text{min}^{-1}$. The column inlet pressure was kept at 0.18 MPa. For the system of DMIP + acetone, the oven, injector and detector temperatures were kept at 313.15 K, 333.15 K, and 343.15 K, respectively. For the three systems of (DMIP + DMAC), (DMIP + furfural) and (acetone + furfural), the oven, injector and detector temperatures were kept at 393.15 K, 413.15 K, and 433.15 K, respectively. A mixture of five known compositions was prepared using FA-1204B electronic balance (Shanghai Tianmei balance instrument co., Ltd) with the uncertainty of 0.0001 g to calibrate GC. The composition of each sample was obtained on the basis of more than three analysis results and the average of three results was adopted.

3. Results and discussion

3.1. Experimental results

In this work, the VLE data for the four binary systems of (DMIP + acetone), (DMIP + DMAC), (DMIP + furfural) and (acetone + furfural) were measured at 101.3 kPa. The VLE data are expressed in mole fraction and listed in Tables 2–5. The *T-x-y* diagrams for the binary systems are presented in Figs. 1–4.

3.2. VLE calculation

The VLE equilibrium experiments were carried out at 101.3 kPa, the vapour phase could be considered as ideal gas. The relationship of vapour–liquid equilibrium can be simplified as follows [35]:

$$y_i p = x_i \gamma_i p_i^s \tag{1}$$

where y_i and x_i are the mole fractions of component *i* in the vapour and liquid phase, respectively. γ_i and P are the activity coefficient of component *i* and the system pressure, respectively. p_i^S, the saturated vapour pressure of the pure component *i*, can be calculated by the following extended Antoine equation:

$$\ln\left(p_i^s/kPa\right) = \mathrm{C}_{1i} + \frac{\mathrm{C}_{2i}}{T + \mathrm{C}_{3i}} + \mathrm{C}_{4i}T + \mathrm{C}_{5i}\ln(T) + \mathrm{C}_{6i}T^{\mathrm{C}_{7i}} \quad for \quad \mathrm{C}_{8i} \leqslant T \leqslant \mathrm{C}_{9i} \tag{2}$$

For each pure component *i*, the Antoine parameters C_{1i} to C_{9i} are listed in Table 6, which were obtained directly from the Aspen property database. The values of γ_i calculated by the Eqs. (2) and (3) for the binary systems are listed in Tables 2–5.

Table 1
Experimental Reagents Information.[a]

Component	CAS	Suppliers	mass fraction	purification method	analysis method	T_b/K at 101.3 kPa	
						This work	literature[b] (NIST)
acetone	67-64-1	Laiyang Economic and Technological Development Zone Fine Chemical Plant	≥0.995	none	GC[a]	329.56	329.24
DMIP	996-35-0	Tokyo Chemical Industry Co., Ltd	>0.990	none	GC[a]	339.13	341.60
DMAC	127-19-5	Sinopharm Chemical Reagent Co., Ltd	≥0.990	none	GC[a]	439.05	439.06
furfural	98-01-1	Sinopharm Chemical Reagent Co., Ltd	≥0.990	none	GC[a]	435.15	434.78

[a] Gas chromatography.
[b] The NIST database.

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