

LLE for the extraction of alcohol from aqueous solutions with diethyl ether and dichloromethane at 293.15 K, parameter estimation using a hybrid genetic based approach

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ABSTRACT

Experimental liquid–liquid equilibrium (LLE) data for the extraction of methanol, ethanol and 1-propanol from water by diethyl ether and dichloromethane at 293.15 K and at ambient pressure were investigated. Data for the binodal curves have been determined by cloud-point titration method and conjugate points on tie-line were obtained by correlating the refractive index of the binodal curves as a function of composition. The experimental ternary (liquid + liquid) equilibrium data have been estimated using the NRTL and UNIQUAC activity coefficient models to obtain the binary interaction parameters of these components by a combination of Levenberg–Marquardt method and the genetic algorithm based method. The distribution coefficients and the selectivity factor of the solvent used were calculated and presented. From our experimental and calculated results, we conclude that for the extraction of alcohol from aqueous solutions with dichloromethane solvent has a higher selectivity factor than the diethyl ether solvent.

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1. Introduction

The recovery and removal of alcohols such as methanol, ethanol, propanol and butanol from aqueous solutions resulting from liquid–liquid extraction processes are important according to the theoretical and industrial concerns. Liquid–liquid extraction provides an important alternative to distillation for the recovery of alcohol from aqueous solutions. The possible extraction agents are diethyl ether (DEE) and dichloromethane (DCM) which were selected as good solvents for alcohols (C1, C2, C3).

Recently many authors have reported some works on the use of liquid–liquid extraction to selectively remove alcohol from water. Solvent extraction techniques have the potential for tremendous energy savings especially in the recovery of fermentation products. The latter have a direct impact on the economics of the entire fermentation process [1–8]. The equilibrium condition between the organic and aqueous phases is a very important aspect of the liquid–liquid extraction process. Four desirable characteristics for a solvent are as follows: (a) a high distribution coefficient for alcohol, (b) a high selectivity for alcohol over water, and (c) a low solubility in water.

Higher values of the equilibrium coefficient indicate the ability of the solvent to recover relatively large quantities of solute, thereby reducing the solvent requirement, and corresponding material cost of the extraction equipment. The ability to selectively remove alcohol from aqueous solution may be described by relative selectivity, which is the ratio of the equilibrium distribution coefficient for alcohol over that for water.

In the present work, mutual solubilities of water and solvents at several temperatures have been previously measured. Also 6 ternary liquid–liquid equilibrium (LLE) phase diagrams of DCM or DEE (1) + water + C1–C3 alcohols (3) systems at 293.15 K and at atmospheric pressure were measured. The 6 measured systems are: {DCM + water + methanol, or ethanol, or 1-propanol}; {DEE + water + methanol, or ethanol, or 1-propanol}. However, equilibrium data for some ternary systems were not found in the literature. The experimented LLE data of the binary and ternary systems were correlated using two activity coefficient models; the NRTL [9] and UNIQUAC [10] models.

2. Experimental

2.1. Materials

The mass purities and source of the chemicals employed are as follows: methanol, ethanol and 1-propanol (Merck, >99.2%);

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Table 1
Refractive index (n_D) at $T = 293.15$ K.

Component	Refractive index (n_D)	
	Exp	Lit
Water	1.3334	1.3333
Methanol	1.3288	1.3290
Ethanol	1.3609	1.3611
1-Propanol	1.3857	1.3854
Diethyl ether	1.3522	1.3524
Dichloromethane	1.4240	1.4241

diethyl ether and dichloromethane (Merck, >99.5%). The measured refractive indices (n_D) of pure components at $T = 293.15$ K along with the literature data available [11–13], were shown in Table 1.

2.2. Apparatus and procedure

The binodal curve and tie-line values have been determined by means of the cloud-point titration method [14–17]. The measurements were made in a glass equilibrium thermostated cell equipped with a magnetic stirrer. The cell temperature was measured with mercury-in-glass thermometer with uncertainty of ± 0.1 K. A known composition, homogeneous and transparent (alcohol + water) mixtures, was prepared in a glass equilibrium cell at constant temperature. The mixture was made by mass using a Sartorius analytical balance (Model Nahita YP402N, accurate to ± 0.001 g). Then diethyl ether or dichloromethane was progressively added to the mixture using a micro-burette.

The cloud point was determined by observing the transition from a homogeneous to a heterogeneous mixture as indicated by the mixture turbidity (cloudiness). This pattern was convenient for providing the water-rich side of the curves. On the other hand, data for the solvent-rich side of the curves were obtained by titrating homogeneous alcohol–solvent with water until the onset of cloudiness due to apparition of second phase.

The tie-line data for experimental results was determined by using a water jacket glass cell with a volume about 200 cm³. Eight systems of heterogeneous mixtures were prepared in this study. Glass cells were filled with each of these mixtures and vigorously stirred for 2 h. Afterward the stirrer was turned off; the contents were immediately introduced into a separator funnel. After complete phase separation, a suitable amount of each layer was removed for analysis. The samples of both phases were collected and the tie-line values were obtained by measuring the refractive index for the two phases at equilibrium.

The solubilities of water and solvent were determined by applying a synthetic method. A weighed amount of the first substance was introduced into the cell; the second was added until permanent heterogeneity (cloudiness) had been observed. An ultra-accurate titrator with an accuracy of ± 0.001 cm³ was used.

3. Results and discussion

The LLE data for the binary system DCM or DEE (1) + water (2) at 288.15, 293.15, 298.15, and 301.15 K, and at atmospheric pressure, were listed in Table S1. The solubility of DCM or DEE in the aqueous phase was less than 0.3 mol% and 1.5 mol% respectively, while the solubility of water in the DCM or DEE phase is higher than 0.25 mol% and 3.91 mol% in the investigated temperature range. The mutual solubility of DCM or DEE and water increases as the temperature is raised.

The compositions of mixtures on the binodal curve of ternary systems of {water (1) + alcohol (2) + solvent (3)} at $T = 293.15$ K are

Table 2
The UNIQUAC structural parameters r and q [available within the HYSYS® database].

Component	r	q
Water	0.9200	1.3997
Methanol	1.4311	1.4320
Ethanol	2.1054	1.9720
1-Propanol	2.7799	2.5129
Diethyl ether	3.3949	3.0160
Dichloromethane	2.2564	1.9880

reported in Tables S2 and S3, the estimated uncertainties in the temperature and refractive index (n_D) values are accurate to ± 0.1 K and 0.001% respectively. Tables S4 and S5 shows the experimental tie-line compositions of the equilibrium phases, the maximum deviation for any data point was 0.02% of its absolute value.

The NRTL and UNIQUAC activity coefficient models were used to correlate the experimental results. The UNIQUAC activity coefficient model for component i are expressed as follows:

$$\ln \gamma_i = \ln \frac{\phi_i}{x_i} + \frac{z}{2} q_i \ln \frac{\theta_i}{\phi_i} + l_i - \frac{\phi_i}{x_i} \sum_{j=1}^n x_j l_j + q_i \times \left[1 - \ln \left(\sum_{j=1}^n \theta_j \tau_{ji} \right) - \sum_{j=1}^n \frac{\theta_j \tau_{ij}}{\sum_{k=1}^n \theta_k \tau_{kj}} \right] \quad (1)$$

where $\theta_i = q_i x_i / q_T$, $q_T = \sum_{k=1}^n q_k x_k$, $\phi_i = r_i x_i / r_T$, $r_T = \sum_{k=1}^n r_k x_k$, $l_i = (z/2)(r_k - q_k) + 1 - r_k$, $\tau_{ij} = \exp(-A_{ij}/T)$, $z = 10$ (coordination number).

The values of r and q used for these ternary systems are presented in Table 2.

The NRTL model for a solution with n components is in the following form:

$$\ln \gamma_i = \frac{\sum_{j=1}^n \tau_{ji} G_{ji} x_j}{\sum_{k=1}^n G_{ki} x_k} + \sum_{j=1}^n \frac{x_j G_{ij}}{\sum_{k=1}^n G_{kj} x_k} \left(\tau_{ij} - \frac{\sum_{i=1}^n x_i \tau_{ij} G_{ij}}{\sum_{k=1}^n G_{kj} x_k} \right) \quad (2)$$

where $\tau_{ji} = (g_{ji} - g_{ii})/RT = A_{ji}/T$, $G_{ji} = \exp(-\alpha_{ji} \tau_{ji})$, $\alpha_{ji} = \alpha_{ij}$

Ternary LLE experimental data were used to determine the optimum NRTL and UNIQUAC binary interaction parameters among water, alcohol, and solvent. The thermodynamic models were fitted to experimental data using an iterative computer program, based on combination of the Newton–Raphson method and the hybrid genetic algorithm [18,19].

The root-mean-square deviation (RMSD) is a measure of the agreement between the experimental data and the calculated values. The RMSD value is defined as follows [20]:

$$\text{RMSD} = \left[\sum_i \sum_j \sum_k \frac{(x_{ijk}^{\text{exp}} - x_{ijk}^{\text{cal}})^2}{6M} \right]^{1/2} \quad (3)$$

where M is the total number of tie-lines, x^{exp} and x^{cal} are the experimental and calculated mass fractions, and the subscripts i, j and k designate the component, phase and tie-line respectively.

The ternary LLE of each system at 293.15 K are plotted in the form of Gibbs triangles in Figs. 1–6. In the case of methanol, ethanol, 1-propanol, the hydrogen bonding in water is stronger than that in the alcohol, since these compounds are completely miscible with water.

The slopes of the tie-lines presented in the figures show that methanol is more soluble in water than in DCM or DEE, while ethanol, 1-propanol are more soluble in DCM or DEE than in water.

Table 3
The UNIQUAC binary interaction parameters and RMSD values for (water + alcohol + solvent) ternary system.

System	UNIQUAC parameters (cal/mol)			RMSD
	<i>i</i> – <i>j</i>	<i>A</i> _{<i>ij</i>}	<i>A</i> _{<i>ji</i>}	
Water (1) + methanol (2) + dichloromethane (3)	1–2	–33.74033	–463.6400	0.0747
	1–3	387.29188	538.58985	
	2–3	833.83401	–181.7904	
Water (1) + ethanol (2) + dichloromethane (3)	1–2	760.28441	–21.30905	0.0447
	1–3	109.68397	777.66522	
	2–3	–72.94883	320.8548	
Water (1) + 1-propanol (2) + dichloromethane (3)	1–2	1403.9433	–256.7923	0.0068
	1–3	877.2448	702.79888	
	2–3	–25.15918	685.53263	
Water (1) + methanol (2) + diethyl ether (3)	1–2	40.7518	–658.9760	0.0695
	1–3	771.7189	755.4027	
	2–3	322.7939	–408.5670	
Water (1) + ethanol (2) + diethyl ether (3)	1–2	–817.5032	76.11281	0.0719
	1–3	882.3018	599.48309	
	2–3	–402.6348	–326.0699	
Water (1) + 1-propanol (2) + diethyl ether (3)	1–2	400.82837	3.06134	0.0024
	1–3	1743.715	608.87276	
	2–3	–266.8471	860.38236	

Table 4
The NRTL binary interaction parameters and RMSD values for (water + alcohol + solvent) ternary system.

System	NRTL parameters (cal/mol)			α_{ij}	RMSD
	<i>i</i> – <i>j</i>	<i>A</i> _{<i>ij</i>}	<i>A</i> _{<i>ji</i>}		
Water (1) + methanol (2) + dichloromethane (3)	1–2	238.5741	566.9689	0.2	0.0114
	1–3	749.2058	1498.0961		
	2–3	–359.8070	1108.6181		
Water (1) + ethanol (2) + dichloromethane (3)	1–2	1602.3454	–100.8599	0.2	0.0608
	1–3	1678.3712	777.1668		
	2–3	–436.4442	–211.4323		
Water (1) + 1-propanol (2) + dichloromethane (3)	1–2	203.3377	1349.5038	0.2	0.0359
	1–3	1146.2821	839.8992		
	2–3	1201.4097	–587.3958		
Water (1) + methanol (2) + diethyl ether (3)	1–2	–146.7742	1021.8459	0.2	0.0168
	1–3	760.7554	1299.6546		
	2–3	25.2773	258.2981		
Water (1) + ethanol (2) + diethyl ether (3)	1–2	863.4116	–326.6993	0.2	0.0514
	1–3	1342.5248	1370.7560		
	2–3	–741.9869	124.2468		
Water (1) + 1-propanol (2) + diethyl ether (3)	1–2	1082.055	–508.4697	0.2	0.0033
	1–3	1577.7041	837.9551		
	2–3	244.2815	644.0485		

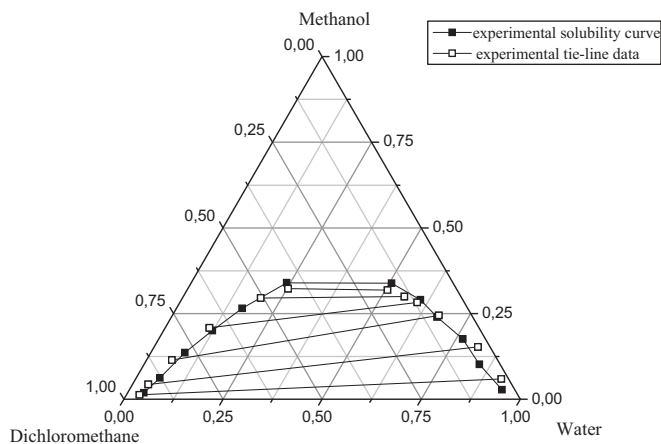


Fig. 1. Ternary diagram for LLE of {water (1) + methanol (2) + dichloromethane (3)} at 293.15 K.

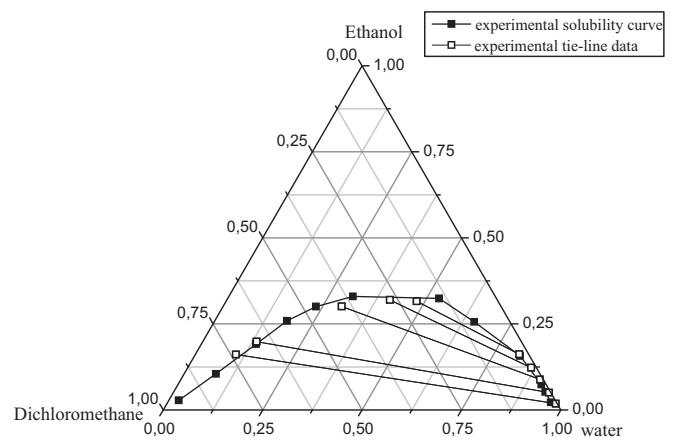


Fig. 2. Ternary diagram for LLE of {water (1) + ethanol (2) + dichloromethane (3)} at 293.15 K.

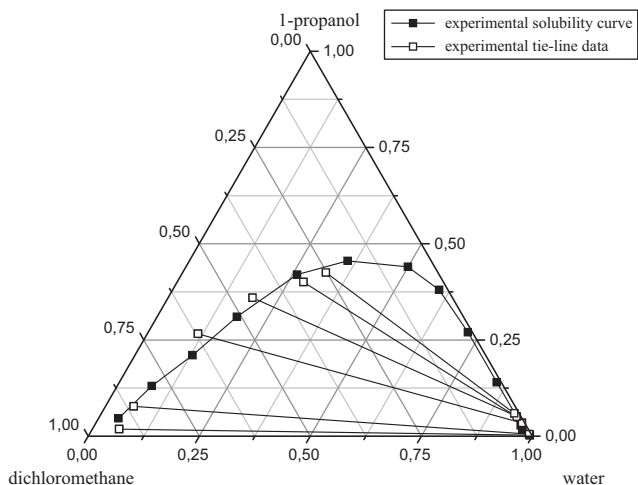


Fig. 3. Ternary diagram for LLE of {water (1) + 1-propanol (2) + dichloromethane (3)} at 293.15 K.

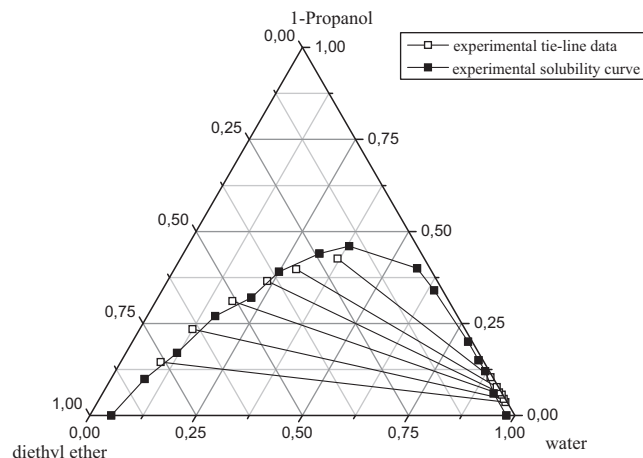


Fig. 6. Ternary diagram for LLE of {water (1) + 1-propanol (2) + diethyl ether (3)} at 293.15 K.

The correlated parameters obtained using the UNIQUAC and NRTL models for the 6 ternary systems are given in Tables 3 and 4. The calculated of the mean deviations (RMSD) values of experimented and calculated phase compositions are listed in Tables 3 and 4. The maximum calculated RMSD between the experimental and calculated mass fractions was 0.06.

3.1. Distribution coefficients and separation factors

The distribution coefficient, D_i , or the ratio of mass fraction of the i th component in DEE or DCM rich-phase, x_{i3} , to that in water rich-phase, x_{i1} , is defined in the following form [21]

$$D_i = \frac{x_{i3}}{x_{i1}} \quad (4)$$

The effectiveness of extraction of alcohols (C1–C3) by DEE or DCM from (water + alcohol) mixtures is given by the selectivity (S) by the following equation [21]

$$S = \frac{D_2}{D_1} \quad (5)$$

where D_2 and D_1 are the distribution coefficient of alcohol (x_{21}/x_{23}) and the distribution coefficient of water (x_{11}/x_{13}), respectively. The experimental values of selectivity factors (S) are given in Table 5. According to Table 5, the selectivity is not constant over the whole two-phase region and the selectivity in all cases is greater than one which means that the extraction of alcohol by dichloromethane or diethyl ether is possible.

3.2. Correlations

The reliability of experimentally measured tie-line data for each system is ascertained by applying the Othmer–Tobias (Eq. (6)) [22] and Hand (Eq. (7)) [23]

$$\ln \left[\frac{1 - x_{33}}{x_{33}} \right] = a_1 + b_1 \ln \left[\frac{1 - x_{21}}{x_{21}} \right] \quad (6)$$

$$\ln \left(\frac{x_{13}}{x_{33}} \right) = a_2 + b_2 \ln \left(\frac{x_{11}}{x_{21}} \right) \quad (7)$$

where a_1 , b_1 , a_2 , and b_2 , the parameters of the Othmer–Tobias and Hand correlations, respectively

The correlations are given in Figs. 7–10, the correlation factor (R^2) being approximately unity and the linearity of the plots indicates the degree of consistency of the measured LLE values in this study.

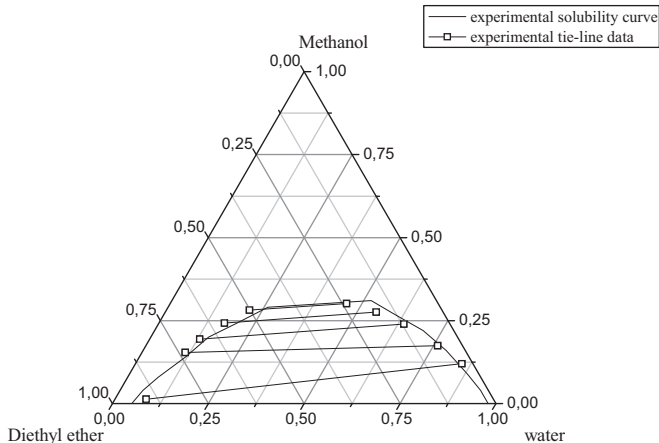


Fig. 4. Ternary diagram for LLE of {water (1) + methanol (2) + diethyl ether (3)} at 293.15 K.

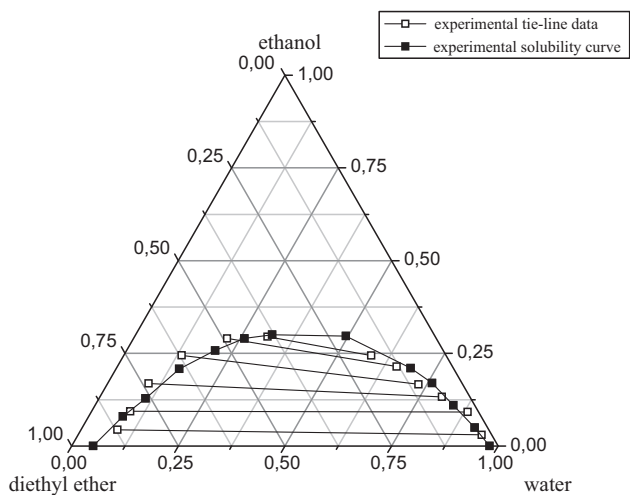


Fig. 5. Ternary diagram for LLE of {water (1) + ethanol (2) + diethyl ether (3)} at 293.15 K.

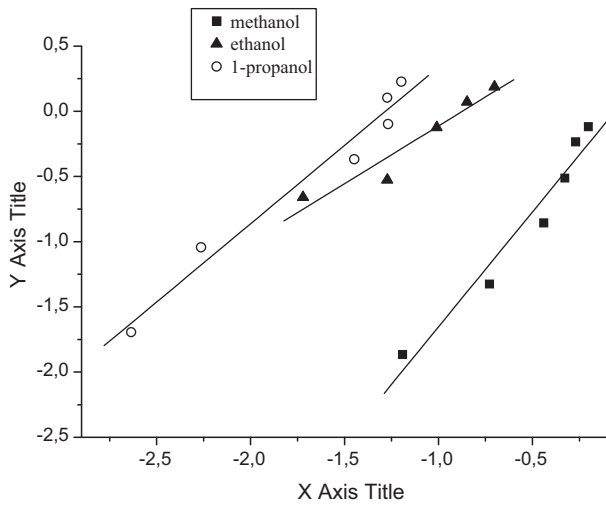


Fig. 7. Hand plots of water–alcohol–dichloromethane ternary systems.

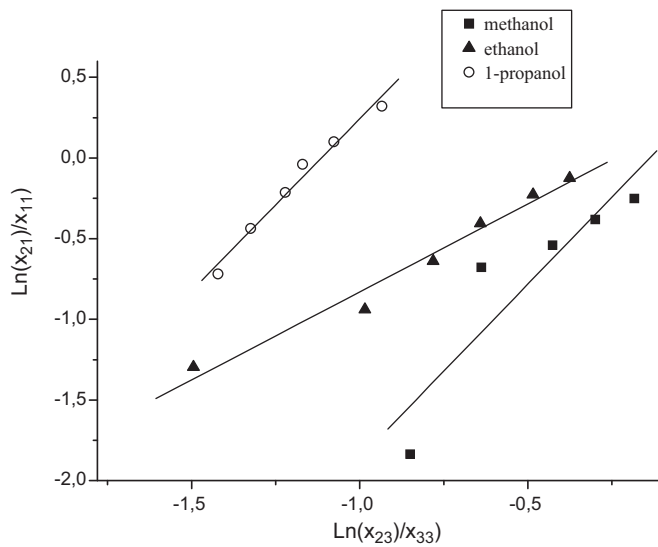


Fig. 8. Hand plots of water–alcohol–diethyl ether ternary systems.

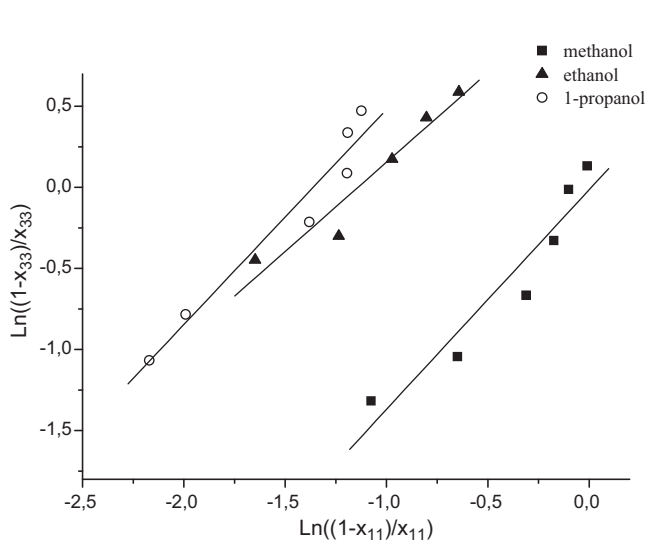


Fig. 9. Othmer–Tobias plots of water–alcohol–dichloromethane ternary systems.

Table 5

The experimental distribution coefficients of water (D_1) and alcohol (D_2) and the selectivity factor (S).

D_1	D_2	S
(Water + methanol + dichloromethane)		
0.0358	0.2185	6.1077
0.0486	0.2836	5.8355
0.0946	0.4679	4.9438
0.1860	0.7392	3.9747
0.3543	0.9856	2.7819
0.5004	1.0141	2.0267
(Water + ethanol + dichloromethane)		
0.1041	8.6667	83.2613
0.1435	3.9325	27.4060
0.3298	3.4061	10.3292
0.4741	2.6064	5.4978
0.5890	1.9494	3.3099
(Water + 1-propanol + dichloromethane)		
0.0608	8.0870	132.9929
0.0643	14.3704	223.3160
0.1191	7.7690	65.2515
0.2030	7.0848	34.9042
0.3028	7.9781	26.3459
0.3473	7.2034	20.7404
(Water + methanol + diethyl ether)		
0.0961	0.1097	1.1418
0.1489	0.8804	5.9136
0.2039	0.8110	3.9774
0.3109	0.8804	2.8318
0.4704	0.9340	1.9854
(Water + ethanol + diethyl ether)		
0.0896	1.4474	16.1509
0.1027	1.0251	9.9824
0.1197	1.2740	10.6389
0.1859	1.4664	7.8884
0.3354	1.3551	4.0400
0.5373	1.2080	2.2483
(Water + 1-propanol + diethyl ether)		
0.0979	3.9918	40.7669
0.1310	5.1929	39.6428
0.1909	5.5062	28.8431
0.2520	5.7968	23.0021
0.3124	5.1543	16.4971
0.4154	4.1050	9.8826

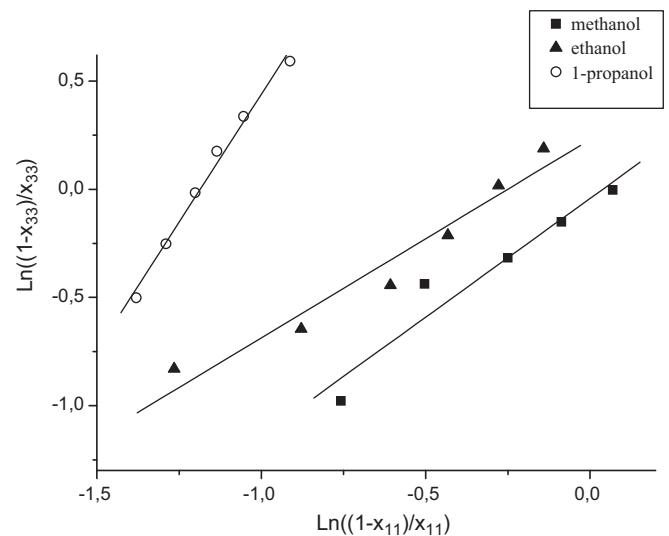


Fig. 10. Othmer–Tobias plots of water–alcohol–diethyl ether ternary systems.

3.3. Parameter estimation algorithm

A hybrid algorithm genetic based approach has been used in this study; the genetic approach is an innovative method for solving optimization problems applied to constrained and unconstrained problems [18,24]. The genetic algorithm is based on repeatedly change of a population from individual solutions. At each step, the genetic algorithm selects individuals at random from the current population of species parents and uses them to produce new generations for the next generation. Over successive generations, the population evolves toward an optimal solution. We can apply the genetic algorithm to solve a variety of complex optimization problems that are not well suited for standard optimization algorithms, including problems in which the objective function is discontinuous, non differentiable, stochastic, or highly non-linear. In each step, genetic algorithm uses three main types of rules (operators) to create the next generation of the population: selection, crossover, and mutation.

The selection is carried out by choosing pairs of individuals surviving from one generation to another and those involved in the reproduction process of the future population. This selection is based on the adaptation of individuals. A certain percentage of the population size is maintained from one generation to another, another percentage, called survival, remains constant over generations. The most traditional method of selection, developed by Goldberg, is based on a random lottery wheel bias. The Goldberg scroll wheel enables the random selection of individuals having the highest fitness function, without banning the selection of less well-adapted individuals. Indeed, it is important to allow some degree of weak selection of individuals to ensure a degree of genetic diversity within the future generation, to make certain that a good exploration of the solution space should be obtained.

The crossover allows the enrichment of the population by acting on the structure of chromosomes. The crossing is applicable to two individuals drawn randomly from a population above the current population. These two individuals are mated to give birth to two other individuals. Despite the randomness, this exchange of information gives genetic algorithms power in their work: sometimes “good” genes from one parent will replace the “bad” genes and create another son better adapted to the environment.

The mutation operator for all these individuals was generated in the new population. The mutation provides genetic algorithms property of flexibility in space. This property indicates that the genetic algorithm will be able to reach all points of the space. In the case of binary encoding, the traditional method, after determining the coordinates to mutate, is to reverse a bit in a chromosome.

The hybrid algorithm in this work is a combination between a genetic method and a classical method such as Levenberg–Marquardt, the basic steps of the algorithm can be summarized as follows:

- 1 Initialization of population of individuals
- 2 Evaluation of the fitness of each individual in that population
- 3 Continue repeatedly on this generation until convergence achieved
- 4 Choose the best-fit individuals for reproduction
- 5 Mate individuals using crossover and mutation operations to give birth to new individuals
- 6 Evaluation of the fitness function of new individuals
- 7 Save least-fit population with new individuals as a vector of solution
- 8 Use the obtained vector by GA as initial vector to be used by Levenberg–Marquardt

- 9 Calculating new vector by Levenberg–Marquardt method
- 10 Checking the objective function
- 11 Repeat calculation until convergence

The purpose of the hybrid method is the benefit of each method, for example the genetic algorithm can be used to get a local minimum and from this value one can use a second method (Levenberg–Marquardt) to ensure that a global minimum is reached. It is to notice that the hybrid function start at the best vector parameter returned by the genetic function. A hybrid function is an optimization method that start running when genetic function stops in order to improve the fitness function. Several methods displayed in the literature can be used as hybrid function; one can cite the simplex search method, pattern search algorithm, large scale optimization (trust region approach), and trust-region reflective optimization. Each hybrid function previously cited can have limitation (facing stabilities problem near local minimum or stopping at this latter) when used alone to calculate the parameters. In this work, the method of Levenberg–Marquardt has been used as a hybrid function [24].

4. Conclusion

In this work, binary LLE for the system diethyl ether or dichloromethane + water was measured in a temperature range of 288.15–303.15 K. It can be concluded that dichloromethane showing low solubilities in water may be an adequate solvent to extract alcohol from its aqueous solutions.

Ternary tie-line data for 6 different systems of DEE or DCM + water + C1–C3 alcohols were measured at 293.15 K.

The NRTL and UNIQUAC activity coefficient models satisfactorily correlated the LLE experimental values and the parameters of the model are presented. The comparison of the values of the mass fraction for the components determined by experimental and calculated means indicates the applicability of these two activity coefficient models for LLE calculations of the (water + alcohol + dichloromethane or diethyl ether) ternary system. The maximum calculated RMSD between the experimental and calculated mass fractions was 0.06.

The separation factor and distribution coefficient for each of organic solvent used in this work were calculated and compared. In the extraction of alcohol from water, the solvent with the higher selectivity and distribution coefficient is preferred. The experimental results indicate the superiority of dichloromethane as the preferred solvent for the extraction of alcohol from its aqueous solutions.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.fluid.2011.07.011.

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