

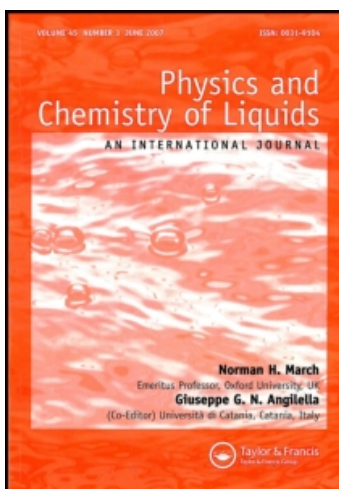
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Vapor–liquid equilibria for binary mixtures containing ethyl tert butyl ether (ETBE) + (*p*-xylene, *m*-xylene and ethylbenzene) at 101.3 kPa

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Isobaric vapor–liquid equilibria data at 101.3 kPa were reported for the binary mixtures ethyl tert butyl ether (ETBE) + (*p*-xylene, *m*-xylene and ethylbenzene). VLE experimental data were tested for thermodynamic consistency by means of a modified Dechema test and was demonstrated to be consistent. The activity coefficients were correlated with the Margules, van Laar, UNIQUAC, NRTL, and Wilson equations. The Analytical Solution Of Groups (ASOG) model also was applied for prediction.

Keywords: ETBE; Aromatics; Oxygenates; Phase equilibria; Interaction parameters

1. Introduction

Oxygenated compounds are added to gasoline to improve the combustion efficiency and to reduce the CO and hydrocarbons emissions. Typical oxygenated compounds are alcohols (Ethanol, Methanol and tert butyl alcohol) and ethers (Methyl tert Butyl Ether (MTBE), Ethyl tert Butyl Ether (ETBE), tert Amyl Ether (TAME), and DIPE (Diisopropyl Ether), being MTBE the most worldwide used followed by ETBE. However, owing to their capacity of dispersion in waters, they are nowadays becoming relevant persistent contaminants [1]. On the other hand, aromatic are majority compounds in gasoline and then the knowledge of thermodynamic properties and phase equilibria of this kind of mixtures enclosing these compounds is of practical interest to develop and optimize cleaning water processes. Tertiary ethers such as MTBE, ETBE and TAME present advantages in relation to ethanol: low Reid vapor pressure, low latent heat of vaporization and relative low water solubility. With regard to these ethers,

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it should be noted that ETBE can be considered as an almost renewable compound, since the raw material used for its production derives from biomass, while MTBE is produced from methanol, derived from synthesized gas. Thus, the production of ETBE reduces the dependence on methanol (from natural gas) and also contributes to reduce the greenhouse effect. ETBE is more important in terms of octane number as well as more attractive in terms of volatility and water solubility, since it presents a lower mixture (gasoline/oxygenate), Reid vapor pressure and lower water solubility than MTBE and ethanol. In the last few years, since the use of MTBE became restricted due to its toxicity and contamination of groundwater, the manufacturing units producing this chemical have been converted to produce ETBE and isooctane. ETBE is a promising compound in ethanol-producing countries where this alcohol is commercially available at a competitive price. With these facts in mind, this work is part of an extensive study related to theoretical and experimental analysis (phase equilibria and thermodynamic properties) of binary mixtures containing oxygenated chemicals and gasoline compounds in order to develop models and procedures for removing contaminants owed to gasoline spills from ground water.

2. Experimental section

2.1. Materials

Pure aromatic compounds were Merck's chromatographic grade. ETBE was supplied by REPSOL YPF with purity superior than 98%. Water was Millipore quality with organic total mass < 5 ppb and resistivity 18.2 MΩ cm. The purity of the materials so obtained was checked by gas liquid chromatography and was found to be better than 99.6 mol%. All products were degassed using ultrasound and dried on molecular sieves (pore diameter of $3 \cdot 10^{-10}$ m from Fluka) before use. Densities, speed of sound, and normal boiling points of the pure substances were checked and listed in table 1 and compared with literature values, as well as other relevant information.

2.2. Apparatus and procedure

VLE measurements were carried out under an atmosphere of dry nitrogen in a modified all-glass Othmer-type ebulliometer with secondary recirculations of both phases [8]. Thermal insulation was ensured with the whole apparatus having been insulated except in the part corresponding to vapor condenser. Boiling temperatures of mixtures were

Table 1. Densities ρ , speed of sound u , and normal boiling temperatures T_b of the pure components.

Component	MW ^a (kg kg ⁻¹ mol)	ρ (298.15 K) (g cm ⁻³)		u (298.15 K) (m s ⁻¹)		T_b (K)	
		Exptl.	Lit.	Exptl.	Lit.	Exptl.	Lit.
ETBE	102.18	0.73481	0.73528 ^b	1031.01	–	345.08	345.85 ^c
<i>p</i> -Xylene	106.167	0.85667	0.85662 ^c	1305.39	1308 ^c	411.43	411.23 ^f
<i>m</i> -Xylene	106.167	0.859471	0.85999 ^d	1318.80	1320 ^d	412.39	412.34 ^a
Ethylbenzene	106.167	0.86312	0.86252 ^d	1312.66	1318 ^d	409.18	409.36 ^a

^aPoling *et al.* [2]; ^bDomanska [3]; ^cGeorge and Sastry [4]; ^dGeorge and Sastry [5]; ^eArce *et al.* [6]; ^fSánchez-Russinyol *et al.* [7].

measured with a digital platinum $100\ \Omega$ resistance thermometer with an accuracy of $\pm 0.01\ \text{K}$ over the entire range of working temperatures. Pressure was kept constant at $(101.3 \pm 9.8 \cdot 10^{-2})\ \text{kPa}$ by a digital manometer regulator (Divatronic DT1 model), manufactured by Leybold with an accuracy of $\pm 0.1\ \text{kPa}$, which introduced nitrogen to the apparatus in order to maintain the pressure difference with respect to the pressure at the laboratory. Each experiment was continued for at least 1 h after the stabilization of the boiling temperature. Both vapor and liquid-phase compositions for the two systems were determined by densimetry and speed of sound. Densities and speed of sound were measured at by using an Anton Paar DSA 5000 vibrating tube densimeter and sound analyzer with an accuracy of $\pm 0.000001\ \text{g cm}^{-3}$ for density and $\pm 0.01\ \text{m s}^{-1}$ for speed of sound. The equipment was calibrated at atmospheric pressure with twice distilled water and dry air. The temperature of the densimeter was maintained with a precision of $\pm 0.001\ \text{K}$ by means of a semiconductor Peltier element and measured by a calibrated platinum resistance thermometer. Prior to measurements, density-calibration and speed-sound curves for these systems were obtained to calculate the compositions of the vapor and liquid phases. The binary mixtures were prepared by directly weighing the constituent components with an electronic balance that has an accuracy of $\pm 0.0001\ \text{g}$. Precautions were taken in order to minimize evaporation losses during storage and preparation of the solutions. The estimated uncertainty in the determination of both liquid and vapor phase mole fractions is ± 0.001 .

3. Results and discussion

Vapor–liquid equilibrium data (T , x_1 , y_1) for ETBE (1) + *p*-xylene (2), ETBE (1) + *m*-xylene (2), and ETBE (1) + ethylbenzene (2) binary systems at 101.3 kPa are presented in table 2. The T - x_1 - y_1 phase diagrams are shown in figures 1, 2, and 3.

The activity coefficients γ_i of the components were calculated from,

$$\gamma_i = \frac{y_i \Phi_i P}{x_i P_i^0} \quad (1)$$

where x_i and y_i are the liquid and vapor mole fractions in equilibrium, Φ_i is a vapor phase correction factor, P is the total pressure, and P_i^0 is the vapor pressure of pure component i .

These vapor pressures were calculated from the Antoine equation,

$$\log(P_i^0) = A_i - \frac{B_i}{(T) + C_i} \quad (2)$$

The vapor pressure P_i^0 is in kPa and the temperature in K. The constants A_i , B_i and C_i are reported in table 3 and their values were obtained from literature.

The vapor phase correction factor is given by,

$$\Phi_i = \frac{\phi_i}{\phi_i^{\text{sat}}} \exp\left[-\frac{V_i(P - P_i^0)}{RT}\right] \quad (3)$$

where ϕ_i is the fugacity coefficient of component i in the mixture, ϕ_i^{sat} is the fugacity coefficient at saturation, and V_i is the molar volume of component i in the liquid phase.

Table 2. Vapor-Liquid equilibrium data for ETBE (1) + *p*-xylene (2), ETBE (1) + *m*-xylene (2) and ETBE (1) + ethylbenzene (2) systems at 101.3 kPa: liquid-phase mole fraction, x_1 ; vapor-phase mole fraction, y_1 ; boiling temperature, T ; activity coefficients, γ_1 and γ_2 .

x_1	y_1	T (K)	γ_1	γ_2	x_1	y_1	T (K)	γ_1	γ_2	x_1	y_1	T (K)	γ_1	γ_2
ETBE (1) + <i>p</i> -Xylene (2)														
0.008	0.050	409.59	1.368	1.009	0.013	0.070	409.65	1.051	1.010	0.025	0.121	405.15	1.184	1.005
0.033	0.164	404.88	1.208	1.030	0.027	0.140	407.42	1.084	1.007	0.045	0.205	402.14	1.182	1.005
0.057	0.254	400.65	1.194	1.055	0.071	0.312	400.70	1.054	1.020	0.062	0.275	399.48	1.214	1.003
0.080	0.367	397.04	1.320	1.013	0.089	0.367	398.22	1.055	1.027	0.089	0.342	396.51	1.117	1.017
0.109	0.453	392.95	1.304	1.013	0.113	0.436	395.04	1.055	1.033	0.109	0.410	393.38	1.166	1.018
0.140	0.525	388.95	1.275	1.023	0.141	0.510	391.26	1.079	1.037	0.145	0.501	389.29	1.169	1.009
0.166	0.584	385.59	1.292	1.021	0.193	0.609	385.65	1.076	1.048	0.167	0.544	386.80	1.163	1.019
0.182	0.615	383.66	1.293	1.021	0.219	0.653	382.84	1.086	1.052	0.206	0.610	382.99	1.151	1.025
0.204	0.642	381.46	1.266	1.044	0.252	0.696	380.06	1.082	1.050	0.238	0.662	379.94	1.159	1.016
0.224	0.672	379.50	1.264	1.042	0.296	0.747	376.13	1.090	1.057	0.287	0.715	376.32	1.129	1.026
0.244	0.705	377.56	1.273	1.022	0.333	0.775	373.45	1.076	1.087	0.312	0.739	374.38	1.124	1.035
0.260	0.724	375.84	1.279	1.029	0.374	0.809	370.59	1.079	1.085	0.334	0.766	372.47	1.140	1.020
0.288	0.749	373.91	1.248	1.037	0.413	0.834	368.25	1.071	1.091	0.372	0.790	370.26	1.114	1.044
0.353	0.784	369.14	1.198	1.150	0.486	0.867	364.51	1.048	1.135	0.411	0.816	368.25	1.095	1.042
0.396	0.819	366.26	1.198	1.139	0.547	0.893	361.39	1.046	1.168	0.450	0.840	366.03	1.088	1.046
0.660	0.931	354.82	1.107	1.159	0.619	0.918	358.28	1.037	1.188	0.606	0.909	358.88	1.055	1.064
0.734	0.951	352.37	1.088	1.146	0.682	0.936	355.70	1.034	1.232	0.687	0.935	355.60	1.045	1.078
0.750	0.954	351.99	1.080	1.151	0.778	0.959	352.22	1.029	1.279	0.748	0.953	353.26	1.043	1.055
0.818	0.969	349.95	1.065	1.150	0.842	0.976	349.48	1.051	1.183	0.794	0.966	352.00	1.032	0.978
0.896	0.985	347.79	1.052	1.074	0.909	0.986	347.86	1.033	1.279	0.844	0.973	350.37	1.024	1.091
0.959	0.996	346.13	1.043	0.822	0.967	0.997	345.98	1.040	0.815	0.926	0.988	347.93	1.016	1.122
ETBE (1) + Ethylbenzene (2)														

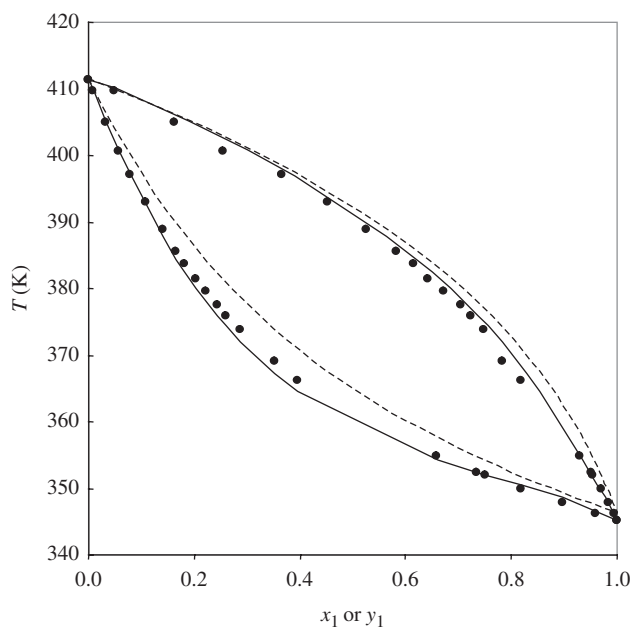


Figure 1. T - x_1 - y_1 diagram for ETBE (1) + p -xylene (2) at 101.3 kPa: (●) experimental data; (—) Wilson correlation; (---) ASOG prediction.

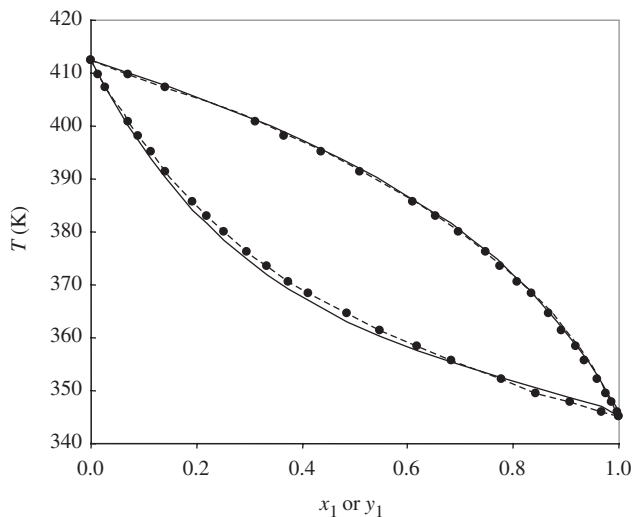


Figure 2. T - x_1 - y_1 diagram for ETBE (1) + m -xylene (2) at 101.3 kPa: (●) experimental data; (—) Wilson correlation; (---) ASOG prediction.

The fugacity coefficients were calculated by virial equation truncated at the second term. The second virial coefficients were calculated by the use of Pitzer's correlation [2] for the second virial coefficient with Tsonopoulos modification for polar molecules [10].

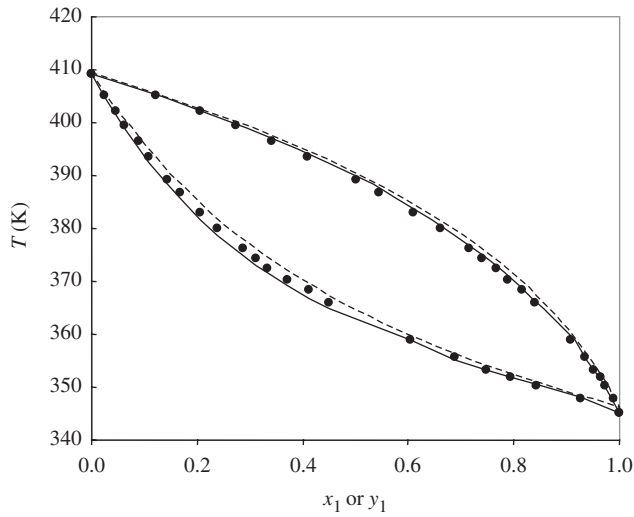


Figure 3. T - x_1 - y_1 diagram for ETBE (1) + ethylbenzene (2) at 101.3 kPa: (●) experimental data; (—) Wilson correlation; (---) ASOG prediction.

Table 3. Antoine coefficients.

Compound ^a	A	B	C
ETBE ^b	15.7267	3874.82	3.24
<i>p</i> -Xylene	14.7186	3757.21	-38.99
<i>m</i> -Xylene	14.7837	3790.23	-38.89
Ethylbenzene	14.1588	3397.18	-53.35

^aAucejo [9] *et al.* ^bArce *et al.* [6].

The mixing rules proposed by Prausnitz *et al.* [11] for the calculation of binary acentric factor and pseudocritical temperature and pressure (ω_{12} , T_{c12} , and P_{c12}) were used and they are calculated as:

$$\omega_{12} = \frac{\omega_1 + \omega_2}{2} \quad (4)$$

where ω_1 and ω_2 are the acentric factors of compounds 1 and 2, and

$$T_{c12} = (1 - k_{ij})(T_{c1}T_{c2})^{0.5} \quad (5)$$

where T_{c1} and T_{c2} are the critical temperatures of compounds 1 and 2, and k_{ij} is the binary interaction constant proposed by Lee and Chen [12]; for both the mixtures $k_{ij} = 0.10$.

Also,

$$P_{c12} = \frac{Z_{c12}RT_{c12}}{V_{c12}} \quad (6)$$

Table 4. Published parameters used for the calculation of fugacity coefficients: critical temperature, T_c , critical pressure, P_c , critical volume, V_c , critical compression factor, Z_c , and acentric factor, ω of pure compounds.

	T_c (K)	P_c (Pa)	V_c m ³ kmol ⁻¹	Z_c	ω
ETBE ^a	509.40	2.9347E6	0.3945	0.273	0.337
<i>p</i> -Xylene ^b	616.20	3.5110E6	0.3780	0.259	0.322
<i>m</i> -Xylene ^b	617.00	3.5410E6	0.3750	0.259	0.327
Ethylbenzene ^b	617.15	3.6090E6	0.3740	0.263	0.304

^aTsonopoulos and Dymond [10]; ^bPoling *et al.* [2].

where Z_{c12} is calculated by,

$$Z_{c12} = \frac{Z_{c1} + Z_{c2}}{2} \quad (7)$$

Z_{c1} and Z_{c2} are the critical compressibility factors, and V_{c12} is defined by the expression

$$V_{c12} = \left(\frac{V_{c1}^{1/3} + V_{c2}^{1/3}}{2} \right)^3 \quad (8)$$

where V_{c1} and V_{c2} are the critical volumes of compounds 1 and 2. Values of P_c , V_c , T_c , Z_c and ω for the pure compounds have been obtained from the literature, and are presented in table 4.

The activity coefficients were correlated with the Margules, van Laar, Wilson, NRTL and UNIQUAC equations [2]. To determine the constants of each model, we have used the method suggested by Gess *et al.* [13]. Estimation of the parameters for the equation was based on the iterative solution, using the maximum likelihood regression of the objective function Q_i [14], with the activity coefficients obtained from the consistency test as experimental values,

$$Q_i = \sum \left(\frac{\gamma_{\text{exptl}} - \gamma_{\text{calcd}}}{\gamma_{\text{exptl}}} \right)^2 \quad (9)$$

where γ_{exptl} are the activity coefficients calculated from experimental data and γ_{calcd} are the coefficients calculated with the correlations. The parameters, the average deviation in T (ΔT), and the average deviation in y (Δy) are listed in table 5. The ASOG [15] method was also used to obtain predictions. The experimental, correlated by Wilson and predicted by ASOG T - x_1 - y_1 phase diagrams are shown in figures 1 and 2.

The thermodynamic consistency of the experimental data was checked by means of a modified Dechema test [16] where the fugacity coefficients are calculated by virial equation, and activity coefficients are calculated by using the four-suffix Margules equation,

$$\bar{g}^E/RT = x_1x_2[Ax_2 + Bx_1 - Dx_1x_2] \quad (10)$$

with the corresponding activity coefficients,

$$\ln \gamma_1 = x_2^2[A + 2(B - A - D)x_1 + 3Dx_1^2] \quad (11)$$

$$\ln \gamma_2 = x_1^2[B + 2(A - B - D)x_2 + 3Dx_2^2] \quad (12)$$

Table 5. Correlation parameters for activity coefficients and average deviation for the studied systems.

Equation	A ₁₂	A ₂₁	ΔT(K)	Δy ₁
ETBE (1) + <i>p</i> -xylene (2)				
Margules ^a	0.2120	0.7547	0.55	0.0095
Van Laar ^a	0.3120	0.9489	0.29	0.0075
Wilson ^b	-1840.50	5158.76	0.25	0.0076
NRTL ^c (α ₁₂ =0.89)	13754.46	1071.30	0.40	0.0099
UNIQUAC ^d	3599.76	-2032.59	0.27	0.0080
ETBE (1) + <i>m</i> -xylene (2)				
Margules ^a	0.1551	0.4105	0.38	0.0027
Van Laar ^a	0.1657	0.6569	0.47	0.0044
Wilson ^b	-2290.85	4962.11	0.41	0.0036
NRTL ^c (α ₁₂ =1.25)	9765.53	589.75	0.22	0.0033
UNIQUAC ^d	3654.76	-2187.28	0.40	0.0038
ETBE (1) + ethylbenzene (2)				
Margules ^a	0.1855	0.3610	0.23	0.0036
van Laar ^a	0.2021	0.4111	0.20	0.0034
Wilson ^b	-1543.06	3037.14	0.20	0.0034
NRTL ^c (α ₁₂ =0.30)	3283.52	-1770.53	0.21	0.0035
UNIQUAC ^d	2613.76	-1744.18	0.18	0.0034

^aMargules and van Laar constants (dimensionless).

^bWilson's interaction parameters (J mol⁻¹).

^cNRTL's interaction parameters (J mol⁻¹).

^dUNIQUAC's interaction parameters (J mol⁻¹).

Parameters A, B, and D were estimated using the error-in-variables regression maximum likelihood technique. The constraint equation for the regression was

$$F = P - \left(\frac{x_1 \gamma_1^* f_1^0}{\phi_1} + \frac{x_2 \gamma_2^* f_2^0}{\phi_2} \right). \quad (13)$$

Here the asterisk (*) denotes a calculated or predicted value. An experimental value has no asterisk; f_1^0 and f_2^0 are the standard state fugacities. The errors in the prediction of y_1 were calculated. Predicted y_1^* values were obtained using the equation,

$$y_1^* = \frac{x_1 \gamma_1^* f_1^0}{\phi_1 P^*}. \quad (14)$$

An average deviation was calculated from,

$$\text{Average deviation} = \frac{\sum_{i=1}^n |\Delta y|}{n}. \quad (15)$$

Here $\Delta y = y_1 - y_1^*$ and n = number of experimental data points. To pass the consistency test, a system must have an average deviation <0.01. All systems included in this work have passed this consistency test. In table 6, we show these results and the values of A, B, and D of equations (10–12).

There was also the Margules constant test carried out using the program of Gess *et al.* [13]. The Margules constant can be used to indicate the ideality of a system. Systems which yield a Margules constant whose absolute value is less than 0.60 can be considered ideal, while those which yield an absolute value greater than 0.60 can be

Table 6. Results of the Thermodynamic Consistency and Margules constant tests.

System	Average deviation Δy_1	A	B	D	Margules constant
ETBE (1) + <i>p</i> -xylene (2)	0.0091	0.4170	0.8906	0.6955	0.5827
ETBE (1) + <i>m</i> -xylene (2)	0.0048	0.3145	0.5477	0.5762	0.3598
ETBE (1) + ethylbenzene (2)	0.0039	0.2531	0.4165	0.2389	0.3056

considered nonideal. This criterion for classification, however, is not rigorous. Table 6 shows the values of this constant.

4. Conclusions

Isobaric vapor–liquid equilibria data at 101.3 kPa, not previously reported in the literature, have been measured for the systems ETBE + *m*-xylene, ETBE + *p*-xylene, and ETBE + ethylbenzene. These data are necessary to study removal of pollutants owed to gasoline spills from ground water. Data were tested and considered thermodynamically consistent.

Three systems present an almost ideal behavior. Parameters for activity coefficient models were also reported. Correlations for both systems are very accurate and similar. The ASOG method prediction shows agreement with experimental data qualitatively in the studied systems.

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