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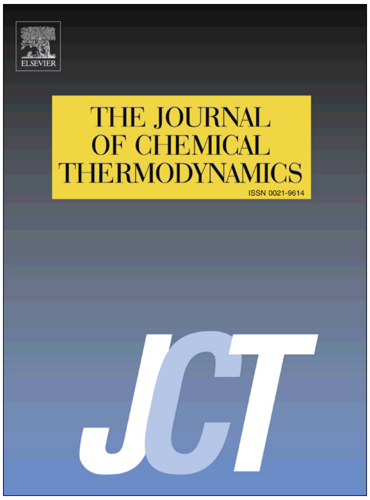

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Highlights

-Vapor-liquid-liquid equilibrium data are determined
-Vapor-liquid-solid and vapor-liquid-liquid-solid equilibrium data are determined
-Results are compared with literature data
-The influence of salt on water + 1-propanol equilibria is studied
-The influence of temperature is also studied.

Keywords

Water; 1-Propanol; NaCl; Isobaric; Vapor-liquid-liquid-solid; Phase diagram

Abstract

Isobaric vapor-liquid-liquid-solid equilibria for the ternary system water + sodium chloride + 1-propanol have been determined at 101.3 kPa by means of a modified recirculating still. The addition of sodium chloride to the solvent mixture results in the appearance of different equilibrium regions. A detailed quantitative analysis of the evolution with temperature of the phase diagram has been carried out.

The experimental data obtained in this way have been compared, on the one hand, with previously published data containing important inconsistencies and, on the other hand, with data calculated by the extended UNIQUAC model.

1. Introduction

A mixed solvent electrolyte system consists of an electrolyte such as a salt dissolved in a solvent, which is a mixture of two or more nonelectrolyte species such as alcohol and water. Knowledge of the experimental equilibrium data of such systems is important because they are ubiquitous in many processes, for example, in the liquid-liquid extraction of mixtures containing salts, extractive distillation and crystallization or regeneration of solvents.

However, there is scant literature available on these systems. There are also no comprehensive studies examining the shapes of the various equilibrium surfaces and regions that arise in them. Finally, they are not even mentioned among all the phase equilibria types in the well-known book by John E. Ricci [1]. Moreover, the different regions in the equilibrium diagrams of these systems are not well understood and, consequently, the relatively small

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amount of existing experimental data in the literature is incomplete. For example, LVE data at salt saturation have been reported without giving the actual value of the salt solubility of the mixture, and LLVE data have been reported without specifying the compositions of the two liquid phases in equilibrium, etc.

Finally, the thermodynamic models developed to represent these systems, such as the electrolyte NRTL model [2], or the extended UNIQUAC model for electrolytes [3], all require the experimental equilibrium data to estimate the model parameters. Until now, the parameters have been estimated from published data although much of it is not thermodynamically consistent, as will be shown below. Consequently, it is necessary to expand, check and complete the existing data for these systems.

In a previous paper [4], we studied the equilibrium diagram of the system water + NaCl + 1-butanol at 101.3 kPa. A detailed analysis of the evolution with temperature of the different equilibrium regions of this system was carried out by investigating the influence of salt on its vapor-liquid-liquid-solid equilibrium. The objective of the present paper is to extend that study with NaCl, which concerned a system containing a solvent consisting of a partially miscible pair, e.g., water+1-butanol, to systems involving a completely miscible solvent.

The chosen system is water + NaCl + 1-propanol at 101.3 kPa. It includes the VL system water + 1-propanol [5, 6] with a minimum boiling azeotrope and two SLV binaries 1-propanol + NaCl and water + NaCl [7, 8]. The equilibrium diagrams near boiling conditions of the VL system and water + NaCl binary are shown in Fig. 1. Moreover, water and 1-propanol are completely miscible but the presence of NaCl can split the mixture into two liquid phases in equilibrium, as can be seen in Fig. 2, where we have plotted the equilibrium diagram of the ternary system in Ref. [9] at 298 K. In this work, equilibrium data are determined and compared with experimental data obtained by various authors. The obtained results permit us to carry out a study of the shape of the phase diagram of the system, and verify the accuracy of calculations done by using a model such as the extended UNIQUAC.

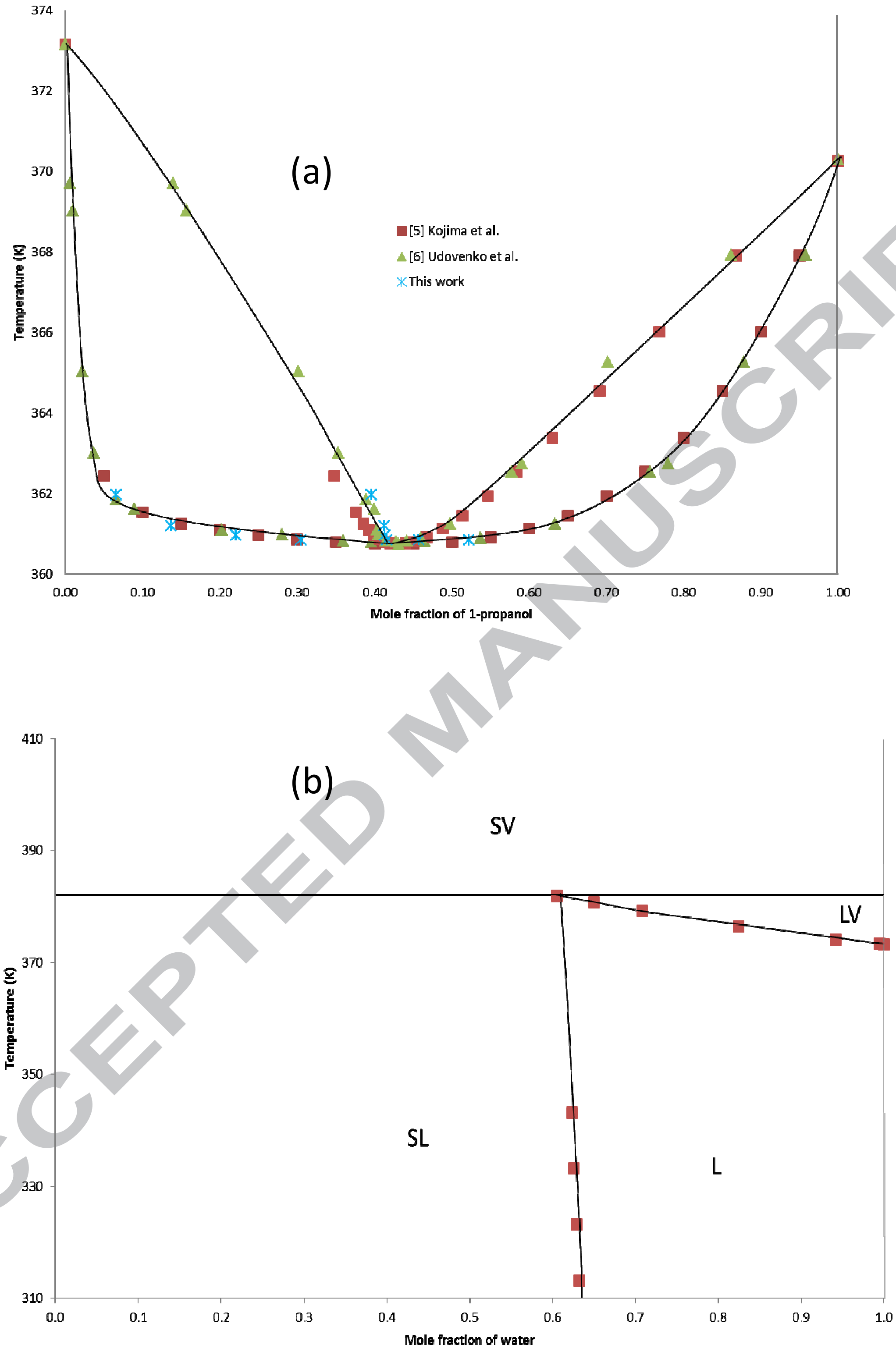


Figure 1. Equilibrium diagrams (mole fraction) of binary system constituents. ___ Tendency lines

a) Txy diagram of water + 1-propanol at 101.3 kPa [5, 6].

b) Txy diagram of water + NaCl at 101.3 kPa [7, 8].

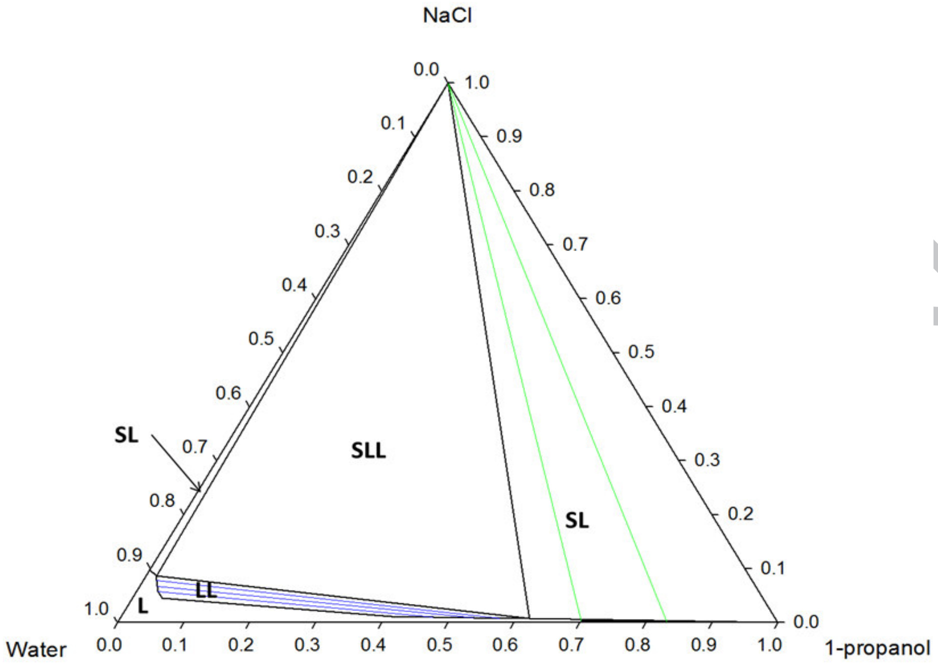


Figure 2. Equilibrium diagram (mole fraction) of the water + NaCl + 1-propanol diagram at 298.15 K [9].

2. Experimental

2.1. Chemicals

Ultrapure water, with a conductivity of less than 1 µS/cm, was used and obtained by means of a MiliQPlus system. The 1-propanol used, was purchased from Merck. The ethanol used as an internal standard for quantitative chromatographic analysis was purchased from VWR. The water content of the organic compounds was checked against the Karl Fischer technique. Analytical grade NaCl was purchased from VWR, and contained less than 0.01 wt. % of inorganic impurities, potassium being the impurity of highest concentration. A chemical sample description is given in Table 1.

2.2. Apparatus and procedures

The equilibrium of the system that is the subject of this work, was determined by means of a modified vapor-liquid Fischer Labodest unit (*Fischer Labor und Verfahrenstechnik*), which was also coupled to an ultrasonic transducer to permit improved mass transfer between phases. The operation of this equipment is described in a previous paper [4]. It employs an ultrasonic probe to enhance the mixing and dispersion of the phases. The experiment is carried out as follows. A mixture of water, 1-propanol and NaCl is placed inside the boiling flask where it is heated at a pressure of 101.3 kPa that is maintained by a Fisher M101 phase equilibrium control system. The boiling temperature is measured using a Pt100 sensor coupled to a digital thermometer (model 3002) with a standard uncertainty of 0.06 K according to its certificate of calibration (scale ITS 90). Using a previously devised method [10], samples of the liquid and vapor phases are taken and analyzed.

Vapor phases are summarily released through a six-port valve into a chromatograph for subsequent analysis by a Thermal Conductivity Detector (TCD). As concerns the liquid phases, they are separated from the solid at the boiling temperature. A small sample of this liquid is introduced into a vial with a known amount of ethanol, which serves as an internal standard. That sample is subsequently analyzed by gas chromatography with TCD to determine the water and 1-propanol content of the liquid phase. Another aliquot of the liquid phase is taken to check its water content against the Karl Fisher Technique (Metrohm 737 KF Coulometer). Finally, yet another aliquot of the liquid phase is collected and the amount of salt it contains determined by a gravimetric method, as the concentration of salt in the liquid samples is high enough.

A Shimadzu GC14B gas chromatograph with a 2 m x 3 mm 80/100 Porapack Q packed column is employed to perform the analysis. When the liquid samples are analyzed, glass wool is put inside the glass insert to avoid NaCl from entering the column. Helium is used as carrier gas at a flow rate of 25 mL/min. The temperature of the oven is set to 463.15 K while the injector and the detector are set to 483.15K. A 100 mA current is used.

The uncertainties were obtained from a statistical calculation of repeated measurements. The temperature standard uncertainty (u) is 0.06 K. The composition relative uncertainty ($u_r = \frac{u}{w}$) is 2% for all components except for the NaCl in the organic phase where the relative uncertainty is 5%.

3. Results

Experimental results. Tables 2 and 3 show the equilibrium data and the boiling temperature of different samples from the S-L_{org}-V and L_{aq}–L_{org}–V regions. The four phase region (S–L_{org}–L_{ac}–V) equilibrium data are recorded in table 4 and remain invariant since they are the only SLLVE data that exists at 101.3 kPa for that system. All these equilibrium data are plotted in Fig. 3 where the boiling point is also indicated. The LLVE region is represented by continuous tie lines joining the two liquid phases in equilibrium, and dashed lines joining the organic liquids with the point that is representative of the vapor phase. In the same way, the SLVE region is represented by continuous lines joining solid and liquid, and dashed lines joining the liquids with the vapor. Lastly, the SLLVE region is represented by a triangle whose three vertices are the solid salt and the compositions of the two liquid phases that are in equilibrium with each other.

Finally, table 5 records several data points corresponding to the vapor-liquid equilibrium when the liquid is homogeneous.

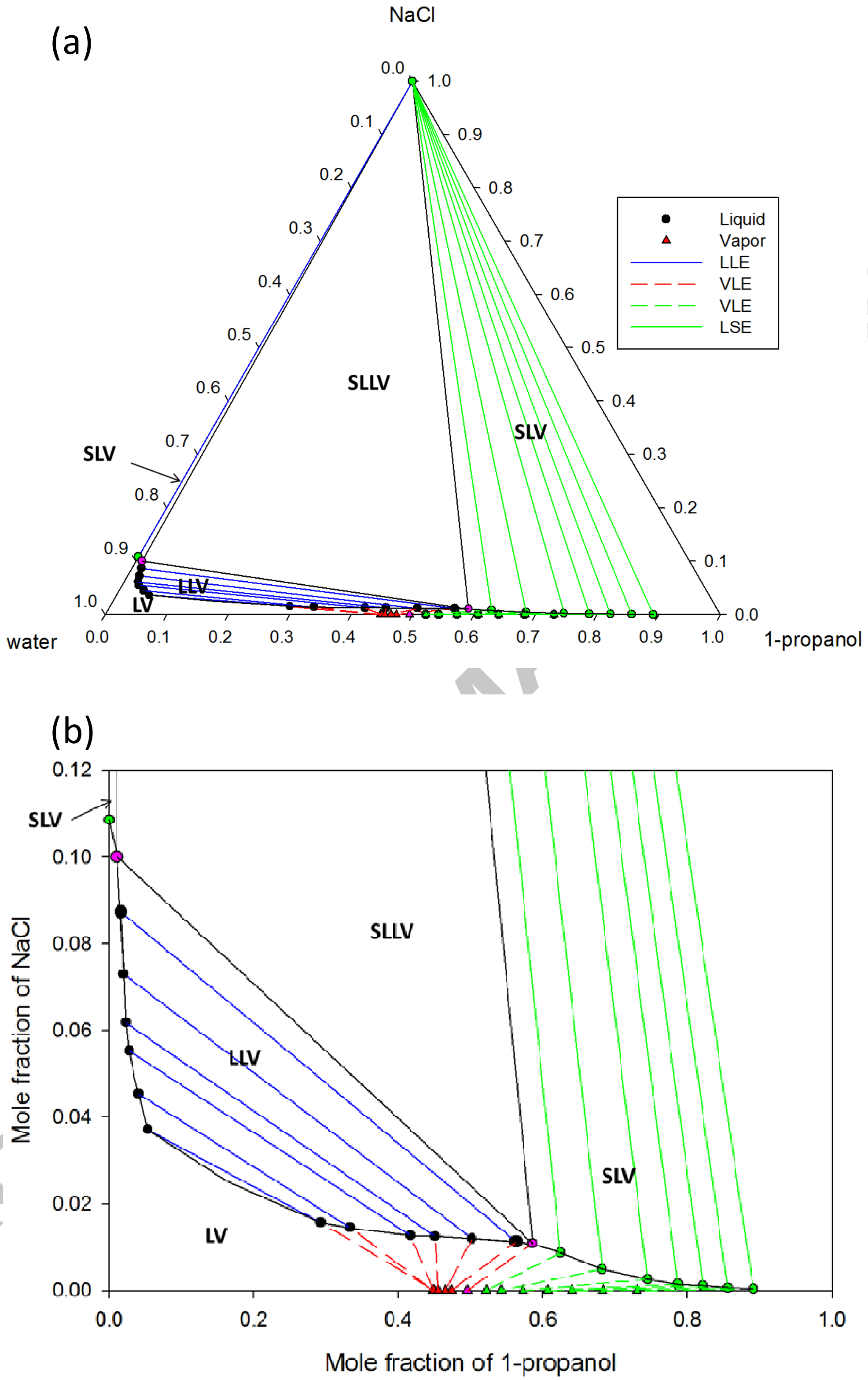
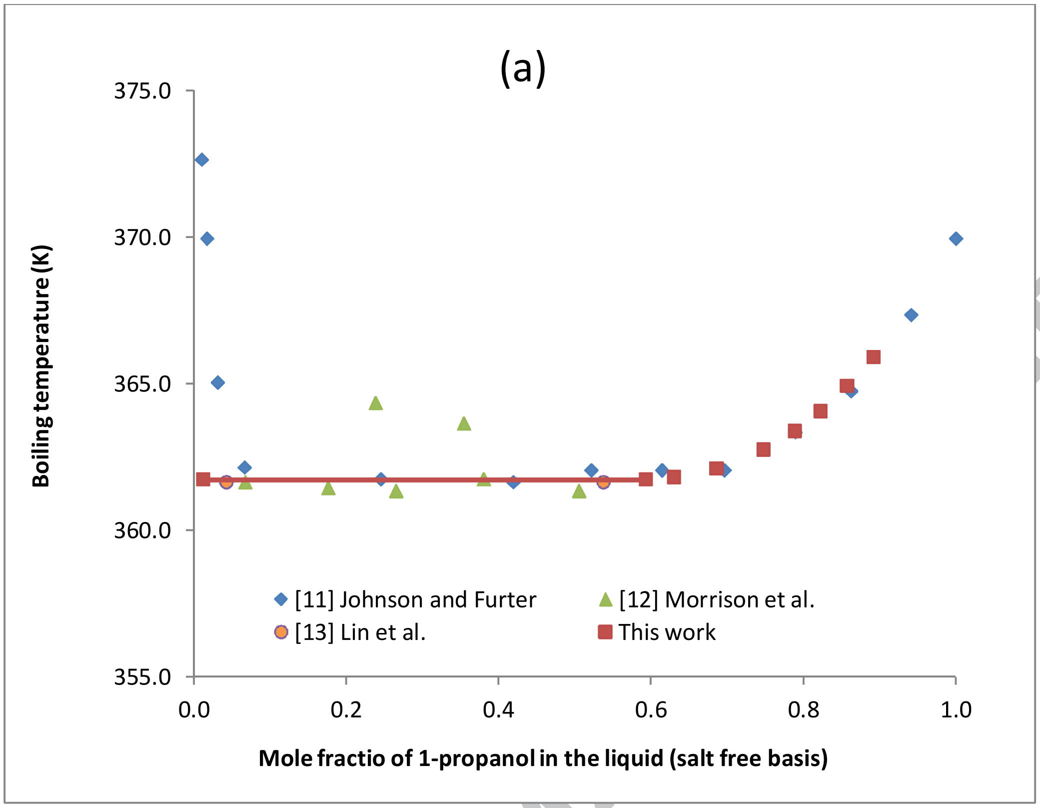


Figure 3. a) Equilibrium diagram (mole fraction) for the water + NaCl + 1-propanol system at 101.3 kPa. b) Enlarged region.

Comparison with previous bibliography results. Various authors have published experimental equilibrium data on the present system. For example, Refs. [5] and [6] report experimental data on the binary system water + 1-propanol. Our experimental data agree perfectly with those data as can be seen in Fig.1a.

With respect to the ternary system, Johnson and Furter [11] determined the effect of the salt on the SLV equilibrium by only studying solutions that are saturated in salt, and by including data from the SLLV region as well. Fig. 4 shows the obtained curve of temperature and mole fraction of 1-propanol in the vapor against the composition of the liquid in a salt free basis. Although the boiling temperature and composition of the vapor of all the samples in the central part of the graph should not change, since they correspond to the SLLV region, the authors report differences of 0.4 K and 0.004 mole fraction, which can be considered very small. We plot our experimental data for these regions in the same figures. The results agree perfectly with those from Ref. [11], except for the point that represents the aqueous phase of the SLLV region, which has a mole fraction of 1-propanol that is lower than the previous result. Sampling was probably not performed adequately in Ref. [11], due to a failure to take into account that the liquid splits into two phases.



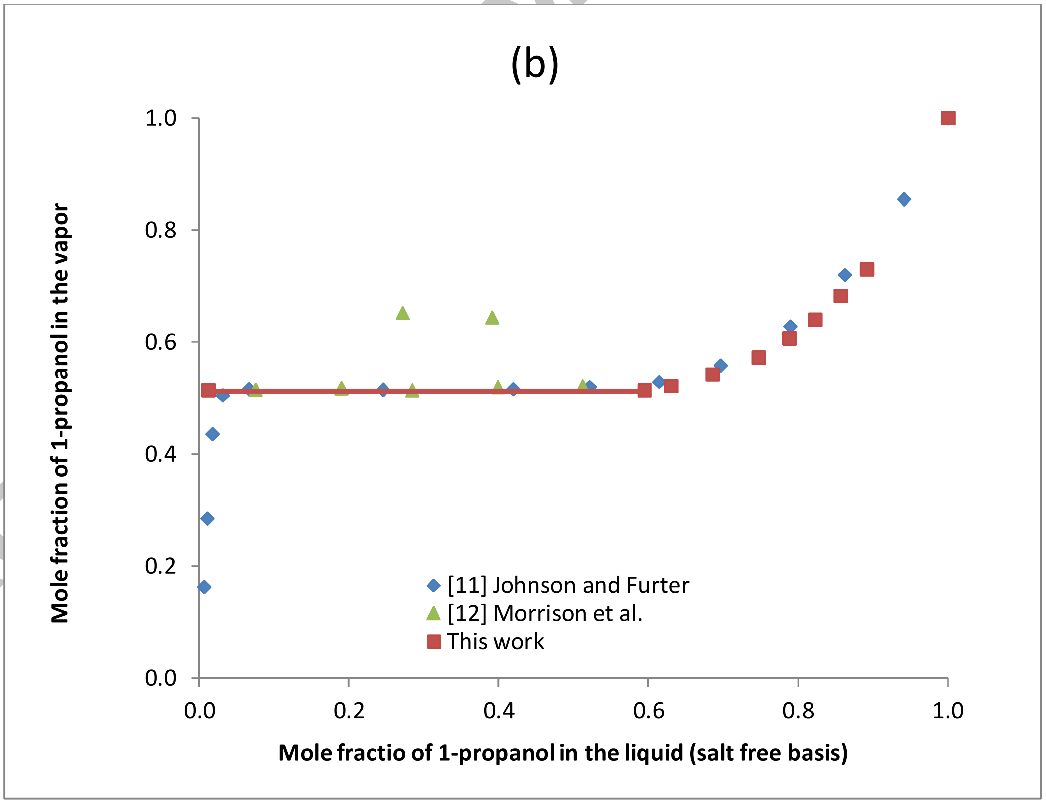


Figure 4. Boiling temperature (a) and composition of the vapor (b) for samples saturated in salt (SLV and SLLV).

The experimental data points obtained by Morrison et al. [12] are also plotted in Fig. 4. These authors determined VLE data for the ternary system, but neither took into account that many of the samples they analyzed belonged to the LLV region, nor that the results should obey certain relationships such as, for example, that the temperature or composition of the vapor phase should be equal for different samples belonging to the same tie line. Assessing their results for internal consistency, we find errors of 3 K in the temperature and 0.14 in the mole fraction for 1-propanol in the vapor phase, which are not acceptable.

Finally, Lin et al. [13] studied the same system by analyzing the LV and LLV regions while, at the very least, taking into account that the liquid samples can split into two liquid phases. Fig. 5 shows their experimental data for the LLV region and several points determined as LV but belonging to the LLV region. The boiling temperature of the points and the composition of the vapor should agree with those of the tie lines. However, as can be seen from Fig. 5, they are not consistent. The inconsistency between the temperatures that are determined by the authors of Ref. [13] are associated with errors of almost 2 K. For instance, the boiling temperature of one of their determined tie lines is 361.4 K ("boxed" numbers) while the temperature of one of their samples ("unboxed" numbers), analyzed as LV but belonging to the same tie line, is 363.3 K.

In addition, there must be an error in the given compositions of the aqueous phase in the LLV region, because the points that are specified for the LL solubility curve are far away from the real solubility curve. In our opinion, a mistake was made during the experimental determination of the concentrations, but not in the case of the temperatures since our experimental VLL data agree well with theirs, to within small discrepancies (< 0.1 K) between the two sets of experimental data.

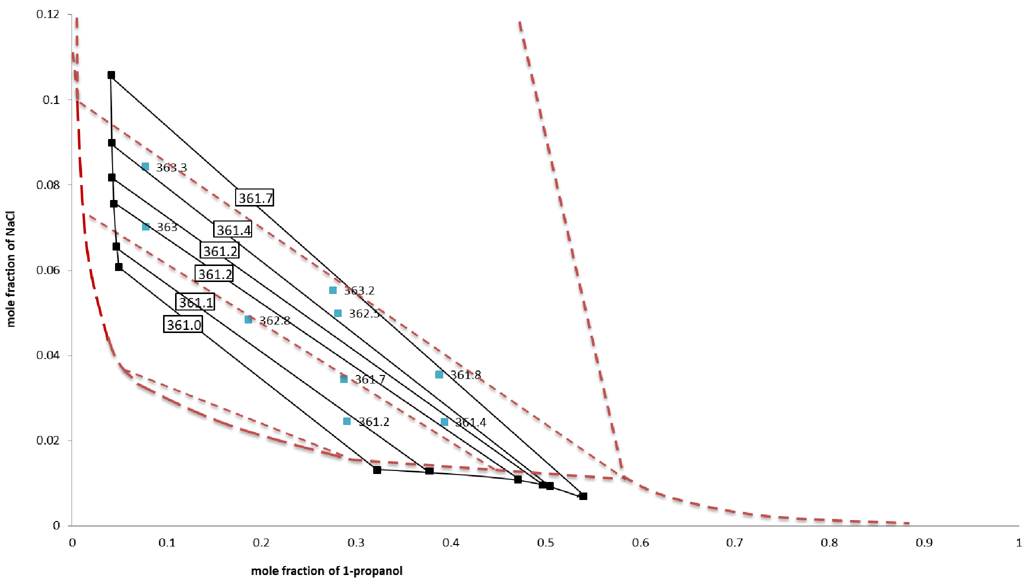


Figure 5. LLV Equilibrium region of water + NaCl + 1-propanol system at 101.3 kPa. Dashed lines = experimental data from this work. Continuous line = data from Ref. [13] The numbers in the diagram are the temperatures (K) for the LV points ("unboxed" numbers) and for the tie lines of LLV ("boxed") both determined in Ref. [13].

However, as these are the only published data available for this system in the literature, and in spite of the obvious errors in the existing experimental data, they had to be used in the correlations that were done to obtain the parameters in the thermodynamic models involving electrolytes. Therefore it is important to know the shape and characteristics of the phase diagrams of these systems to avoid such errors.

Phase diagram and evolution with temperature. By interpolating the experimental points in tables 2-5, we have constructed Figs. 6 and 7. Fig. 6 shows the boiling temperature isotherms of the ternary system obtained as a function of the liquid mole fraction. Only the lower part of the diagram, corresponding to a low concentration in salt, has been reproduced since the SLLVE region remains invariant with a constant boiling temperature and the isotherms of the SL regions are straight lines joining the point of the SL curve with the point representing the salt. In Fig. 7, the mole fraction of 1-propanol and salt in the liquid are represented by a simple grid, and the mole fraction of 1-propanol in the vapor and in equilibrium with the liquid, is shown as parametric curves. In systems containing electrolytes, unlike other ternary systems, two figures such as these completely define the system, since the vapor phase does not contain salt.

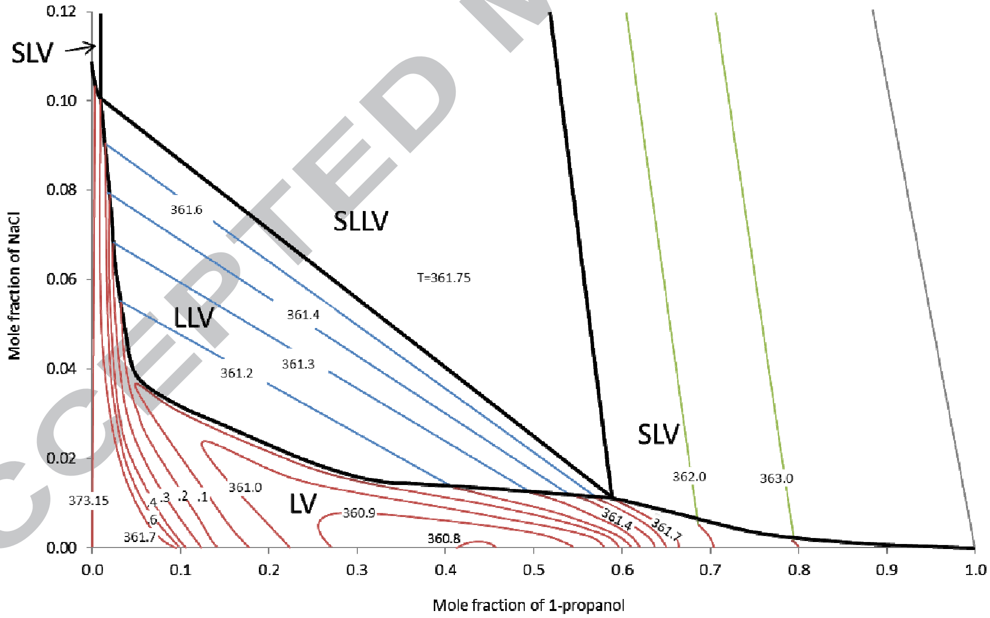


Figure 6. LLV and LV isotherms for the ternary system water + NaCl + 1-propanol at 101.3 kPa. Parameter of the curves = boiling temperature (K).

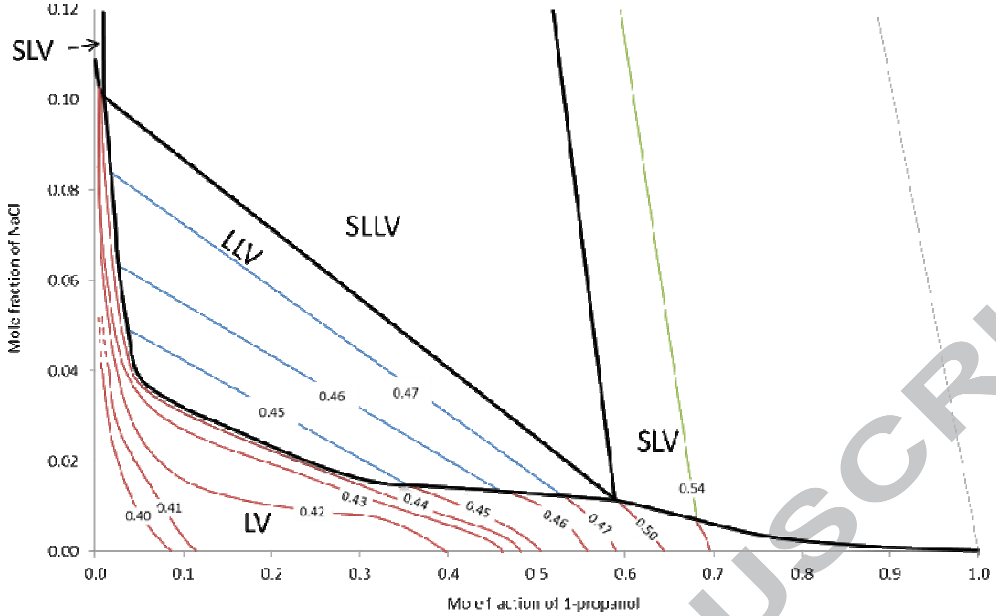


Figure 7. Curves of isoconcentration of 1-propanol in the vapor phase for the system water + NaCl + 1-propanol at 101.3 kPa. Parameter of the curves = mole fraction of 1-propanol in the vapor.

It can be seen that the boiling points of most of the mixtures in the system occur within a very narrow range of temperatures of only 1 K between the minimum boiling temperature of the binary azeotrope water+1-propanol (360.83 K) and the boiling temperature of the SLLV mixture (361.75 K). Notice that the isotherms and isoconcentration lines in the LLV region are straight lines that coincide with tie lines, a feature that was not taken into account in previous works on the same system. Notice also the shape of the curves in the LV region when they are close to the LLV region.

With these curves it is possible to analyze the evolution with temperature (increasing T) of the different equilibrium regions of the system. At temperature T < 360.78 K (the azeotropic point of the binary system water + 1propanol), five different regions co-exist in Fig. 8a: one LL, one SLL, two SL and one L. When the temperature reaches the binary azeotropic boiling point, a vapor point appears, and if the temperature increases slightly further, a new vapor-liquid region appears (Fig. 8b). At higher temperatures (Fig 8c), the size of the VL region increases until it touches the solubility LL curve of the system (Fig. 8d), and divides the L region into two separate ones, the LV region in two LV regions and a new LLV region appears.

At higher temperatures the two L regions shrink in size, the size of the VLL region and the two VL regions increases while the size of the LL region decreases until it disappears (Fig. 8e), and a new SLLV region is formed.

When the temperature increases further a new SV region appears (Fig. 8f), as well as two SLV regions on either side of it. With higher temperatures, the SV region grows and pushes the VLS, LV and LS regions to the sides while shrinking the L regions (Fig. 8g).

Finally, one of the liquid regions disappears (Fig. 8h) and the point is reached at which the 1-propanol that is saturated in salt, boils. At this point, only SV, SL, single L and VL regions remain since the boiling point of water is higher. The temperature then produces the same effect of increasing the SV region while shrinking that of the liquid until only a SLV point and a SV region are left, at which point the salt-saturated aqueous phase boils. At an even higher temperature only a SV region exists.

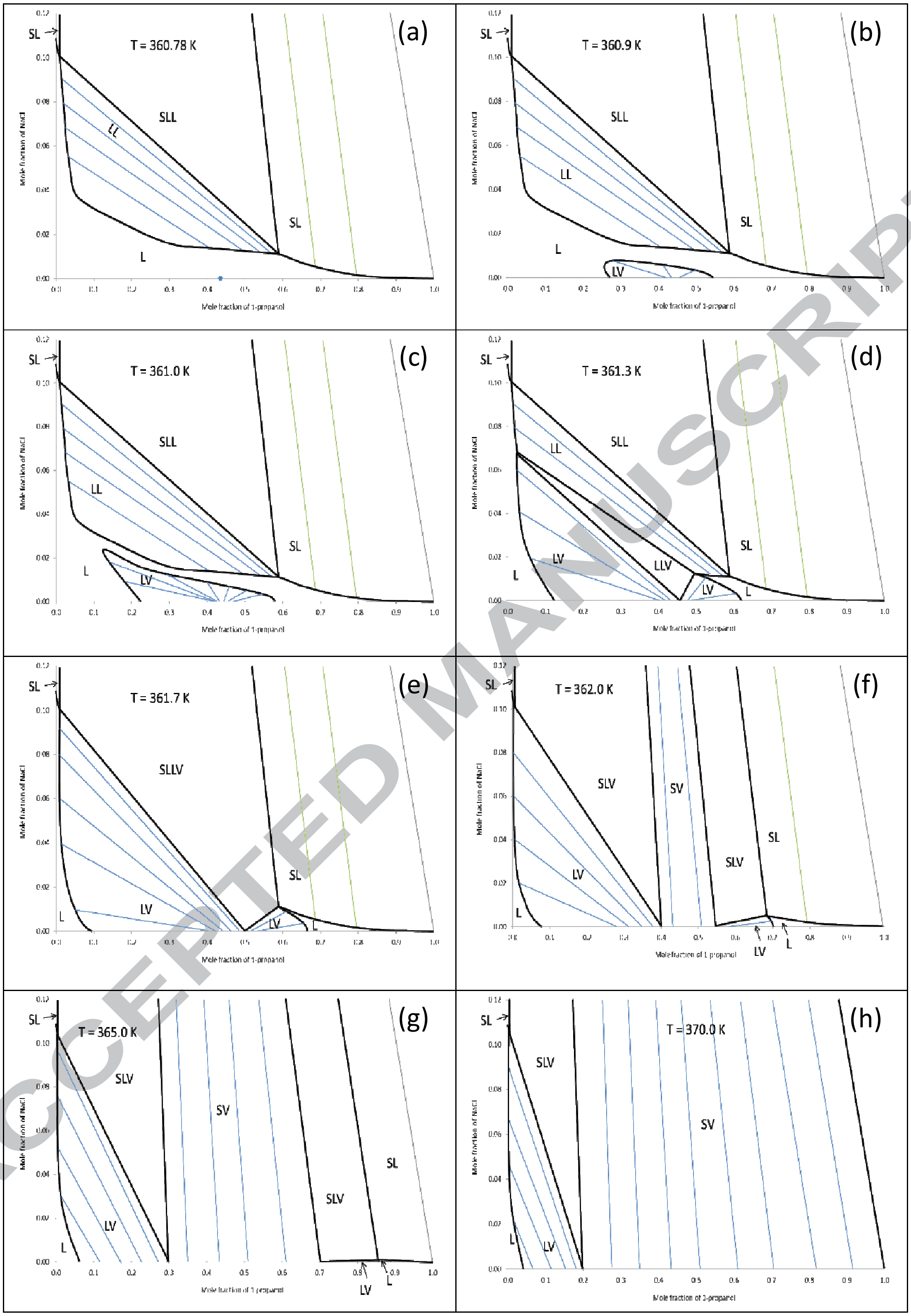


Figure 8 (a-h). Evolution with temperature (increasing T) of the different equilibrium regions of the system water + NaCl + 1-propanol (mole fraction) at 101.3 kPa.

Comparison with the equilibrium data calculated by the extended UNIQUAC model.

Finally, the SLLV equilibrium has been calculated by using the extended UNIQUAC model [3], which allows the determination of SLLVE of ternary systems containing inorganic salts. To do the calculations, the UNIQUAC parameters of water, 1-propanol and NaCl provided in Ref. [3] have been used. Fig. 9 shows the experimental lines separating different regions and several tie lines. It is true that the model correctly predicts the number and shape of the different regions but there are a number of discrepancies between experimental and calculated bubble temperatures and the compositions of the equilibrium phases. In particular, in the organic rich region the calculated salt concentrations are greater than the experimental ones.

It is important to recognize that the binary interaction parameters in the model have been calculated from previously published experimental data that are not accurate, as already pointed out in this paper. For this reason, there is need of reliable experimental data that can be used toward the development of new models or for obtaining new interaction parameters.

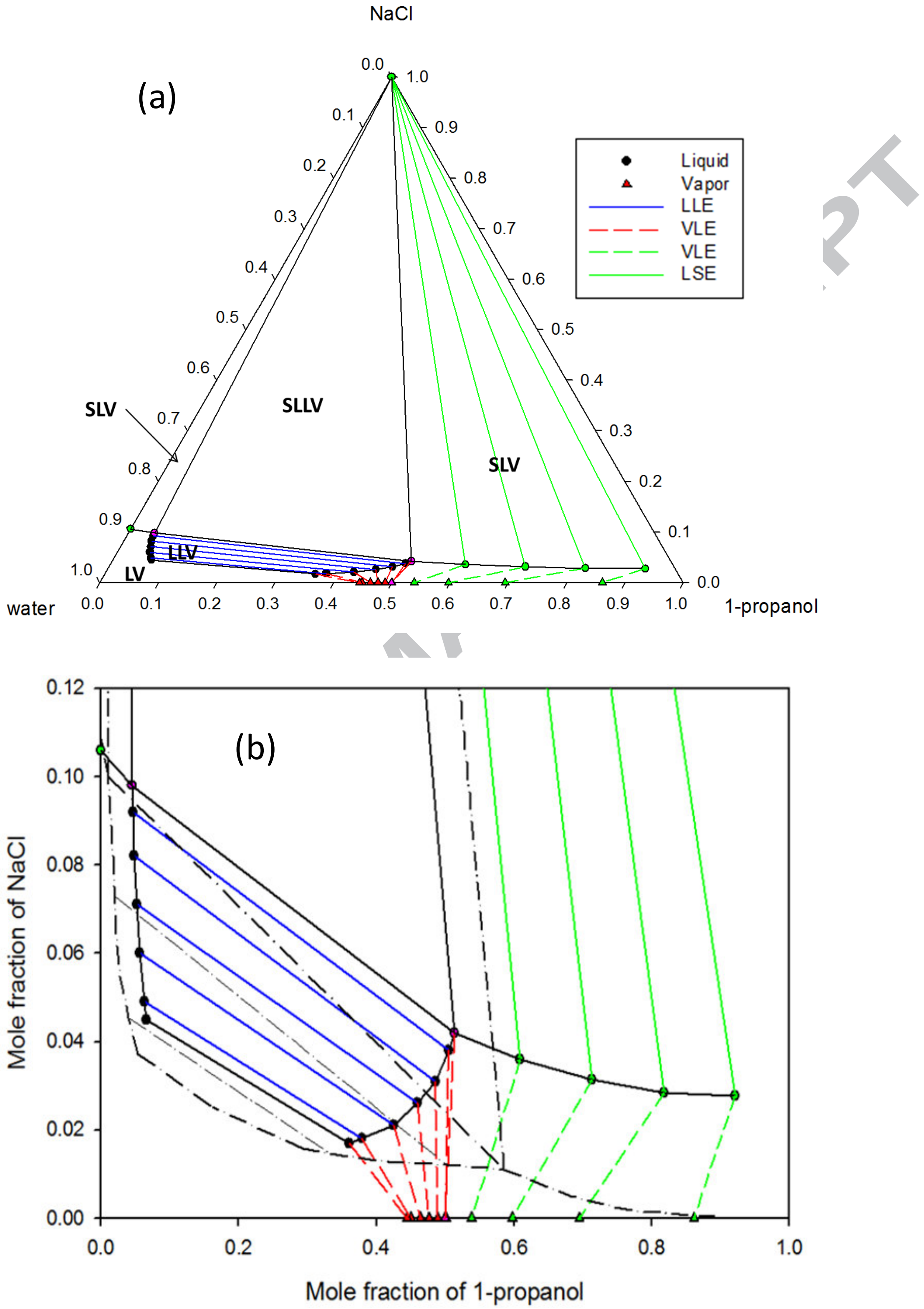


Figure 9. a) Equilibrium diagram (mole fraction) for the water + NaCl + 1-propanol system (mole fraction) at 101.3 kPa calculated by using the extended UNIQUAC model [3] b) Enlarged region. _ . _ . _ Model calculation; ______ Tendency line; _ _ _ _ VLE Tie-lines

4. Conclusions

The phase equilibria of the water + NaCl + 1-butanol mixture have been studied at 101.3 kPa in order to determine the influence of the salt on the vapor-liquid-liquid-solid equilibrium. It has been shown that the concentration of the salt changes the shape of the different equilibrium surfaces.

Previously published experimental data from the literature have been analyzed and compared with the data obtained in this work. It has been demonstrated that most of the previously published data was inconsistent and inaccurate. It is really important to have reliable experimental data that is suitable for the development of new thermodynamic models or obtaining new binary interaction parameters.

A quantitative analysis of the results shows that with an increase in temperature, the LL region, the SLL and SL regions, present at temperatures below 360.78K, lead to other regions in which vapor appears (VL, VS, VLL, VLS and VLLS).

Finally, it has been shown that the extended UNIQUAC model by Thomsen et al. [3] is suitable for predicting the number and shape of the different equilibrium regions. However, there are several discrepancies in temperatures and composition, especially where the organic rich phase is concerned.

Acknowledgment

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TABLES

Table 1. Specifications of chemical compounds.

Chemical	Provider	Initial purity (mass %)	Water content KF[a] (mass %)	Purification method	Analysis method
NaCl	VWR	> 99.9		none	
1-Propanol	Merck	> 99.5	0.06	none	GC [b]
Ethanol	VWR	> 99.8	0.02	none	GC [b]

[a] KF = Karl Fischer Technique

[b] GC = Gas chromatography

Table 2. Equilibrium data (mole fraction) corresponding to the three phase region (1S - 1L$_{org}$ - 1 V) of the water + NaCl + 1-propanol system at 101.3 kPa. Solid composition is pure NaCl.

	Liquid			Vapor	
T/K	Water[1,3]	NaCl[2]	1-Propanol[1]	Water[1]	1-Propanol[1]
361.83	0.367	0.0087	0.624	0.478	0.522
362.12	0.313	0.0049	0.682	0.458	0.542
362.76	0.252	0.0026	0.745	0.428	0.572
363.40	0.212	0.0014	0.787	0.393	0.607
364.07	0.178	0.0011	0.821	0.360	0.640
364.94	0.143	0.00064	0.856	0.317	0.683
365.92	0.108	0.00038	0.891	0.270	0.730

T is in K with a standard uncertainty of 0.06 K, the pressure standard uncertainty is 0.1 kPa and composition relative standard uncertainty is $u_r = \frac{u}{w}$ is 2% except for the NaCl where the relative standard uncertainty is 5%.

[1] obtained by GC-TCD

[2] obtained by gravimetric analysis

[3] checked against Karl Fischer Technique

Table 3. Equilibrium data (mole fraction) corresponding to the three phase region (1 L_{aq} - 1L_{org} - 1 V) of the water + NaCl + 1-propanol system at 101.3 kPa.

	Aqueous Liquid			Organic Liquid			Vapor	
T/K	Water[1]	NaCl[2]	1-Propanol[1]	Water[1,3]	NaCl[2]	1-Propanol[1]	Water[1]	1-Propanol[1]
361.07	0.909	0.0372	0.0534	0.691	0.0157	0.293	0.551	0.449
361.11	0.914	0.0454	0.0403	0.652	0.0145	0.333	0.552	0.448
361.17	0.917	0.0553	0.0276	0.570	0.0127	0.417	0.550	0.450
361.20	0.915	0.0618	0.0230	0.537	0.0125	0.451	0.545	0.455
361.35	0.907	0.0730	0.0201	0.486	0.0120	0.502	0.535	0.465
361.52	0.897	0.0873	0.0159	0.425	0.0113	0.563	0.526	0.474

T is in K with a standard uncertainty of 0.06 K, the pressure standard uncertainty is 0.1 kPa and composition relative standard uncertainty is $u_r = \frac{u}{w}$ is 2% except for the NaCl in the organic phase where the relative standard uncertainty is 5%.

[1] obtained by GC-TCD

[2] obtained by gravimetric analysis

[3] checked against Karl Fischer Technique

Table 4. Equilibrium data (mole fraction) corresponding to the four phase region (1S - 1 L_{aq} - 1 L_{org} - 1 V) of the water + NaCl +1-propanol system at 101.3 kPa.

Phase	Water[1,3]	NaCl[2]	1-Propanol[1]
Solid	0	1	0
Organic Liquid	0.403	0.0109	0.586
Aqueous Liquid	0.889	0.100	0.0105
Vapor	0.505	0	0.495
Temperature (K)	361.75		

T is in K with a standard uncertainty of 0.06 K, the pressure standard uncertainty is 0.1 kPa and composition relative standard uncertainty is $u_r = \frac{u}{w}$ is 2% except for the NaCl in the organic phase where the relative standard uncertainty is 5%.

[1] obtained by GC-TCD

[2] obtained by gravimetric analysis

[3] checked against Karl Fischer Technique

Table 5. Equilibrium data (mole fraction) corresponding to the liquid-vapor region at 101.3 kPa.

	Liquid			Vapor	
T(K)	Water[1,3]	NaCl[2]	1-Propanol[1]	Water[1]	1-Propanol[1]
360.84	0.478	--[4]	0.522	0.542	0.458
361.19	0.475	0.0080	0.517	0.545	0.455
360.85	0.695	--[4]	0.305	0.584	0.416
360.92	0.694	0.0066	0.300	0.584	0.416
360.99	0.681	0.0123	0.307	0.563	0.437
360.97	0.779	--[4]	0.221	0.586	0.414
361.00	0.776	0.0088	0.215	0.579	0.421
361.02	0.763	0.0173	0.219	0.573	0.427
361.20	0.863	--[4]	0.137	0.587	0.413
361.20	0.854	0.0075	0.139	0.601	0.399
361.10	0.852	0.0148	0.134	0.576	0.424
361.97	0.934	--[4]	0.066	0.604	0.396
361.67	0.928	0.0075	0.065	0.597	0.403
361.38	0.923	0.0148	0.062	0.595	0.405
361.19	0.917	0.0224	0.061	0.577	0.423

T is in K with a standard uncertainty of 0.06 K, the pressure standard uncertainty is 0.1 kPa and composition relative standard uncertainty is $u_r = \frac{u}{w}$ is 2% except for the NaCl in the liquids where the relative standard uncertainty is 5%.

[1] obtained by GC-TCD

[2] obtained by gravimetric analysis

[3] checked against Karl Fischer Technique

[4] binary water + 1-propanol points