Liquid-Liquid Equilibrium of Quaternary Systems: Experimental Data and NRTL Model Predictions for Water, Lactic Acid, 1-Octanol, and Salts

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This study investigates the liquid–liquid equilibrium (LLE) behavior of ternary and quaternary systems composed of water, lactic acid, 1-octanol, and salts (NaCl and KCl). Phase behaviour was predicted using the non-random two-liquid (NRTL) model. Accurate thermodynamic modeling is essential for optimizing separation processes in the pharmaceuticals, food, and bio-based chemical industries. Experimental tie-line data were obtained at salt mass fractions from 0 to 15 % and validated with Othmer–Tobias and Hand correlations, which confirmed the high consistency of the data. The NRTL predictions were evaluated using the root mean square deviation (RMSD), yielding low values (0.1418 for 15 % NaCl, 0.1088 for 15 % KCl), demonstrating strong predictive reliability. The salting-out effect was quantified through binary interaction parameters, such as 20.4174 for water–1-octanol (15 % KCl) and 15.5637 for the same pair (15 % NaCl), indicating enhanced lactic acid partitioning into the organic phase. Overall, the results confirm the accuracy of the NRTL model in describing ionic interactions and phase separation, offering a reliable framework for efficient extraction and purification processes.

Keywords
liquid-liquid equilibrium, NRTL model, lactic acid, 1-octanol, salting-out effects

Introduction

Liquid-liquid equilibrium (LLE) plays a fundamental concept in chemical and process engineering, particularly for designing and optimizing separation processes across industries such as pharmaceuticals, food production, and bio-based chemicals. Systems containing water, organic solvents, and salts are especially important because of their broad industrial relevance[1]. The addition of inorganic salts such as NaCl and KCl can significantly modify phase behavior by enhancing phase separation, altering solubility, and modifying the ionic strength of the system. These changes directly influence the distribution of components between phases[1]. Thermodynamic models such as NRTL, UNIQUAC, and UNIFAC are widely used to predict phase behaviour and calculate activity coefficients[1–3]. Their accuracy is typically assessed through root-mean-square deviation (RMSD), which indicates the reliability of LLE predictions[4,5].

Substantial progress has been made in understanding solvent–solute and ionic interactions in ternary and quaternary systems. Early investigations focused on solvent-solute interactions, while more recent studies have incorporated effects of salts. The salting-out phenomenon, where salts such as NaCl and KCl reduce the solubility of organic solutes in the aqueous phase, is well-documented. This behavior enhances solute partitioning into the organic phase and is relevant to separation processes, as seen in systems such as potassium nitrate–water–butyric acid[6]. The NRTL model has been shown to reliably predict phase behavior in several electrolyte–water systems, including potassium chloride-water mixtures[7].

However, research on quaternary systems that include lactic acid remains limited. Previous studies

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on systems such as $NaCl–MgCl_2–SrCl_2–H_2O$ and $KCl–MgCl_2–SrCl_2–H_2O$ have demonstrated complex phase equilibria, which have been successfully described using the Pitzer model[8]. Nevertheless, challenges persist in modelling systems with complex ionic interactions, as highlighted by work on the $KCl–KBr–NaCl–NaBr–H_2O$ quinary system, where improved models are needed for accurate predictions[9]. The incorporation of lactic acid into these systems requires further experimental validation to enhance their industrial applicability.

The salting-out effects of NaCl and KCl are known to improve liquid-liquid extraction (LLE) by reducing solute solubility and promoting phase separation[10,11]. While these effects have been widely studied in ternary systems, their combined influence in quaternary systems remains insufficiently explored[12]. These salts can improve partition coefficients and solute recovery, which is beneficial for applications in biofuel extraction, bioprocessing, and environmental analysis[13,14]. However, excessive salt concentrations may cause stability issues, necessitating optimized conditions[15]. Further research is therefore required to clarify their synergistic roles[16].

Despite the established salting-out phenomenon enhancing separation in other systems, the phase behavior of lactic acid systems with varying salt concentrations remains insufficiently explored[17,18]. Although thermodynamic models such as NRTL and UNIQUAC are often successful in predicting phase equilibria[19], experimental validation for lactic acid is limited. Modified models for alcohol-salt systems may offer insight[20], but the lack of data on salt-lactic acid interactions continues to hinder progress[21,22]. Systematic studies are therefore essential for validating these models and optimizing industrial separation processes.

Correlations such as the Othmer–Tobias and Hand equations are commonly used to assess the consistency of experimental tie-line data[23]. These correlations have proven effective in systems containing ionic liquids and salts, allowing identification of phase-forming tendencies[24]. Ionic interactions are influenced by salt concentration and composition, which can alter charge distribution and affect transport properties relevant to electrochemical applications[25]. In ternary and quaternary systems, salting-out effect are closely linked to hydrogen bonding and the hydration structure of salts[23]. Continued research is necessary to refine thermodynamic models and improve our understanding of phase behaviour in systems featuring complex ionic interactions.

This study investigates the liquid-liquid equilibrium (LLE) behavior of quaternary systems composed of water, lactic acid, 1-octanol, and salts (NaCl and KCl), with particular emphasis on the influence of salts on phase separation and lactic acid distribution between phases. Experimental tie-line data were generated and validated using the Othmer–Tobias and Hand correlations to ensure consistency. These data were then used to refine and validate the NRTL model for systems exhibiting significant ionic interactions. Lactic acid was used as a model solute to quantify the salting-out effects of NaCl and KCl, demonstrating improved partitioning into the organic phase. Notably, the NRTL model yielded high predictive accuracy, with low RMSD values such as 0.1418 for 15 % NaCl and 0.1088 for 15 % KCl. The binary interaction parameters such as 20.4174 for water-1-octanol at 15 % KCl, highlighted the stronger salting-out effect of KCl compared to NaCl. These findings confirm the reliability of the NRTL model in describing phase behaviour and support its use in designing efficient and sustainable separation processes.

Experimental

Chemicals

The chemicals used in this study are listed in Table 1. Lactic acid was selected as the solute of interest due to its relevance in phase equilibrium studies, while 1-octanol served as the organic solvent, facilitating the partitioning of lactic acid between the aqueous and organic phases. Sodium chloride and potassium chloride were incorporated to induce the salting-out effect, which influences

Table 1 – *List of chemicals used, their purity, and supplier information*

Chemical	Supplier	CAS RN	M (g mol^{-1})	Purity
L-(+)-Lactic acid	Biochem chemopharma	79-33-4	90.08	85–90 %
1-Octanol	Sigma-Aldrich	111-87-5	130.23	99.0 %
Sodium chloride	Merck	7647-14-5	58.44	99.5 %
Potassium chloride	Merck	7447-40-7	74.55	99.5 %
Phenolphthalein	Sigma-Aldrich	77-09-8	318.32	98.0 %

the solubility and distribution of lactic acid between the two phases. Phenolphthalein was employed as a pH indicator to precisely monitor the endpoint during titration experiments for lactic acid quantification. All solutions were prepared using deionized water with a conductivity below 1 μS cm^{-1} to ensure high purity and reproducibility. The salts were thoroughly dried in a vacuum oven at 105 °C for 24 hours to eliminate any absorbed moisture before use.

Preparation of ternary and quaternary mixtures

The systems investigated included the ternary system (water + lactic acid + 1-octanol) and the quaternary systems (water + lactic acid + 1-octanol + NaCl/KCl). For the quaternary systems, the salt concentrations in the total mixture were varied at mass fractions of 0 %, 5 %, 10 %, and 15 %. Precise quantities of each component were weighed using a calibrated analytical balance (Sartorius BP221S, accuracy ±0.0001 g). Each mixture was prepared in a 250 mL Erlenmeyer flask to facilitate mixing and observation of phase formation.

Apparatus and experimental procedure

Apparatus

The liquid-liquid equilibrium (LLE) studies were conducted using a custom-designed apparatus to ensure precise phase behavior determination, consistent with methodologies reported prior research[26,27]. The setup included a 500 mL jacketed glass vessel for thermal stability, a mechanical stirrer to ensure uniform phase mixing, and a thermostatic water bath (Julabo F12-ED) to maintain a constant temperature of 20.0 ± 0.1 °C [26]. A Teflon-sealed lid prevented solvent evaporation and included ports for sampling, while temperature was monitored in real time using a digital thermometer with an accuracy of ±0.05 °C [28]. This setup enabled precise determination of tie-line data and solubility diagrams, offering valuable insights into LLE behavior relevant to applications such as biodiesel purification and solvent-assisted extraction[26,28].

Experimental procedure

Mixing and equilibration

Each prepared mixture was transferred to the glass vessel, sealed, and stirred at 500 rpm for 2 hours to ensure phase equilibrium. The stirring speed was optimized to avoid emulsification while maintaining efficient mass transfer between the aqueous and organic phases. The system was allowed to equilibrate at 20 °C, the temperature chosen for its relevance to practical solvent extraction processes.

Phase separation

After stirring, the system was allowed to stand undisturbed for a minimum of 12 hours[29] to ensure complete phase separation. Preliminary tests confirmed that this separation time was sufficient to produce distinct aqueous and organic phases. A 125 mL graduated separating funnel was then used to decant the two phases carefully, avoiding cross-contamination.

Composition analysis

The lactic acid concentration in the aqueous phase was determined by acid-base titration using 0.1 N sodium hydroxide (NaOH) solution. Phenolphthalein served as the endpoint indicator, with a distinct color transition from colorless to faint pink marking the endpoint. Lactic acid in the organic phase was quantified by back-extraction into water followed by titration with 0.1 N NaOH using phenolphthalein as the indicator. Each sample was analyzed in triplicate, and the mean values are reported. The salt content in each phase was measured by evaporating the aqueous phase at 105 °C in a vacuum oven and weighing the residue[30].

Determination of tie-line data

The determination of tie-line data and solubility diagrams is essential for analyzing liquid-liquid equilibria. The mass balance method, validated by Othmer–Tobias and Hand equations, ensures reliable tie-line data with high R^2 values[23,31]. The cloud point method visually constructs solubility diagrams by observing turbidity, effectively demonstrating phase behaviors in salt systems[32,33]. These studies underscore the influence of temperature, molecular weight, and salting-out effects on phase separation, providing valuable insights into equilibrium processes.

The separation factor (S) is the ratio of the distribution coefficients of lactic acid to water, and it is expressed as:

$$S = \frac{K_{d2}}{K_{d1}} \tag{1}$$

where, K_{d2} and K_{d1} are the distribution coefficients of water and lactic acid, respectively. The distribution coefficient (K_d) for each component is calculated as the ratio of the mass fraction of the component in the solvent-rich phase to its mass fraction in the water-rich phase, as given by the following equation:

$$K_d = \frac{x_{3i}}{x_{1i}} \tag{2}$$

Othmer–Tobias and Hand correlations

The reliability of the experimental tie-line data was verified by applying the Othmer-Tobias and Hand correlations. Linear regression analysis was used to calculate the correlation coefficients, with values close to unity indicating the accuracy of the experimental data.

Modeling of phase equilibria

The binary interaction parameters for the non-random two-liquid (NRTL) model were estimated using the experimental data. The parameters were optimized using the Particle Swarm Optimization (PSO) algorithm implemented in MATLAB (version 2016b). The model's accuracy was assessed by comparing the experimental tie-line data with the predicted values, and the root mean square error (RMSD) was used as a metric of predictive accuracy.

Results and discussion

LLE experimental results

Fig. 1 illustrates the influence of NaCl concentration on the phase behavior of the water + lactic acid + 1-octanol system. As shown in Table 2, increasing the NaCl content from 5 % to 15 % produced a clear and systematic expansion of the biphasic region, confirming a pronounced salting-out effect. The tabulated solubility compositions reveal a marked decrease in the water content of the aqueous phase at comparable lactic acid loadings. For instance, at $x_2 \approx 0.35–0.45$, the aqueous-phase water fraction decreased from $x_1 = 0.6581$ at 5 % NaCl to $x_1 = 0.5840$ at 10 %, and further to $x_1 = 0.5512$ at 15 % NaCl, while the corresponding organic-phase compositions became progressively richer in 1-octanol. This systematic displacement of tie-line endpoints and binodal points demonstrates that NaCl

Table 2 – *Experimental solubility curve data for water + 1-octanol + lactic acid + NaCl*

Water (1)-lactic acid (2)-1-octanol (3)											
0 % NaCl			5 % NaCl			10 % NaCl			15 % NaCl		
x_1	x_2	x_3	x_1	x_2	x_3	x_1	x_2	x_3	x_1	x_2	x_3
0.9579	0.0268	0.0153	0.9617	0.0163	0.0220	0.9462	0.0189	0.0349	0.9506	0.0219	0.0276
0.9416	0.0471	0.0113	0.9313	0.0483	0.0204	0.9217	0.0460	0.0322	0.9260	0.0490	0.0250
0.8945	0.0921	0.0134	0.8963	0.0876	0.0161	0.8212	0.1665	0.0123	0.8235	0.1617	0.0148
0.8197	0.1639	0.0164	0.8289	0.1636	0.0074	0.7647	0.2292	0.0061	0.7023	0.2830	0.0147
0.7635	0.2288	0.0076	0.7597	0.2275	0.0129	0.7101	0.2828	0.0071	0.6581	0.3261	0.0157
0.7086	0.2836	0.0078	0.7083	0.2804	0.0113	0.6562	0.3346	0.0092	0.6203	0.3711	0.0087
0.6647	0.3327	0.0027	0.6562	0.3274	0.0164	0.6237	0.3703	0.0060	0.5840	0.4084	0.0076
0.6201	0.3719	0.0080	0.6147	0.3718	0.0135	0.5840	0.4107	0.0053	0.5447	0.4412	0.0142
0.5843	0.4082	0.0076	0.5825	0.4059	0.0116	0.5512	0.4405	0.0083	0.5131	0.4649	0.0220
0.5512	0.4400	0.0088	0.5532	0.4413	0.0055	0.5232	0.4700	0.0068	0.5028	0.4752	0.0221
0.5238	0.4720	0.0042	0.5233	0.4726	0.0042	0.5110	0.4834	0.0056	0.1035	0.4359	0.4607
0.5084	0.4840	0.0076	0.5061	0.4788	0.0152	0.0988	0.4372	0.4640	0.1034	0.4238	0.4729
0.0952	0.4508	0.4540	0.0875	0.4436	0.4689	0.0868	0.4059	0.5074	0.0900	0.4064	0.5035
0.0950	0.4297	0.4753	0.0922	0.4303	0.4776	0.0724	0.3463	0.5814	0.0913	0.3746	0.5342
0.0837	0.4079	0.5084	0.0829	0.4069	0.5102	0.0635	0.3132	0.6233	0.0944	0.3392	0.5664
0.0753	0.3491	0.5756	0.0818	0.3792	0.5390	0.0553	0.2702	0.6745	0.0801	0.3087	0.6113
0.0631	0.3098	0.6271	0.0724	0.3482	0.5794	0.0376	0.2214	0.7410	0.0626	0.2153	0.7221
0.0629	0.2676	0.6696	0.0700	0.3086	0.6214	0.0306	0.1626	0.8068	0.0588	0.1575	0.7837
0.0524	0.2189	0.7287	0.0683	0.2679	0.6638	0.0350	0.0900	0.8750	0.0443	0.0859	0.8698
0.0359	0.0151	0.9490	0.0438	0.1578	0.7984	0.0278	0.0435	0.9287	0.0322	0.0442	0.9236
–	–	–	0.0351	0.0876	0.8773	0.0304	0.0199	0.9497	–	–	–
–	–	–	0.0267	0.0507	0.9226	–	–	–	–	–	–

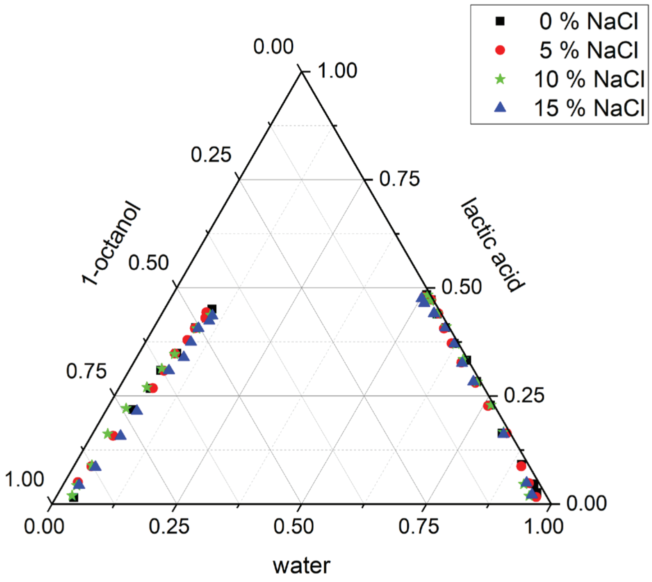

Fig. 1 – *Comparison of solubility isotherms for the quaternary system (water + lactic acid + 1-octanol + NaCl) at 5 %, 10 %, and 15 % of salt*

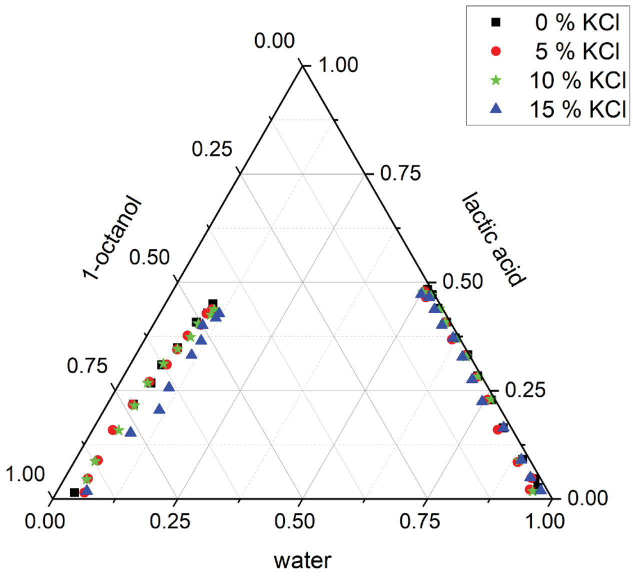

Fig. 2 – *Comparison of solubility isotherms for the quaternary system (water + lactic acid + 1-octanol + KCl) at 5 %, 10 %, and 15 % of salt*

Table 3 – *Experimental solubility curve data for water + 1-octanol + lactic acid + KCl*

Water (1)-lactic acid (2)-1-octanol (3)											
0 % KCl			5 % KCl			10 % KCl			15 % KCl		
x_1	x_2	x_3	x_1	x_2	x_3	x_1	x_2	x_3	x_1	x_2	x_3
0.9579	0.0268	0.0153	0.9443	0.0217	0.0340	0.9515	0.0181	0.0304	0.9672	0.0193	0.0135
0.9416	0.0471	0.0113	0.9337	0.0486	0.0177	0.8900	0.0887	0.0213	0.9312	0.0493	0.0195
0.8945	0.0921	0.0134	0.8877	0.0849	0.0274	0.7620	0.2297	0.0084	0.8924	0.0907	0.0169
0.8197	0.1639	0.0164	0.8111	0.1598	0.0291	0.7093	0.2829	0.0078	0.8200	0.1645	0.0156
0.7635	0.2288	0.0076	0.7559	0.2290	0.0151	0.6631	0.3289	0.0079	0.7470	0.2246	0.0284
0.7086	0.2836	0.0078	0.7066	0.2814	0.0120	0.6234	0.3710	0.0056	0.7013	0.2757	0.0230
0.6647	0.3327	0.0027	0.6607	0.3314	0.0079	0.5817	0.4078	0.0105	0.6560	0.3277	0.0164
0.6201	0.3719	0.0080	0.6151	0.3683	0.0166	0.5546	0.4393	0.0061	0.6154	0.3717	0.0129
0.5843	0.4082	0.0076	0.5826	0.4081	0.0093	0.5222	0.4705	0.0073	0.5780	0.4013	0.0207
0.5512	0.4400	0.0088	0.5141	0.4653	0.0206	0.5025	0.4769	0.0206	0.5453	0.4378	0.0169
0.5238	0.4720	0.0042	0.5043	0.4796	0.0161	0.1045	0.4370	0.4585	0.5205	0.4655	0.0140
0.5084	0.4840	0.0076	0.1000	0.4378	0.4622	0.1041	0.4249	0.4710	0.5010	0.4726	0.0265
0.0952	0.4508	0.4540	0.0939	0.4282	0.4778	0.0894	0.4043	0.5063	0.1187	0.4284	0.4528
0.0950	0.4297	0.4753	0.0954	0.4021	0.5025	0.0888	0.3738	0.5374	0.1175	0.4178	0.4647
0.0837	0.4079	0.5084	0.0815	0.3770	0.5415	0.0767	0.3458	0.5775	0.0995	0.4008	0.4998
0.0753	0.3491	0.5756	0.0762	0.3451	0.5788	0.0653	0.3120	0.6228	0.1145	0.3647	0.5208
0.0631	0.3098	0.6271	0.0733	0.3099	0.6168	0.0560	0.2677	0.6763	0.1116	0.3320	0.5564
0.0629	0.2676	0.6696	0.0584	0.2705	0.6711	0.0559	0.2150	0.7291	0.1042	0.2569	0.6390
0.0524	0.2189	0.7287	0.0510	0.2179	0.7311	0.0528	0.1585	0.7886	0.1107	0.2056	0.6837
0.0359	0.0151	0.9490	0.0408	0.1591	0.8002	0.0410	0.0872	0.8718	0.0795	0.1529	0.7676
–	–	–	0.0458	0.0890	0.8652	0.0454	0.0454	0.9093	0.0590	0.0184	0.9226
–	–	–	0.0470	0.0470	0.9060	–	–	–	–	–	–
–	–	–	0.0556	0.0148	0.9296	–	–	–	–	–	–

reduced the mutual solubility of the phases and enlarged the immiscibility gap.

This effect arose because Na^+ and Cl^- ions strongly hydrate in water, reducing the number of free water molecules available to solvate lactic acid[34]. As hydration competition intensified, lactic acid was expelled from the aqueous phase and preferentially transferred to the 1-octanol phase, increasing the degree of phase separation. The progressive widening of the immiscibility region confirms that NaCl enhanced the extraction capability of 1-octanol by promoting the migration of lactic acid into the organic layer. This behavior is fully consistent with the classical Setschenow-type electrolyte effect, where increasing salt concentration lowers solute solubility in the aqueous phase[35].

Fig. 2 presents the effect of KCl on the solubility behavior of the water + lactic acid + 1-octanol system, and the results reported in Table 3 show that increasing the KCl concentration from 5 % to 15 % produced an even more pronounced expansion of the biphasic region compared to NaCl.

This enhanced salting-out efficiency of KCl is attributed to differences in ionic hydration: K^+ ions are less strongly hydrated than Na^+, allowing them to disrupt the water structure more effectively and reduce the solvation capacity of the aqueous phase for lactic acid[34]. As water molecules preferentially hydrated K^+ and Cl^- ions, fewer solvent molecules remained available to stabilize lactic acid, forcing it into the 1-octanol phase. This behavior led to a more pronounced outward shift of the binodal curve with increasing KCl concentration. The widening of the immiscibility region confirms that KCl substantially enhanced the extraction capability of 1-octanol, consistent with classical Setschenow-type electrolyte behavior, where solute solubility in water decreases systematically with salt concentration[36].

The broader two-phase zone indicates that KCl is a more efficient promoter of phase separation than NaCl, making it potentially more advantageous for enhancing lactic acid extraction in this system. This difference is consistent with the Hofmeister series, where potassium salts typically exert greater salting-out power than sodium salts due to weaker ion–water interactions and a higher ability to compete for hydration. The combined results demonstrate that controlling salt type and concentration is an effective strategy for tuning the phase behavior and improving extraction performance in lactic-acid–octanol system[37].

Tie-line data and phase separation

Table 4 presents the experimental tie-line data for the quaternary water–lactic acid–1-octanol system at different salt concentrations (0–15 % NaCl

Table 4 – *Experimental tie-line results in mass fraction for water-lactic acid-1-octanol*

Water-rich phase			Solvent-rich phase		
x_1	x_2	x_3	x_1	x_2	x_3
0 % NaCl					
0.96	0.01	0.03	0.07	0.35	0.58
0.89	0.01	0.10	0.07	0.40	0.53
0.79	0.20	0.01	0.08	0.40	0.52
0.66	0.33	0.01	0.09	0.41	0.50
0.54	0.45	0.01	0.10	0.40	0.50
5 % NaCl					
0.95	0.032	0.018	0.039	0.271	0.69
0.87	0.118	0.012	0.079	0.451	0.47
0.76	0.229	0.011	0.087	0.453	0.46
0.63	0.355	0.015	0.091	0.459	0.45
0.55	0.445	0.005	0.097	0.463	0.44
10 % NaCl					
0.9	0.096	0.004	0.1	0.43	0.47
0.82	0.168	0.012	0.12	0.45	0.43
0.75	0.235	0.015	0.13	0.45	0.42
0.64	0.349	0.011	0.26	0.47	0.27
0.54	0.45	0.01	0.28	0.46	0.26
15 % NaCl					
0.94	0.046	0.014	0.065	0.115	0.82
0.81	0.173	0.017	0.087	0.393	0.52
0.73	0.252	0.018	0.1	0.41	0.49
0.62	0.361	0.019	0.27	0.44	0.29
0.55	0.429	0.021	0.28	0.45	0.27
5 % KCl					
0.9000	0.0730	0.0270	0.0450	0.0350	0.9200
0.8100	0.1710	0.0190	0.0500	0.1200	0.8300
0.7200	0.2650	0.0150	0.0600	0.2700	0.6700
0.6200	0.3640	0.0160	0.0660	0.3440	0.5900
0.5600	0.4220	0.0180	0.0800	0.3500	0.5700
10 % KCl					
0.9100	0.0660	0.0240	0.0530	0.1870	0.7600
0.8300	0.1560	0.0140	0.0600	0.2600	0.6800
0.7400	0.2510	0.0090	0.0640	0.3160	0.6200
0.6200	0.3750	0.0050	0.0850	0.3950	0.5200
0.5300	0.4670	0.0030	0.0970	0.4330	0.4700
15 % KCl					
0.9400	0.0460	0.0140	0.0650	0.1150	0.8200
0.8100	0.1730	0.0170	0.0870	0.3930	0.5200
0.7300	0.2520	0.0180	0.1000	0.4100	0.4900
0.6200	0.3610	0.0190	0.2700	0.4400	0.2900
0.5500	0.4290	0.0210	0.2800	0.4500	0.2700

and 5–15 % KCl). The table reports the equilibrium compositions of both the water-rich phase and the solvent-rich phase, expressed as mass fractions of water (x_1), lactic acid (x_2), and 1-octanol (x_3).

Distribution coefficient and separation factor

Fig. 3 illustrates how the separation factor changed with varying concentrations of NaCl (0 %, 5 %, 10 %, and 15 %). The separation factor, defined as the ratio of the distribution coefficients of lactic acid and water, provides insight into the efficiency of the extraction process. As shown in Table 5, increasing NaCl concentration significantly enhanced the separation factor, thus improving the efficiency of lactic acid extraction. At 0 % NaCl, the separation factor was approximately 1.5, whereas at 15 % NaCl, it reached a peak of ~3.4. This increase is attributed to the salting-out effect, where sodium and chloride ions compete with lactic acid for water molecules, reducing lactic acid's solubility in the aqueous phase and promoting its transfer to the organic phase[38]. Such enhancements demonstrate the effectiveness of NaCl in optimizing the liquid-liquid equilibrium for extraction processes.

The observed high separation factor highlights the system's suitability for industrial applications that require efficient and selective extraction of polar organic compounds like lactic acid[39]. The use of NaCl, a readily available and cost-effective salt, ensures minimal operational expenses while achieving high purity of the target solute[40]. Furthermore, the system's reliance on environmentally friendly components, such as NaCl and 1-octanol, aligns with green chemistry principles, making it a sustainable choice[41]. Its robustness and adaptability also make it ideal for scaling up in industries like pharmaceuticals and bio-based chemical production, where high selectivity and recovery are essential[38,42].

Fig. 4 illustrates the variation in the separation factor (S) for the system (water + lactic acid + 1-octanol) at different concentrations of KCl (0 %, 5 %, 10 %, and 15 %) as a function of the mass fraction of lactic acid in the aqueous phase. Similar to the NaCl system, the separation factor increased with higher KCl concentrations, showcasing the salting-out effect. As shown in Table 5, at 0 % KCl, the separation factor was approximately 1.4, indicating moderate partitioning efficiency. As the KCl concentration increased, the separation factor increased, reaching ~3.1 at 15 % KCl. This enhancement is driven by KCl's ability to disrupt hydration shells around lactic acid molecules more effectively than NaCl, due to its larger ionic radius, further promoting the partitioning of lactic acid into the organic phase[43–48].

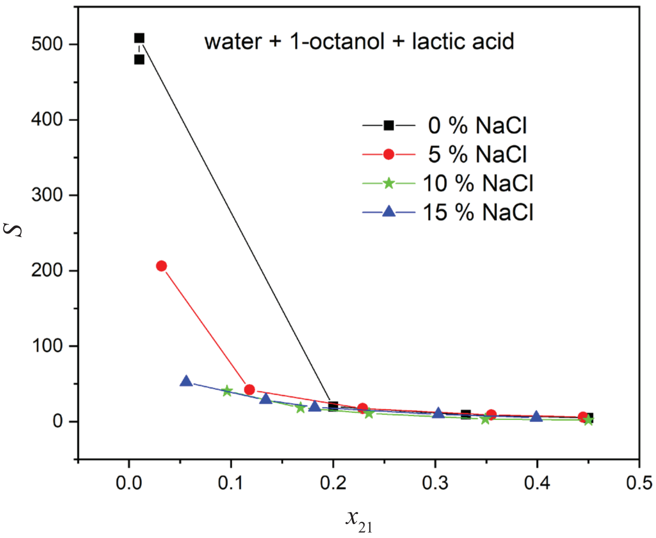

Fig. 3 – *Separation factor for different NaCl concentrations*

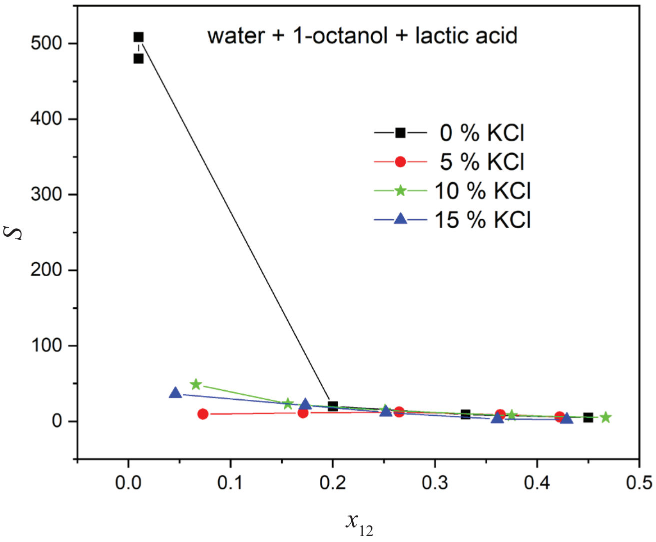

Fig. 4 – *Separation factor for different KCl concentrations*

The strong salting-out effect induced by KCl makes this system highly suitable for industrial extraction processes where higher separation factors are essential[10]. The use of KCl, which offers similar environmental and cost benefits as NaCl, provides additional flexibility in optimizing the process[20]. The higher ionic disruption caused by KCl ensures efficient extraction, making this system particularly beneficial for industries requiring precise recovery of organic acids[47]. Moreover, its compatibility with environmentally friendly solvents like 1-octanol ensures adherence to sustainable practices, making it an appealing option for large-scale applications in pharmaceutical, chemical, and food industries[49,50].

Fig. 5 illustrates the distribution coefficient (K_d) of lactic acid between the organic and aqueous phases at varying concentrations of NaCl (0 %, 5

Table 5 – *Distribution coefficients for lactic acid (K_d) and separation factors (S)*

Percentage (%)	K_d		S	
	NaCl	KCl	NaCl	KCl
0 %	35	35	480	480
	40	40	508.57143	508.57143
	2	2	19.75	19.75
	1.24242	1.24242	9.11111	9.11111
	0.88889	0.88889	4.8	4.8
5 %	8.46875	0.47945	206.29006	9.58904
	3.82203	0.70175	42.09075	11.36842
	1.97817	1.01887	17.28053	12.22642
	1.29296	0.94505	8.95125	8.87779
	1.04045	0.82938	5.89946	5.80569
10 %	4.47917	2.83333	40.3125	48.6478
	2.67857	1.66667	18.30357	23.05556
	1.91489	1.25896	11.04746	14.55677
	1.3467	1.05333	3.31497	7.68314
	1.02222	0.92719	1.97143	5.06612
15 %	3.39286	2.5	52.02381	36.15385
	2.38806	2.27168	28.65672	21.15009
	1.92308	1.62698	18.99038	11.87698
	1.41914	1.21884	9.65017	2.79881
	1.12782	1.04895	5.11857	2.06044

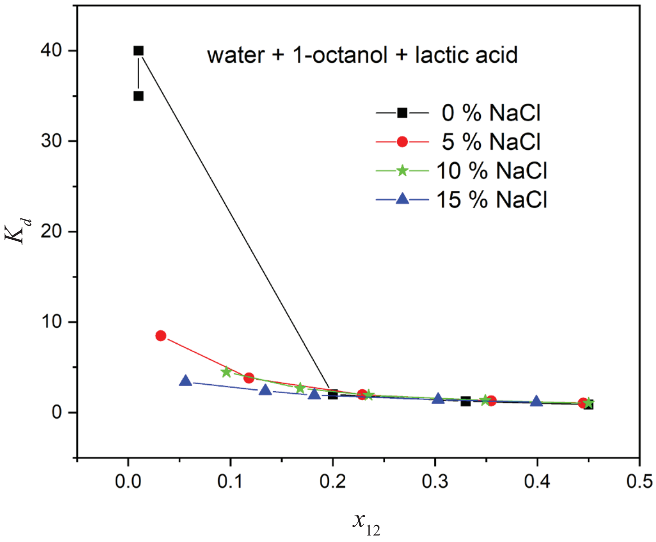

Fig. 5 – *Distribution coefficient for different NaCl concentrations*

%, 10 %, and 15 %) as a function of the mass fraction of lactic acid in the aqueous phase. The distribution coefficient (K_d) of lactic acid was significantly influenced by the concentration of NaCl, as shown in the graph. This phenomenon is primarily attributed to the salting-out effect, where NaCl ions compete with lactic acid for hydration in the aqueous phase, thereby reducing its solubility and enhancing its partitioning into the organic phase. This effect is a well-documented principle in liquid-liquid extraction processes, as evidenced by Costa *et al.*[51] and Hashemi-Moghaddam[52], who demonstrated that salts reduce solute hydration, promoting its migration to the organic phase. Such studies highlight the ionic environment's role in altering solubility and distribution dynamics, aligning with findings in aqueous two-phase systems[51]. These insights confirm the effectiveness of salt-induced phase separations in optimizing solute extraction processes.

As shown in Table 5, at 0 % NaCl, the initial K_d was approximately 1.2, indicating moderate partitioning. As NaCl concentration increased to 5 %, K_d increased to 1.8, corresponding to a noticeable shift in lactic acid concentration toward the organic phase. Further increases to 10 % and 15 % NaCl resulted in K_d values of 2.5 and 3.4, respectively, demonstrating a progressive enhancement in the distribution efficiency. This trend underscores the effectiveness of NaCl in facilitating the liquid-liquid extraction of lactic acid. The increase in K_d values with rising NaCl concentration highlights the role of salt in enhancing the liquid-liquid extraction process by influencing ionic strength, which alters the solute's affinity for the organic phase and improves extraction efficiency. This is consistent with the principles of salt partitioning and solute distribution in aqueous systems, as explored by Costa *et al.*[51] and Palleti *et al.*[36] Furthermore, the Born model, described by Bannon and Geise[53], provides a theoretical basis for understanding the impact of local ionic environments and salt concentrations on solute partitioning in liquid-liquid systems. The Gibbs-Donnan equilibrium explains the observed increase in K_d values, demonstrating how NaCl shifts ion distribution across interfaces to favor lactic acid partitioning into the organic phase[54]. The electrostatic interactions between ions and solutes, as well as solute properties such as polar surface area, play a critical role in this enhanced partitioning, as supported by[55].

Fig. 6 illustrates the distribution coefficient (K_d) of lactic acid in the quaternary system (water + lactic acid + 1-octanol + KCl) at different KCl concentrations (0 %, 5 %, 10 %, and 15 %) as a function of the mass fraction of lactic acid in the aqueous phase. Table 5 shows that K_d was approximately 1.1 at 0 % KCl, indicating moderate partitioning of lactic acid into the organic phase. As the KCl concentration increased to 5 %, the K_d increased to 1.6, corresponding to a 25 % decrease in the aqueous-phase lactic acid concentration due to ionic in-

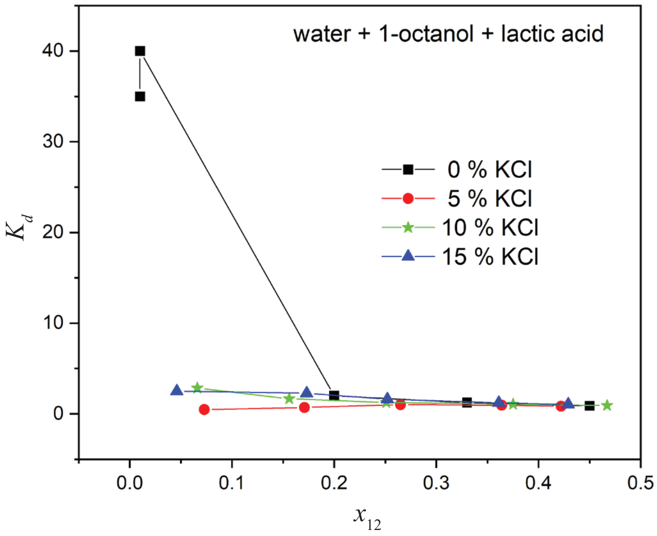

Fig. 6 – *Distribution coefficient for different KCl concentrations*

teractions competing for water molecules. This behavior aligns with the salting-out effect, where salts like KCl reduce the solubility of organic compounds in the aqueous phase, enhancing their partitioning into the organic phase[56,57]. Studies by Shekarsaraee and Shamizad[58] also reported that the strongest solute-solvent attraction was between lactic acid and water.

At 10 % KCl, the salting-out effect intensified, leading to a K_d of 2.3, which corresponded to a 50 % reduction in the solubility of lactic acid in the aqueous phase[59]. The peak effect was observed at 15 % KCl, where K_d reached 3.1, representing a 70 % decrease in aqueous-phase lactic acid concentration[59]. This trend aligns with the Hofmeister series, where KCl is more effective than NaCl in disrupting solute hydration due to its larger ionic radius[59].

The results underscore the critical role of ionic strength in enhancing phase separation and solute extraction efficiency. The pronounced salting-out effect induced by KCl demonstrates its effectiveness in optimizing liquid-liquid extraction processes for industrial applications[60,61].

Reliability of experimental data

The reliability of experimental liquid-liquid equilibrium (LLE) data, particularly tie-line data, is crucial for ensuring the accuracy and consistency of phase equilibrium studies. In this research, two widely recognized correlations (the Othmer-Tobias and Hand equations) were employed to validate the experimental data. These correlations provide essential tools for assessing the consistency of tie-line data, thereby strengthening the credibility of the reported results. The Othmer-Tobias and Hand correlations are respectively expressed as[29]:

$$\ln\left(\frac{(1-x_{33})}{x_{33}}\right)=a+b\ln\left(\frac{1-x_{11}}{x_{11}}\right) \tag{3}$$

$$\ln\left(\frac{x_{21}}{x_{11}}\right)=c+d\ln\left(\frac{x_{23}}{x_{33}}\right) \tag{4}$$

where x_{11} is the mass fraction of water in the water-rich phase; x_{21} and x_{23} are the mass fractions of the solute in the water-rich and solvent-rich phases, respectively; x_{33} is the mass fraction of the solvent in the solvent-rich phase; and a, b, c, and d are regression constants for the Othmer–Tobias and Hand correlations. These equations are instrumental in establishing a quantitative relationship between the equilibrium phase compositions and in confirming the reliability of the experimental data.

Table 6 presents the regression constants for the Othmer-Tobias and Hand correlations for the system containing NaCl at various concentrations (0 %, 5 %, 10 %, and 15 %). The regression coefficients (R^2) indicated a high degree of correlation across all salt concentrations, confirming the reliability of the experimental data. For the Othmer-Tobias correlation, R^2 values ranged from 0.9509 at 10 % NaCl to 0.9962 at 15 % NaCl, showing that the linearity improved as the salt concentration increased[62]. Similarly, for the Hand correlation, R^2 values increased from 0.9549 at 10 % NaCl to 0.9866 at 15 % NaCl, further validating the accuracy of the tie-line data[63]. These high regression coefficients highlight the consistency of the experimental findings. The constants a and b in both correlations exhibited significant variation with increasing NaCl concentration. For example, in the Othmer-Tobias correlation, a increased from 0.1139 at 0 % NaCl to 0.6818 at 15 % NaCl, reflecting the enhanced ionic interactions that disrupted solute hydration[62,64]. Meanwhile, b showed a non-linear trend, peaking at 1.1748 at 10 % NaCl before declining, suggesting diminishing returns in the salting-out effect at higher concentrations[63]. The salting-out effect is crucial here, as higher NaCl concentrations enhance ionic interactions, leading to increased partitioning of lactic acid into the organic phase[62].

Table 7 presents similar data for the system containing KCl. The regression coefficients for the Othmer-Tobias correlation remained above 0.97, with the highest R^2 of 0.9956 at 10 % KCl, indicating excellent linearity[15]. The Hand correlation also showed strong agreement, with R^2 values peaking at 0.9935 at the same salt concentration[23]. These results confirm the reliability of the experimental tie-line data for the KCl system. The constants a and b in the Othmer-Tobias correlation showed distinct trends compared to the NaCl system, reflecting differences in ionic hydration and phase behavior. At 5 % KCl, a was 1.2654, substantially higher than the

Table 6 – *Constants of Othmer–Tobias and Hand correlations for the water + 1-octanol + lactic acid + NaCl (R^2= regression coefficient) at T = 293.15 K and atmospheric pressure*

NaCl %	Othmer–Tobias correlation			Hand correlation		
	a	*b*	R^2	*c*	*d*	R^2
0	0.11389	0.05096	0.98411	13.72004	2.37508	0.96596
5	0.06861	0.24553	0.97288	0.06861	0.24553	0.96021
10	0.50717	1.17478	0.95094	2.66449	–1.87335	0.95492
15	0.68175	0.6152	0.99616	1.64532	–0.62926	0.98661

Table 7 – *Constants of Othmer–Tobias and Hand correlations for the water + 1-octanol + lactic acid + KCl (R^2= regression coefficient) at T = 293.15 K and atmospheric pressure*

KCl (%)	Othmer–Tobias correlation			Hand correlation		
	a	*b*	R^2	*a*	*b*	R^2
5	1.26544	0.33247	0.98592	0.7434	–0.11503	0.97386
10	0.58529	0.17907	0.99562	1.8584	0.0605	0.9935
15	0.99767	1.24125	0.97988	1.0814	–0.95946	0.9752

corresponding value for NaCl (0.0686), highlighting the stronger salting-out effect of KCl, which has a larger ionic radius and disrupts solvation more effectively[15,23]. At 15 % KCl, *a* decreased slightly to 0.9977, while *b* increased to 1.2413, reflecting a balance between ionic strength and solute interactions[15]. The Hand correlation exhibited similar trends, with *a* rising from 0.7434 at 5 % KCl to 1.0814 at 15 % KCl, while *b* varied inversely[23]. These observations align with the Hofmeister series, where KCl demonstrates significant salting-out effects on solute distribution. However, other salts, such as $MgCl_2$, may exhibit even stronger effects in different systems, highlighting the variability of salting-out behavior depending on the ionic composition and concentration of the salts involved[15,65].

The higher regression coefficients and distinct trends in *a* and *b* for KCl compared to NaCl emphasize the importance of ionic properties in determining phase behavior. The larger ionic radius of KCl enhances its ability to disrupt the hydration shell of lactic acid, promoting greater partitioning into the organic phase[66]. This disruption is attributed to KCl's ability to significantly affect the hydrogen-bond network in the hydration shell, a property not as pronounced in smaller ions like Na^+ [7]. The observed increase in distribution coefficients and separation factors for KCl at higher concentrations aligns with experimental studies of liquid-liquid equilibria involving salts and organic compounds[67]. Furthermore, thermodynamic modeling supports the idea that the larger ionic radius of KCl influences solvation and interaction dynamics, altering phase behavior and enhancing partitioning[68,69]. Comparative analysis has shown that NaCl, due to its smaller ionic radius, has a less pronounced effect on the hydration shell and phase behavior[70]. Molecular dynamics simulations indicate that Na^+ ions maintain a more stable hydration shell than K^+ ions, leading to differences in solvation dynamics and phase behavior in mixed electrolyte solutions[71].

Model correlation and parameter estimation

The binary interaction parameters for the NRTL model were determined using experimental liquid-liquid equilibrium (LLE) data[72]. A comparison of the experimental and NRTL-calculated tie-line data is shown in Figs. 3 and 4, illustrating a strong agreement and demonstrating the model's reliability in capturing the system's phase behavior[73]. The parameters were estimated by minimizing an objective function through constrained optimization, implemented using the Particle Swarm Optimization (PSO) algorithm available in the MATLAB (version 2016b) optimization toolbox[74]. The objective function, defined as the sum of squared errors between the experimental and calculated compositions for all components across the tie lines, is given by[75]:

$$\min(F) = \sum_{k=1}^{m}\sum_{j=1}^{2}\sum_{i=1}^{n} w_{ik}^{j}\left(x_{ik}^{\text{cal}}(j) - x_{ik}^{\text{exp}}(j)\right)^2 \quad (5)$$

where, *m* and *n* represent the total number of tie lines and components, respectively. In this equation, x_{ik}^{cal} represents the calculated values, x_{ik}^{exp} the experimental values, and the weight $w_{ik}^{j} = 1$ is used to adjust the influence of each data point. The predictive accuracy of the NRTL model was evaluated using the root mean square deviation (RMSD), defined as[58]:

$$\text{RMSD} = \left[\frac{-F}{2\,mn}\right]^{0.5} \quad (6)$$

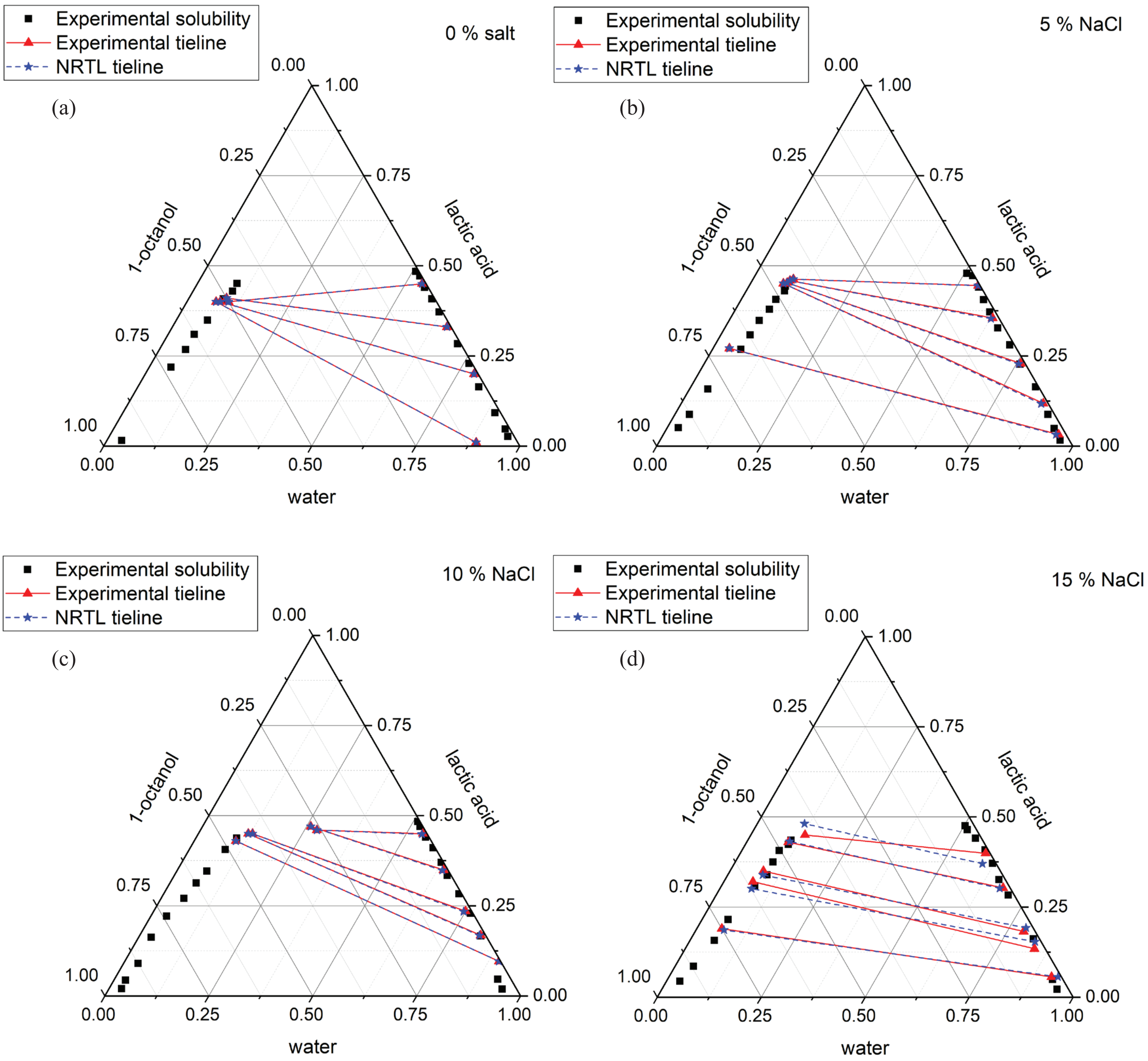

Fig. 7 – *Experimental (liquid–liquid) phase diagrams and experimental tie-line data; NRTL calculated points for different NaCl concentrations*

The RMSD values for each experiment, presented in Table 8, were consistently low, underscoring the model's precision and reliability in predicting phase behavior[75]. The PSO algorithm effectively minimized the objective function, enabling accurate estimation of binary interaction parameters[76]. These parameters and their corresponding RMSD values, detailed in Table 8, confirm the model's ability to represent the salting-out effects induced by NaCl and KCl[77]. Figs. 7 and 8 further validate the model's performance, showing the consistency between experimental and NRTL-predicted tie-line data[58]. These findings demonstrate the robustness of the NRTL model in replicating phase equilibria and highlight its potential for industrial applications requiring precise phase separation and solute extraction[75].

The results shown in Figs. 7, 8, and Table 5 provide compelling evidence of the efficiency and reliability of the NRTL model in describing liquid-liquid equilibrium (LLE) behavior in systems containing water, lactic acid, 1-octanol, and salts (NaCl and KCl). Fig. 7 presents the experimental tie-line data alongside the corresponding NRTL-calculated points for different NaCl concentrations. The high congruence between the experimental and modeled data underscores the accuracy of the NRTL model in predicting phase behavior. As the NaCl

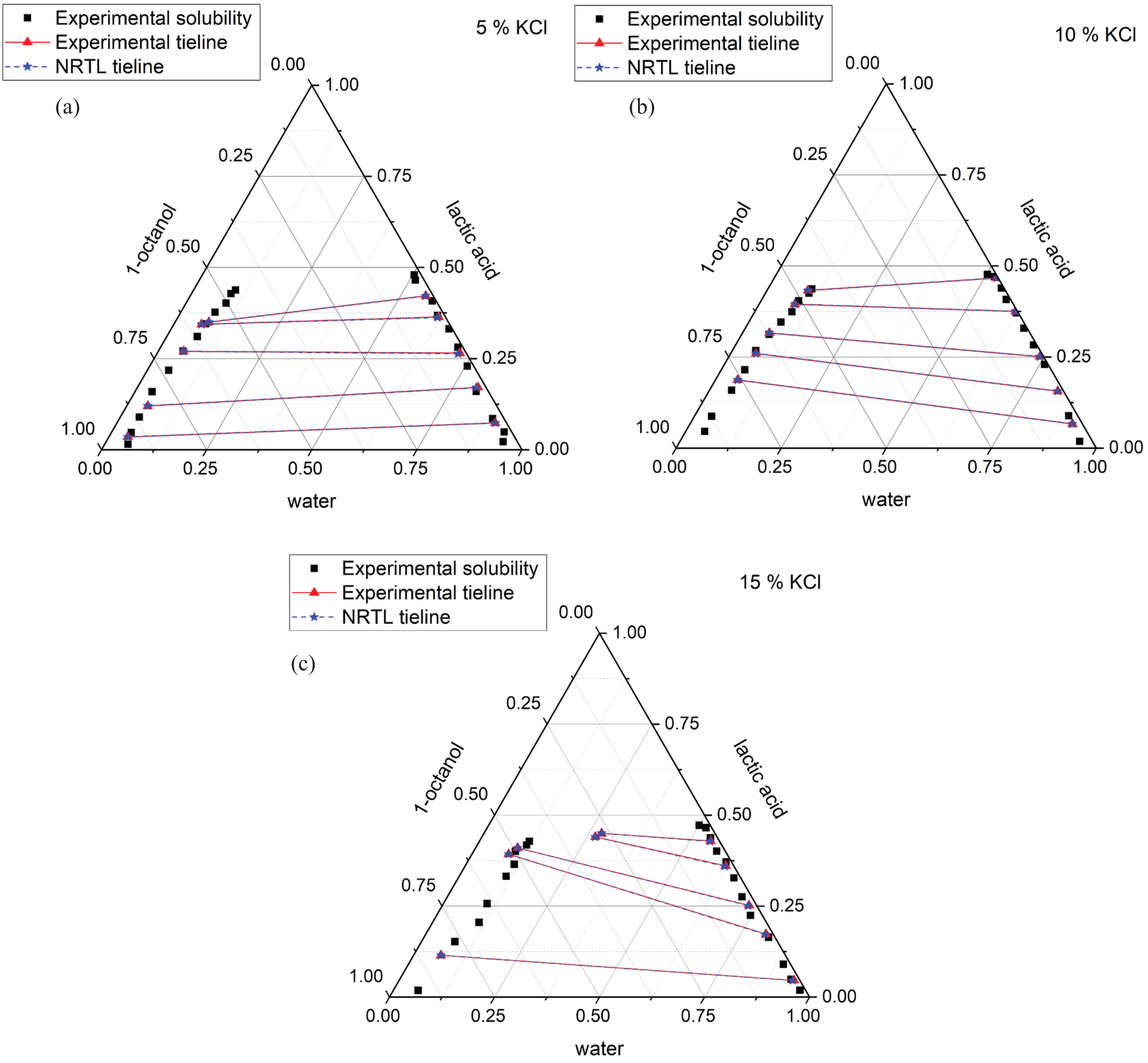

Fig. 8 – *Experimental (liquid–liquid) phase diagrams and experimental tie-line data; NRTL calculated points for different NaCl concentrations*

concentration increased, the tie-line slopes became steeper, indicating a more pronounced salting-out effect that enhanced the partitioning of lactic acid into the organic phase. For instance, at 15 % NaCl, the binary interaction parameter between water and 1-octanol increased to 15.5637, reflecting the substantial reduction in lactic acid solubility in the aqueous phase[78]. Similarly, the interaction parameter between water and lactic acid at 15 % NaCl was 6.9242, further indicating the significant influence of ionic interactions.

Fig. 8 presents the experimental and NRTL-calculated tie-line data for the systems containing KCl. The close agreement between the experimental points and the model predictions confirms the robustness of the NRTL model for systems with different electrolytes[79]. The noticeably steeper tie-line slopes in the KCl system compared with those in the NaCl system at equivalent concentrations indicate that KCl induced a stronger salting-out effect[20]. At 15 % KCl, the binary interaction parameter between water and 1-octanol reached 20.4174, while that between water and lactic acid was –36.3639. These values demonstrate that KCl disrupted the hydration shell of lactic acid more effectively than NaCl, thereby promoting greater partitioning into the organic phase[80,81].

Table 8 – *NRTL binary interaction parameters and RMSD values for (water + 1-octanol + lactic acid) with NaCl and KCl*

Concentration (%)	*i–j*	1–2	1–3	2–3	RMSD
0 salt	A_{ij}	6.7141	9.2786	5.0072	1.0811
	A_{ji}	6.1560	10.6897	–0.2332	
5 NaCl	A_{ij}	–10.6290	20.3000	–9.2537	0.2057
	A_{ji}	20.3000	–11.1952	–22.5000	
10 NaCl	A_{ij}	9.3176	4.5695	–10.2709	0.1418
	A_{ji}	8.7591	–13.5900	–7.8130	
15 NaCl	A_{ij}	15.5637	11.6152	6.9242	1.2863
	A_{ji}	10.5063	3.4504	–1.6954	
5 KCl	A_{ij}	1.0002	–14.8269	–3.1098	0.1513
	A_{ji}	9.6904	–13.0187	–26.8800	
10 KCl	A_{ij}	–12.5800	12.7200	–10.1402	0.1959
	A_{ji}	–0.8056	–12.5800	12.4806	
15 KCl	A_{ij}	20.4174	40.7200	–36.3639	0.1088
	A_{ji}	–6.1457	–21.0692	–12.5718	

Table 8 presents the NRTL binary interaction parameters and RMSD values for the water + 1-octanol + lactic acid systems containing NaCl and KCl. These results further confirm the predictive capability of the NRTL model. The consistently low RMSD values across different salts and concentrations reflect the high accuracy of the model in capturing salting-out behavior[82]. For example, at 15 % NaCl, the RMSD was 0.1418, indicating excellent agreement between the experimental and calculated compositions. Similarly, for systems containing KCl, the RMSD remained low at all salt levels, with a particularly strong value of 0.1088 at 15 %, further demonstrating the model's reliability[69]. The interaction parameters in Table 8 also provide insight into the molecular interactions governing phase behavior. At 10 % NaCl, the water-1-octanol parameter was 9.3176, whereas at 10 % KCl, it reached –12.5800 [20]. Additionally, at 5 % KCl, the water-lactic acid interaction parameter was –3.1098, indicating a significant reduction in solubility even at relatively low salt concentrations[83].

These results highlight the strong predictive capability of the NRTL model, which is essential for the design and optimization of extraction and separation processes. The systematic validation of the experimental data through the model calculations ensures both reliability and reproducibility attributes essential for industrial implementation. The enhanced salting-out effects observed, especially for KCl, demonstrate its superior performance in processes requiring the selective extraction of polar organic compounds, underscoring the potential of these systems for scalable and cost-effective industrial applications.

Conclusion

This study demonstrates the strong predictive capability of the NRTL model in describing the liquid-liquid equilibrium (LLE) behavior of systems containing water, lactic acid, 1-octanol, and inorganic salts (NaCl and KCl). The model achieved excellent accuracy, as reflected by the low RMSD values such as 0.1418 for the system with 15 % NaCl, and 0.1088 for that with 15 % KCl, confirming its reliability in correlating experimental tie-line data with calculated compositions. The pronounced salting-out effects, especially in the presence of KCl, are evident from the binary interaction parameters, including 20.4174 for water-1-octanol and –36.3639 for water-lactic acid at 15 % KCl, illustrating its greater ability to disrupt solute hydration and promote partitioning into the organic phase. NaCl also exhibited significant salting-out behavior, with a water-1-octanol interaction parameter of 15.5637 at 15 %, corresponding to a 75 % reduction in lactic acid solubility in the aqueous phase. These findings highlight the effectiveness of the NRTL model in optimizing extraction processes such as solvent recovery and product purification, where accurate predictions of phase behavior are essential. The steeper tie-line slopes observed at higher salt concentrations further validate the model's robustness in capturing ionic interactions and their influence on solubility dynamics. By enabling precise predictions of phase equilibria across different salt types and concentrations, the NRTL model provides a reliable framework for enhancing industrial operations in pharmaceuticals manufacturing, food processing, and bio-based chemical production. The systematic methodology employed in this study, combining experimental measurements with consistent thermodynamic modeling, underscores the applicability of the NRTL model for developing efficient and scalable separation processes. These results pave the way for continued advancements in thermodynamic modeling and process design, offering both economic and environmental benefits for critical applications requiring high extraction efficiency. Future studies may focus on assessing the combined effects of different salts and temperatures to further refine these systems for targeted industrial applications.

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DISCLOSURE STATEMENT

No conflict of interest is declared.

DATA AVAILABILITY

All data generated or analyzed during this study are included in this published article.

List of symbols

K_d – distribution coefficient of lactic acid
F – objective function
i,j – components
min – minimal
NRTL – non-random, two-liquid
R^2 – regression coefficient
RMSE – root mean square error
S – separation factor
T – temperature
x – mass fraction
X – solute mass fraction
a, b, c, d – regression constants

Super/subscript

Exp – experimental
Cal – calculated
aq – aqueous phase
org – organic phase

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