

Effect of pressure and the capability of 2-methoxyethanol as a solvent in the behaviour of a diisopropyl ether–isopropyl alcohol azeotropic mixture

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Abstract

Consistent vapour–liquid equilibrium data for the binary and ternary systems of diisopropyl ether + isopropyl alcohol at 30 and 101.3 kPa and diisopropyl ether + 2-methoxyethanol, isopropyl alcohol + 2-methoxyethanol and diisopropyl ether + isopropyl alcohol + 2-methoxyethanol at 101.3 kPa are reported. The activity coefficients of the solutions were correlated with its composition using the Wilson, NRTL and UNIQUAC models. It is shown that the models allow a very good prediction of the phase equilibrium of the ternary system using the pertinent parameters of the binary systems. Moreover, the effect of pressure and the entrainer capability of 2-methoxyethanol were studied.

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1. Introduction

The most common method of manufacturing isopropyl alcohol is by the dehydration of propylene using sulphuric acid as the catalyst [1]. However, before the isopropyl alcohol can be removed from the reaction mixture, some of it reacts with sulphuric acid to form diisopropyl ether. Isopropyl alcohol and diisopropyl ether form a binary homogeneous azeotrope. Normally a mixture of several solvents are separated and recovered by fractionation in a rectification column and the ease of separation depends upon the difference in boiling points of the compounds to be separated. However, the separation of a homogeneous azeotrope mixture, that is a common task in the chemical industry, cannot be carried out by conventional distillation. For the solution of the separation problem, there are several techniques, including extractive distillation [2] and pressure-swing distillation [3]. Extractive dis-

tillation can be used to separate the components of an azeotropic mixture by adding an agent (entrainer) that modifies the relative volatility of the mixture although a previous selection of a possible entrainer is required. In this work, the selection has been done by Scheibel's criterion [2], and by means of the calculation of the separation factor. We chose 2-methoxyethanol (widely known by its trade name, methyl cellosolve) which is a common solvent in laboratory practice and in the chemical industry. Also, pressure-swing distillation (PSD) is often mentioned as an alternative process to the broad applied processes azeotropic distillation and extractive distillation. In PSD the dependency of the azeotropic concentration on the system pressure is used to overcome the azeotrope. The general advantages of the PSD process are the saving of energy and the abdication of entrainers compared to well-known alternatives.

The thermodynamic analysis, prediction and computer simulation of phase equilibrium help to understand and evaluate the different separation processes. In any case, a reliable knowledge of the phase equilibrium behaviour is necessary for synthesis, design and optimisation of any distillation system.

In this work, to improve the knowledge of the phase behaviour we report VLE data for the binary system diisopropyl ether (1)+isopropyl alcohol (2) at two pressures (30 and 101.3 kPa). Moreover, we measured isobaric VLE data for

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the ternary system diisopropyl ether (1)+isopropyl alcohol (2)+2-methoxyethanol (3) and two constituent binary systems diisopropyl ether (1)+2-methoxyethanol (3) and isopropyl alcohol (2)+2-methoxyethanol (3) at 101.3 kPa. In a recent literature review only isobaric VLE for the diisopropyl ether (1)+isopropyl alcohol (2) system at atmospheric pressure has been found [4].

VLE data of binary and ternary systems were found to be thermodynamically consistent. Data reduction was carried out using the Wilson, NRTL and UNIQUAC equations to relate activity coefficients with compositions.

2. Experimental

2.1. Chemicals

Diisopropyl ether (>99.0 mass%, analytical grade) was purchased from Fluka, isopropyl alcohol (>99.5 mass%, HPLC grade) and 2-methoxyethanol (>99.8 mass%, analytical grade) were purchased from Aldrich Ltd. The reagents were used without further purification after chromatography failed to show any significant impurities. The water content, determined using a Karl Fischer volumetric automatic titrator (Metrohm, 701 KF Titrino), was small in all chemicals (<0.05 mass%). Before measurements, the liquids were degassed and subsequently dried over molecular sieves (Union Carbide, type 4 Å, 1/16 in. pellets). The refractive indexes of the pure components were measured at 298.15 K using an Abbe refractometer Atago 3T, and the densities were measured at 298.15 K using an Anton Paar DMA 58 densimeter. Temperature was controlled to ± 0.01 K with a thermostated bath. The accuracy in refractive index and density measurements are ± 0.0002 and $\pm 0.01 \text{ kg m}^{-3}$, respectively. The experimental values of these properties and the boiling points are given in Table 1 together with those given in the literature. Appropriate precautions were taken when handling the reagents in order to avoid hydration.

2.2. Apparatus and procedure

The equilibrium vessel used in the measurements (Labodest VLE 602/D) was an all-glass dynamic-recirculating still equipped with a Cottrell circulation pump, manufactured by Fischer Labor und Verfahrenstechnik (Germany). The Cottrell pump ensures that both liquid and vapour phases are in intimate contact during boiling and also in contact with the temperature sen-

sing element. The equilibrium temperature was measured with a digital Hart Scientific thermometer model 1502A and a Pt 100 probe Hart Scientific model 5622 calibrated at the ENAC-accredited Spanish *Instituto Nacional de Técnica Aeroespacial*. The accuracy is estimated to be ± 0.01 K. The temperature probe was checked against the ice and steam points of distilled water. A Fisher M101 pressure control system was used to measure and control the pressure and the heating power. The pressure accuracy is ± 0.1 kPa. The manometer was calibrated using the vapour pressure of ultrapure water.

In each experiment, the pressure was fixed and the heating and stirring system of the liquid mixture was turned on. The still was operated at constant pressure until equilibrium was reached. Equilibrium conditions were assumed when constant temperature and pressure were obtained for 30 min or longer. Then, samples of liquid and condensate were taken for analysis. The sampling was carried out with special syringes that allowed withdrawal of small volume samples.

2.3. Analysis

The composition of the sampled liquid and condensed vapour phase were determined using a CE Instruments GC 8000 Top gas chromatograph (GC), after calibration with gravimetrically prepared standard solutions. A flame ionization detector was used together with a 30 m, 0.454 mm i.d., capillary column (DB-MTBE, J & Scientific). The GC response peaks were treated with Chrom-Card for Windows. Column, injector and detector temperatures were 363, 473, and 498 K, respectively, for all systems. Very good peak separation was achieved under these conditions and calibration analyses were carried out to convert the peak area ratio to the mass composition of the sample. In general, two analyses were made of each liquid and vapour composition. The standard deviation in the mole fraction was usually less than 0.001.

3. Results and discussion

3.1. Pure component vapour pressures

The pure component vapour pressure for 2-methoxyethanol was determined experimentally as a function of the temperature using the same equipment as that for obtaining the VLE data. The pertinent results appear in Table 2. The measured vapour

Table 1
Density d , refractive index n_D , and normal boiling point T_b of pure components

Component	d (kg m^{-3}) (298.15 K)		n_D (298.15 K)		T_b (K)	
	Exptl.	Lit.	Exptl.	Lit.	Exptl.	Lit.
Diisopropyl ether (1)	0.7183	0.7182 ^a	1.3652	1.3655 ^a	341.49	341.45 ^a
Isopropyl alcohol (2)	0.7827	0.7813 ^a	1.3754	1.3752 ^a	355.35	355.41 ^a
2-Methoxyethanol (3)	0.9601	0.9602 ^a	1.4002	1.4023 ^b	397.44	397.55 ^b

^a TRC tables [5].

^b Thornton and Garner [6].

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