

Extraction of toluene from alkane using [Bmim][NO₃] or [Omim][NO₃] ionic liquid at 298.15 K and atmospheric pressure



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ABSTRACT

Extraction of toluene from alkane (hexane, heptane or octane) was studied with the ionic liquid 1-butyl 3-methylimidazolium nitrate ([Bmim][NO₃]) or 1-methyl 3-octylimidazolium nitrate ([Omim][NO₃]) at 298.15 K and atmospheric pressure. The liquid–liquid equilibrium (LLE) data for ternary systems of toluene + alkane + ionic liquid were measured and the selectivity values were calculated. The consistency of the experimental data was examined, using the Othmer–Tobias and Hand equations. The selectivity values for the ternary systems with [Bmim][NO₃] is more than [Omim][NO₃]. The experimental data were correlated with the NRTL thermodynamic model.

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1. Introduction

Extraction of toluene from alkane is a serious problem in industry because the mixtures of these components have a close boiling point and several azeotropes are formed. The separation process of these mixtures cannot be carried out by simple distillation. Liquid–liquid extraction or solvent extraction is one of the conventional processes for these mixtures.

There are different solvents used for the separation of aromatic from alkane include sulfolane [1–8], *N*-methylpyrrolidone [8](NMP), *N*-formyl morpholine (NFM) and ethylene glycol [8–10]. These organic solvents are toxic, flammable and highly volatile. Ionic liquids can be replaced by these solvents. The properties of ionic liquids include low vapor pressure, non-toxicity, non-flammability and being liquid in a wide range of temperature [11]. The extractions of toluene from alkane using ILs have been studied by many researchers in the last decades [12–21]. Meindersma et al. studied the application of IL for the separation of aromatic from alkane mixtures [22]. They have examined several types of ILs for the separation of toluene from alkane and found some ILs can be replaced the conventional solvents for extraction of toluene or other aromatic compound from alkane [22]. Recently, Corderi et al. studied the extraction of toluene from alkane mixture using ILs with different cation [23].

This research is the continuation of our previous work on the extraction of aromatic from their mixtures with alkane and ILs [24–26]. In this work, LLE data for the ternary systems {hexane + toluene + 1-butyl 3-methylimidazolium nitrate [Bmim][NO₃] or 1-methyl 3-octylimidazolium nitrate [Omim][NO₃]}, {heptane + toluene + [Bmim][NO₃] or [Omim][NO₃]} and {octane + toluene + [Bmim][NO₃] or [Omim][NO₃]} was determined at 298.15 K and atmospheric pressure. In order to analyze the suitability of these ILs as a new solvent, the selectivity (*S*), and the distribution coefficient ratio (*β*) were calculated from the experimental data. The reliability of experimental data was tested by the Othmer–Tobias [27] and Hand [28] equations. Finally, the LLE data were correlated using the NRTL activity coefficient model [29].

2. Experiments

2.1. Chemicals

The suppliers and the purities of the chemicals are reported in Table 1.

The ILs were synthesized and purified in our laboratory. They were prepared from [Bmim][Cl] or [Omim][Cl] according to the procedure in literature [30]. [Bmim][Cl] or [Omim][Cl] were synthesized according to the method described in the literature [31,32] and the procedure is available in our previous work [12]. The structure of the synthesized IL was checked with nuclear magnetic resonance (NMR) spectroscopy. The purified ILs was dried and

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Table 1
The purities and suppliers of the chemicals.

Chemical name	Supplier	Mass fraction purity
Hexane	Merck	0.99
Heptanes	Merck	0.99
Octane	Merck	0.99
Toluene	Merck	0.99
Acetone	Merck	0.99
[Bmim][NO ₃]	Synthesized in lab	0.99
[Omim][NO ₃]		0.99

degassed for 24 h at 343.15 K under vacuum, and was kept in bottle under argon gas. The water content of the dried IL was measured by a 684 Karl Fischer coulometer. The mass fraction of water was less than 1×10^{-3} .

2.2. Apparatus and procedure

The LLE data for the ternary mixtures of toluene + alkane (hexane or heptane or octane) + IL ([Bmim][NO₃] or [Omim][NO₃]) were determined at 298.15 K. The detail descriptions of the experimental procedure are available in previous papers [25,26]. The mixtures of the ternaries systems were weighted by a laboratory balance with the precision of $\pm 10^{-4}$ g. The weight of each ternary mixture was 10 g and the composition of the mixtures was selected in a region that the two phases were formed. In order to reach the equilibrium condition, the ternary mixtures were placed into a water bath (Julabo, FP50) at 298.15 K for 12 h. The precision of the water bath temperature was ± 0.1 K and the standard uncertainty of the measured temperature was less than ± 0.1 K. The pressure was

measured using mercury pressure gauge and the standard uncertainty was ± 1 KPa. After 12 h, the two-phase systems were in equilibrium and the top and bottom phases were separated. The compositions of each phase were measured using gas chromatography (GC). The model of GC was Varian, cp 3800 with a FID detector and capillary column (Chrompack, 30 m $\times$ 0.25 mm $\times$ 1.2 μ m).

The compositions of the top phases were toluene and alkane and no IL detected in this phase. The NMR analysis is confirmed this conclusion. The samples of the top phase were injected directly into the GC. The experimental standard uncertainties were less than $\pm 3 \times 10^{-3}$ in mole fraction. The samples of the bottom phase were dissolved in acetone and injected into the GC. The IL of the bottom phase samples was collected into the injector liner because GC cannot detect the IL composition. The experimental standard uncertainty was estimated as $\pm 1 \times 10^{-3}$ in mole fraction.

3. Result and discussion

The experimental LLE data for ternary systems of [Bmim][NO₃] or [Omim][NO₃] (1) + alkane (2) + toluene (3) at 298.15 K and ambient pressure (88 KPa) are reported in Tables 2 and 3.

The reliability of the LLE data was tested by the Othmer–Tobias [27] and Hand [28] equations:

$$\ln \left(\frac{1 - w_2^I}{w_2^I} \right) = a + b \ln \left(\frac{1 - w_1^{II}}{w_1^{II}} \right), \quad (1)$$

$$\ln \left(\frac{w_3^I}{w_2^I} \right) = a + b \ln \left(\frac{w_3^{II}}{w_1^{II}} \right), \quad (2)$$

Table 2
Experimental LLE data in mole fraction for ternary systems of [Bmim][NO₃] (1) + alkane (2) + toluene (3) and calculated aromatic distribution coefficient (β) and selectivity (S) values at 298.15 K and 88 KPa.

Feed			Alkane rich phase ^a		IL rich phase ^b		β	S
x_1	x_2	x_3	x_2^I	x_3^I	x_2^{II}	x_3^{II}		
[Bmim][NO₃] (1) + hexane (2) + toluene (3)								
0.303	0.564	0.133	0.832	0.168	0.008	0.060	0.36	38.88
0.305	0.428	0.267	0.644	0.356	0.009	0.0961	0.27	19.94
0.306	0.359	0.335	0.548	0.452	0.010	0.115	0.26	14.12
0.301	0.285	0.414	0.435	0.565	0.011	0.138	0.24	10.16
0.309	0.145	0.546	0.231	0.769	0.010	0.191	0.25	5.82
0.223	0.655	0.122	0.856	0.144	0.008	0.053	0.37	40.05
0.225	0.523	0.252	0.693	0.307	0.009	0.085	0.28	21.35
0.228	0.403	0.369	0.544	0.456	0.010	0.113	0.27	15.21
0.230	0.270	0.500	0.368	0.632	0.011	0.148	0.23	7.81
0.227	0.138	0.635	0.193	0.807	0.008	0.226	0.28	6.69
[Bmim][NO₃] (1) + heptane (2) + toluene (3)								
0.331	0.526	0.143	0.815	0.185	0.007	0.066	0.36	43.70
0.326	0.390	0.284	0.612	0.388	0.007	0.105	0.27	24.15
0.332	0.318	0.350	0.511	0.489	0.006	0.126	0.26	20.63
0.317	0.255	0.428	0.406	0.594	0.006	0.153	0.26	18.25
0.320	0.129	0.551	0.216	0.784	0.007	0.226	0.29	9.24
0.248	0.617	0.135	0.837	0.163	0.007	0.058	0.35	40.87
0.245	0.485	0.270	0.664	0.336	0.008	0.092	0.27	22.25
0.243	0.362	0.395	0.501	0.499	0.008	0.125	0.25	15.34
0.248	0.237	0.515	0.338	0.662	0.010	0.179	0.27	9.30
[Bmim][NO₃] (1) + octane (2) + toluene (3)								
0.342	0.500	0.158	0.786	0.214	0.004	0.061	0.29	57.45
0.339	0.362	0.299	0.589	0.411	0.005	0.123	0.30	37.52
0.321	0.302	0.377	0.487	0.513	0.006	0.158	0.31	26.31
0.330	0.237	0.433	0.399	0.601	0.006	0.195	0.32	21.31
0.318	0.115	0.567	0.204	0.796	0.007	0.284	0.36	10.88
0.265	0.590	0.145	0.818	0.182	0.004	0.053	0.29	56.01
0.258	0.458	0.284	0.642	0.358	0.005	0.104	0.29	36.63
0.250	0.332	0.418	0.473	0.527	0.006	0.164	0.31	25.77
0.246	0.216	0.538	0.318	0.682	0.005	0.239	0.35	21.76

^a Exp standard uncertainty = ± 0.003 .

^b Exp standard uncertainty = ± 0.001 .

Table 3

Experimental LLE data in mole fraction for ternary systems of [Omim][NO₃] (1) + alkane (2) + toluene (3) and calculated aromatic distribution coefficient (β) and selectivity (S) values at 298.15 K and 88 KPa.

Feed			Alkane rich phase ^a		IL rich phase ^b		β	S
x_1	x_2	x_3	x_2^I	x_3^I	x_2^{II}	x_3^{II}		
[Omim][NO₃] (1) + hexane (2) + toluene (3)								
0.254	0.605	0.141	0.846	0.154	0.093	0.115	0.75	6.84
0.256	0.458	0.286	0.677	0.323	0.100	0.224	0.69	4.71
0.257	0.384	0.359	0.583	0.417	0.101	0.278	0.67	3.68
0.258	0.309	0.433	0.489	0.511	0.100	0.341	0.67	3.34
0.261	0.156	0.583	0.292	0.708	0.007	0.501	0.71	3.14
0.183	0.688	0.129	0.863	0.137	0.100	0.103	0.75	6.68
0.183	0.544	0.273	0.700	0.300	0.106	0.198	0.66	4.39
0.187	0.421	0.392	0.561	0.439	0.109	0.288	0.66	3.37
0.189	0.283	0.530	0.407	0.593	0.093	0.431	0.73	3.17
0.206	0.139	0.655	0.244	0.756	0.061	0.578	0.77	3.08
[Omim][NO₃] (1) + heptane (2) + toluene (3)								
0.272	0.557	0.171	0.812	0.188	0.075	0.141	0.75	8.11
0.270	0.419	0.311	0.640	0.360	0.066	0.233	0.65	6.25
0.270	0.349	0.381	0.548	0.452	0.066	0.278	0.61	5.09
0.270	0.281	0.449	0.452	0.548	0.066	0.328	0.60	4.09
0.260	0.157	0.583	0.269	0.731	0.045	0.435	0.60	3.52
0.204	0.653	0.143	0.847	0.153	0.083	0.116	0.76	7.71
0.202	0.518	0.280	0.692	0.308	0.085	0.211	0.69	5.61
0.198	0.385	0.417	0.526	0.474	0.068	0.290	0.61	4.76
0.198	0.254	0.548	0.361	0.639	0.059	0.380	0.60	3.63
0.194	0.127	0.679	0.192	0.808	0.047	0.516	0.64	2.68
[Omim][NO₃] (1) + octane (2) + toluene (3)								
0.298	0.547	0.155	0.824	0.176	0.060	0.117	0.76	9.17
0.295	0.399	0.306	0.642	0.358	0.058	0.233	0.65	7.17
0.292	0.329	0.379	0.546	0.454	0.058	0.287	0.63	5.95
0.289	0.263	0.448	0.452	0.548	0.054	0.338	0.62	5.15
0.285	0.128	0.587	0.244	0.756	0.043	0.463	0.61	3.50
0.224	0.630	0.146	0.839	0.161	0.061	0.107	0.66	9.04
0.218	0.501	0.281	0.685	0.315	0.062	0.201	0.64	7.10
0.216	0.364	0.420	0.516	0.484	0.054	0.292	0.60	5.81
0.210	0.237	0.553	0.351	0.649	0.050	0.392	0.60	4.27
0.208	0.118	0.674	0.186	0.814	0.040	0.525	0.65	2.80

^a Exp standard uncertainty = ± 0.003 .

^b Exp standard uncertainty = ± 0.001 .

where w_2^I and w_3^I are the mass fractions of alkane and toluene, respectively, in the alkane rich phase; w_1^{II} and w_3^{II} are the mass fractions of IL and toluene, respectively, in the IL rich phase; and a , b , c and d are adjustable parameters. The adjustable parameters and the standard deviation (R^2) for both equations are given in Tables 4 and 5. The standard deviations are near to unity and good agreements exist for the experimental data with Othmer–Tobias and Hand equations.

Table 4

The Othmer–Tobias parameters and the standard deviation (R^2) for ternary systems of IL (1) + alkane (2) + toluene (3) at 298.15 K and 88 KPa.

a	b	R^2
[Bmim][NO₃] (1) + hexane (2) + toluene (3)		
5.750	2.135	0.993
[Bmim][NO₃] (1) + heptane (2) + toluene (3)		
5.171	2.008	0.997
[Bmim][NO₃] (1) + octane (2) + toluene (3)		
3.336	1.431	0.991
[Omim][NO₃] (1) + hexane (2) + toluene (3)		
1.988	1.506	0.994
[Omim][NO₃] (1) + heptane (2) + toluene (3)		
2.998	2.001	0.994
[Omim][NO₃] (1) + octane (2) + toluene (3)		
2.485	1.731	0.997

In order to evaluate the ILs capacities as a solvent for extraction of toluene from alkanes, the selectivity (S) and distribution ratio factor (β) were calculated.

$$S = \frac{x_3^{II} x_2^I}{x_3^I x_2^{II}}, \quad (3)$$

$$\beta = \frac{x_3^{II}}{x_3^I}, \quad (4)$$

Table 5

The Hand parameters and the standard deviation (R^2) for ternary systems of IL (1) + alkane (2) + toluene (3) at 298.15 K and 88 KPa.

c	d	R^2
[Bmim][NO₃] (1) + hexane (2) + toluene (3)		
5.592	2.019	0.992
[Bmim][NO₃] (1) + heptane (2) + toluene (3)		
4.995	1.893	0.997
[Bmim][NO₃] (1) + octane (2) + toluene (3)		
3.284	1.383	0.990
[Omim][NO₃] (1) + hexane (2) + toluene (3)		
1.897	1.191	0.995
[Omim][NO₃] (1) + heptane (2) + toluene (3)		
2.673	1.546	0.995
[Omim][NO₃] (1) + octane (2) + toluene (3)		
2.295	1.400	0.990

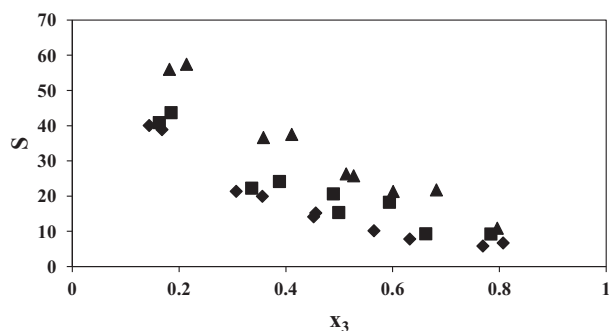


Fig. 1. Toluene selectivity as a function of the toluene mole fraction in the alkane rich phase for the ternary systems [Bmim][NO₃] (1) + alkane (2) + toluene (3) at 298.15 K: ♦ hexane, ■ heptane, ▲ octane.

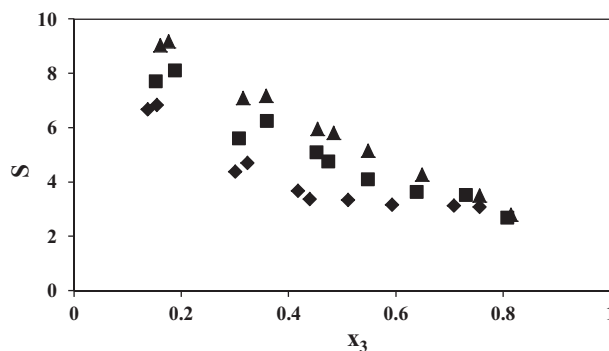


Fig. 2. Toluene selectivity as a function of the toluene mole fraction in the alkane rich phase for the ternary systems [Omim][NO₃] (1) + alkane (2) + toluene (3) at 298.15 K: ♦ hexane, ■ heptane, ▲ octane.

Table 6

The correlated binary interaction parameters (Δg) for NRTL model and the root mean square deviation (rmsd) for experimental ternary systems of IL + alkane + toluene.

Component	NRTL parameter		rmsd
<i>i</i> - <i>j</i>	Δg_{ij} (kJ mol ⁻¹)	Δg_{ji} (kJ mol ⁻¹)	
[Bmim][NO₃] (1) + hexane (2) + toluene (3)			
1-2	-3.550	20.773	0.18
1-3	4.469	18.331	
2-3	5.810	4.341	
[Bmim][NO₃] (1) + heptane (2) + toluene (3)			
1-2	-3.389	20.182	0.20
1-3	4.831	19.387	
2-3	6.565	19.111	
[Bmim][NO₃] (1) + octane (2) + toluene (3)			
1-2	-4.185	23.442	0.23
1-3	2.156	19.318	
2-3	3.966	6.691	
[Omim][NO₃] (1) + hexane (2) + toluene (3)			
1-2	0.845	8.629	0.23
1-3	3.706	13.645	
2-3	3.814	9.218	
[Omim][NO₃] (1) + heptane (2) + toluene (3)			
1-2	12.907	25.248	0.39
1-3	-1.365	2.877	
2-3	-0.606	8.374	
[Omim][NO₃] (1) + octane (2) + toluene (3)			
1-2	6.891	19.876	0.37
1-3	-0.603	8.397	
2-3	-1.369	4.112	

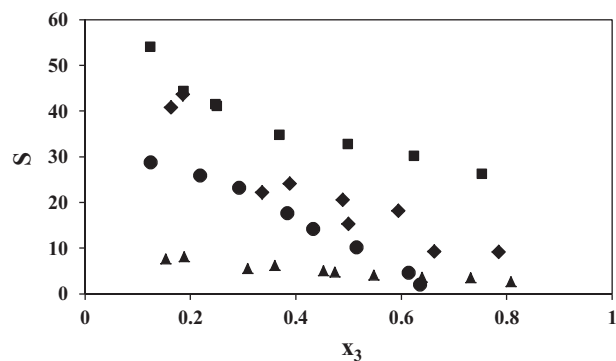


Fig. 3. The comparison of the selectivity with mole fraction for different types of ILs for the ternary systems {IL (1) + heptane (2) + toluene (3)}: ♦ [Bmim][NO₃] (this work); ▲ [Omim][NO₃] (this work); ■ [EMPy][ESO₄] [14]; ● sulfonate [5].

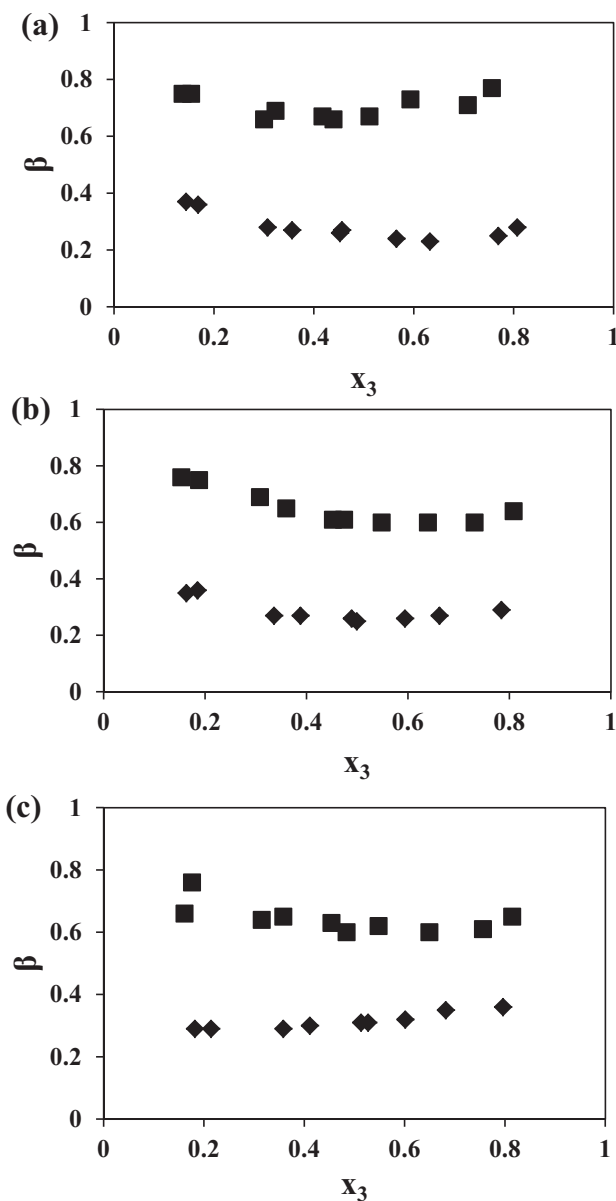


Fig. 4. Toluene distribution coefficient against toluene mole fraction in the alkane rich phase for the ternary system IL (1) + alkane (2) + toluene (3) at 298.15 K, ♦ [Bmim][NO₃], ■ [Omim][NO₃], (a) hexane, (b) heptane, (c) octane.

where x_2^I and x_3^I are the mole fraction of alkane and toluene, respectively in the alkane rich phase and x_2^{II} and x_3^{II} are the mole fraction of the alkane and toluene in the IL rich phase, respectively. As Tables 2 and 3 show, the selectivities for [Bmim][NO₃] are higher than [Omim][NO₃] systems that means fewer stages are required for the separation process [33]. As Marciniak and Krolikowski [33] were pointed out, higher selectivity corresponds to fewer stages required for a given separation process and higher distribution coefficient corresponds to a lower solvent usage for given separation, consequently smaller apparatus and lower operating costs.

The effect of alkane chain length on selectivity of toluene is represented in Figs. 1 and 2. In these figures, the toluene selectivity against the toluene mole fraction in alkane rich phase is plotted. As the figures show, the selectivity of toluene is increased with enlargement of the alkane chain length. The experimental selectivity values for both ILs are compared with the selectivity values of other ILs and sulfolane [5,15] for the ternary system of IL (1) + heptane (2) + toluene (3) in Fig. 3. As the figure shows, the selectivity values for the [Bmim][NO₃] are higher than sulfolane that means [Bmim][NO₃] is a suitable substitution for the conventional solvent. The comparison of selectivity values of both types of studied ILs indicates that an increase of the alkyl chain length of the IL cation leads to a decrease of the values of selectivity. This

phenomenon was observed by Corderi et al. [34] for extraction of toluene from alkane using other ILs.

Also the distribution coefficient values of both types of studied ILs against toluene mole fraction in the alkane rich phase are compared in Fig. 4. As the figure shows, the distribution coefficient values of [Omim][NO₃] are more than the [Bmim][NO₃] that indicate, in equal conditions less amount of [Omim][NO₃] will be used as a solvent.

4. Thermodynamic modeling

The NRTL thermodynamic model [29] was used for correlating the LLE data. The nonrandomness factor (α) was set to 0.3, and the binary interaction parameter, Δg_{ij} , were estimated from experimental data. The objective function was defined to minimize the difference between the experimental and calculated mole fraction of each component in each phase.

$$F = \sum_i^m \sum_j^{n-1} \sum_k^2 \left(\frac{x_{ijk}^{\text{exp}} - x_{ijk}^{\text{cal}}}{x_{ijk}^{\text{exp}}} \right)^2, \quad (5)$$

where m is the number of tie lines, n is the number of component, k is the number of phases, and superscripts of exp and cal referred

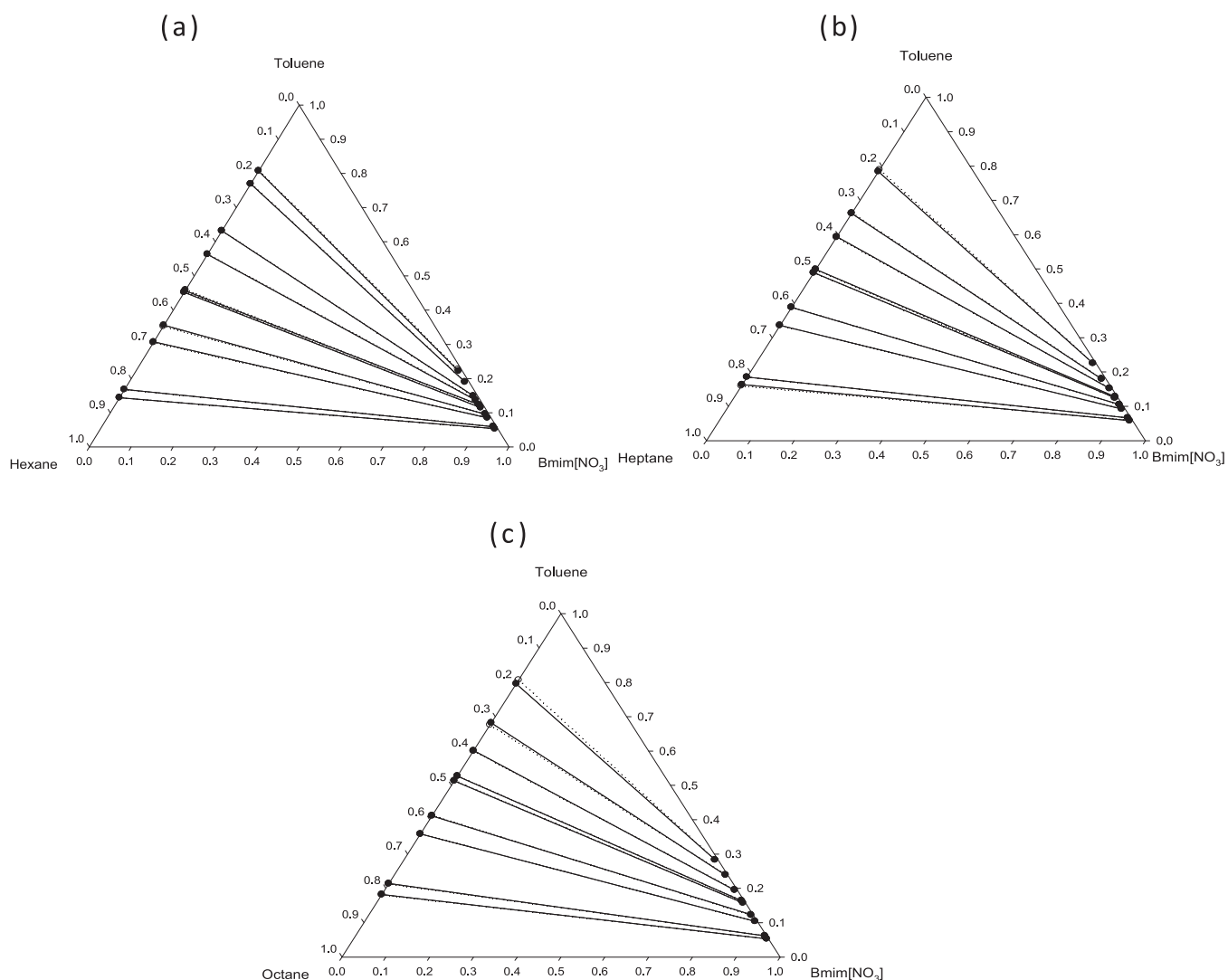


Fig. 5. Experimental and calculated LLE of the ternary systems [Bmim][NO₃] (1) + alkane (2) + toluene (3) at 298.15 K, solid lines indicate the experimental data and dashed line indicate the NRTL correlation.

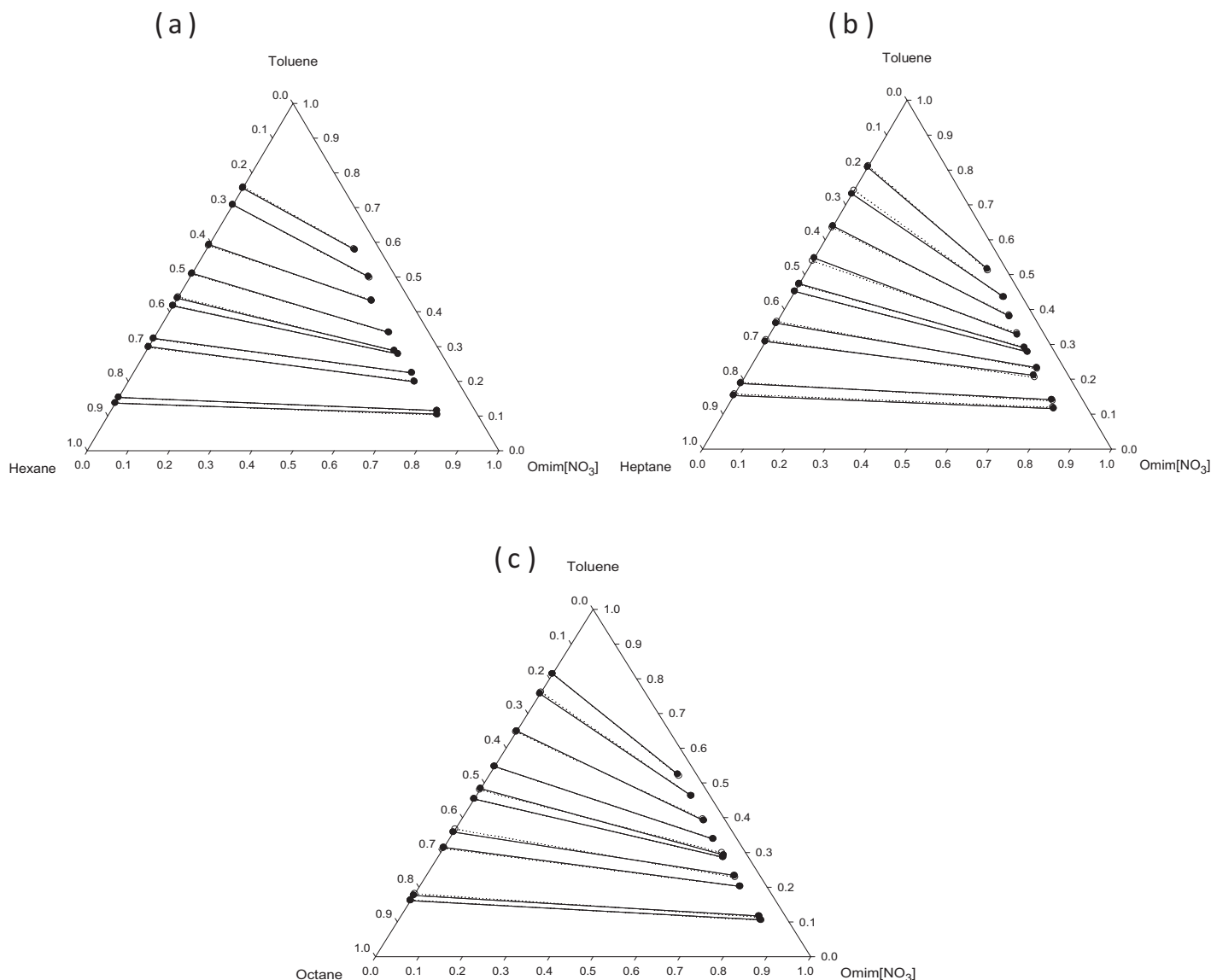


Fig. 6. Experimental and calculated LLE of the ternary systems [Omim][NO₃] (1) + alkane (2) + toluene (3) at 298.15 K, solid lines indicate the experimental data and dashed lines indicate the NRTL correlation.

to experimental and calculated mole fraction values, respectively. The binary interaction parameters were calculated by minimization of the objective function and the deviation from the experimental data was calculated. The minimization was performed with the solver excel software. The root mean square deviation is defined as follow:

$$\text{rmsd} = \left[\frac{\sum_i^m \sum_{j=1}^{n-1} \sum_k^2 \left(\frac{x_{ijk}^{\text{exp}} - x_{ijk}^{\text{cal}}}{x_{ijk}^{\text{exp}}} \right)^2}{2mn} \right]^{1/2} \times 100, \quad (6)$$

The binary energy parameters and the values of rmsd are reported in Table 6.

The ternary phase diagrams for the studied systems together with the correlation obtained by the NRTL model are plotted in Figs. 5 and 6. As the figures show, the NRTL model correlates the LLE data with a good accuracy.

5. Conclusion

The (liquid + liquid) equilibrium data for the six ternary systems of {[Bmim][NO₃] or [Omim][NO₃] (1) + hexane (2) + toluene (3)}, {[Bmim][NO₃] or [Omim][NO₃] (1) + heptane (2) + toluene (3)} and {[Bmim][NO₃] or [Omim][NO₃] (1) + octane (2) + toluene (3)} were determined at 298.15 K and atmospheric pressure. The selectivity and distribution coefficient values were calculated in order to evaluate the potential of ILs for extraction of toluene from alkane. The experimental data indicated, the selectivity values are higher than unity for all the studied ILs, so these ILs could be used as solvents for extraction of toluene from alkane. The selectivity values increase with enlargement the alkane chain length. Therefore the selectivity values of octane are more than heptane and hexane. The selectivity values for systems with [Bmim][NO₃] are higher than [Omim][NO₃] and [Bmim][NO₃] is suitable solvent for toluene extraction. The Othmer–Tobias and Hand equation were used to examine the reliability of the experimental data and a good accuracy was observed. Finally, the LLE data were successfully correlated by the NRTL model

and the binary interaction parameters of the model were determined.

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