Abstract

The extraction of toluene from heptane was analyzed by liquid extraction with 1,3-dimethylimidazolium methylsulfate [MMim][MeSO₄] ionic liquid, as a solvent. The experimental (liquid + liquid) equilibrium (LLE) data were determined at T= 318.15 K and atmospheric pressure. Selectivity and distribution coefficient, derived from the tie-lines, were used to determine the ability of this ionic liquid as solvent for the separation of toluene from its mixtures with heptane. The degree of consistency of the experimental LLE data was ascertained using the Othmer–Tobias equation. The experimental data for LLE of the ternary system were correlated with the Non-Random Two-Liquid (NRTL) and UNIversal QUAsi-Chemical (UNI-QUAC) thermodynamic models.

Key Words

Liquid-liquid equilibrium, ionic liquids, [MMim][MeSO₄], extraction, toluene.

Introduction

The removal of aromatic hydrocarbons from gasoline to comply with stricter legislative limits in the early 1990s has resulted in a continuous attempt to improve current industrial processes for the separation of aromatic compounds [1]. Industrially, the use of toxic and flammable solvents as sulfolane, ethylene glycols or N-methylpyrrolidone requires further operations, additional investments and energy consumption [2-6]. In the last decade, ionic liquids have received great attention as solvents in experimental measurements of LLE in ternary systems [ionic liquids + aromatic hydrocarbons + aliphatic hydrocarbons] [7-24]. Among the different groups of ionic liquids, alkylsulfate-based ionic liquids have been shown to be less expensive, less viscous, more hydrolytically stable, halogen free and more environmental friendly than other ionic liquids [22, 25]. In this context, alkylsulfate-based ionic liquids have emerged as a novel alternative to traditional organic solvents due to their non-volatile nature [26]. Research activities of our laboratory comprise, among others, the extraction of aromatic compounds from aliphatic/aromatic mixtures using alkylsulfate-based ionic liquids as solvent. In previous paper [27], LLE of benzene + (hexane, heptane and cyclohexane) with 1-ethyl-3-methylimidazolium ethylsulfate, [EMim][EtSO₄] at 308.15 K was studied.
In this work, LLE for the ternary system \{heptane(x_1) + toluene(x_2) + [MMim][MeSO_4](x_3)\} was determined at T = 318.15 K and atmospheric pressure. From the experimental data, selectivity, S, and distribution coefficient, ß, were calculated to determine the feasibility of [MMim][MeSO_4] in solvent extraction processes. The consistency of tie-line data was ascertained by applying Othmer–To-bias equation [28]. The Non-Random Two-Liquid (NRTL) and UNIversal QUAsi-Chemical (UNIQUAC) models were used to correlate the experimental data for the ternary system studied [29, 30]. Figure 1 show the structure of [MMim][MeSO_4].

![Structure of 1,3-dimethylimidazolium methylsulfate ([MMim][MeSO_4]).](image)

**Experimental**

**Chemicals**

Toluene and heptane were purchased from Fluka with purity higher than 99.5% and 99.9% by mass, respectively and used as received, without further purification. The [MMim][MeSO_4] with purity higher than 98%, was obtained from Sigma-Aldrich. It was purified by heating in a vacuum at 353.15 K for 24 h to remove any traces of volatile components including water. Water content was measured using a 787 Karl Fischer Titrino, and the ionic liquid showed that the mass fraction of water was less than 3.2x10^{-3}. The ionic liquid was kept in bottles with inert gas.

The physical properties (density and refractive index) of the pure components at T = 298.15 K and literature data [31-33] are compared in Table 1. The small differences between experimental and literature data may be attributed to the presence of water or non-volatile impurities in the samples.

**Experimental procedure**

For the preparation of samples, a Mettler Toledo balance with an accuracy of ± 10^{-4} g was used and the temperature of mixtures was maintained constant in a thermostatic bath (PoliScience digital temperature controller) with a precision ± 0.01 K. Density measurements were carried out using a pycnometer of approximately 20 cm³. Firstly, the pycnometer was calibrated with distilled water of known density, and then it was filled with sample and immersed in a thermostatic bath. About 120 min later, the samples was weighed. A cover was used in order to prevent the samples from absorbing water or volatilizing. The uncertainty of the density measurements was estimated to be ± 0.05%. The refractive index was measured in a Reichert AR 200 refractometer. The uncertainty in the measurements is ± 1x10^{-4}.

**Table 1. Density (\(\rho\)) and refractive index (\(n_D\)) of pure components at T= 298.15 K.**

<table>
<thead>
<tr>
<th>component</th>
<th>(\rho) (g cm^{-3})</th>
<th>(n_D)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Experimental</td>
<td>Literature</td>
</tr>
<tr>
<td>[MMim][MeSO_4]</td>
<td>1.3271</td>
<td>1.3272 [31]</td>
</tr>
<tr>
<td>heptane</td>
<td>0.6796</td>
<td>0.6796 [32]</td>
</tr>
<tr>
<td>toluene</td>
<td>0.8622</td>
<td>0.86229 [33]</td>
</tr>
</tbody>
</table>
Determination experimental LLE.

The LLE experiments with the ternary system were performed in 30 cm³ glass jacketed vessels with screw caps providing hermetic sealing. The mixtures were gravimetrically prepared, introduced into glass jacketed vessels with magnetic stirrers, and closed. The jackets were connected to a thermostat bath to maintain a constant temperature of 318.15 K in the vessels. Previous experiments showed that equilibrium was established after about 3 h stirring (800 rpm) and then 12 h to ensure thermodynamic equilibrium and complete phase separation at constant temperature [34]. Then, samples of both layers were withdrawn using syringes. Samples from the hydrocarbon-rich phase were analyzed by ¹H NMR and showed no detectable signals arising from the ionic liquid. The phase composition was analyzed by gas chromatography using an internal standard method. It is important to mention that since the ionic liquid is immiscible with the binary systems heptane + toluene, the presence of ionic liquid in the hydrocarbon-rich phase was assumed to be zero and the ionic liquid composition in the other phase was calculated by mass balance. The gas chromatograph used was an HP 6890 series equipped with a flame ionization detector (FID), an HP-FFAP capillary column (25 m x 0.2 mm x 0.3 μm), and an empty precolumn to protect the column and collect the ionic liquid that could not be retained by liner. Measurements were done by triplicate. The averages of the three measurements were used in our results. The estimated uncertainties in the compositions were calculated as the standard deviation of the measurements, less than 0.001.

Results and discussion

The experimental LLE data (tie-lines) for the system in study at 318.15 K are show in Table 2. The binodal curve and tie-lines presented in Figure 2 show that toluene and heptane are partially soluble in [MMim][MeSO₄]. The size of the two-phase region and, consequently, the tie-lines, is significantly large. This suggest that a good separation of toluene from heptane using [MMim][MeSO₄] could be possible. The percent removal of the aromatic, distribution coefficient, β, and selectivity, S, were calculated from the experimental composition of the tie-line ends as:

\[
\% \text{removal aromatic} = \frac{w_{\text{LI}}}{w_H + w_{\text{LI}}} \quad (1)
\]

where \(w_{\text{LI}}\) and \(w_H\) are the amount toluene in ionic liquid-rich phase and hydrocarbon-rich phase, respectively.

\[
\beta = \frac{x_2^\text{II}}{x_2} \quad (2)
\]

\[
S = \frac{x_2^\text{II}x_1}{x_2^\text{II}x_1^\text{II}} = \beta \frac{x_1^\text{I}}{x_1} \quad (3)
\]

\(x_1^\text{I}\) and \(x_2^\text{I}\) are the mole fractions of heptane and toluene, respectively, in the hydrocarbons-rich phase, and \(x_1^\text{II}\) and \(x_2^\text{II}\) are the mole fractions of heptane and toluene, respectively, in the ionic liquid-rich phase. Table 2 shows the percent removal of the aromatic, distribution coefficient and selectivity.

Figure 2. Ternary phase diagram of heptane\((x_1)\) + toluene\((x_2)\) + [MMim][MeSO₄]\((x_3)\) at 318.15 K.
The selectivity is shown in Figure 3, while the distribution coefficient is shown in Figure 4. Comparisons with literature data for the ternary systems \{heptane (\(x_1\)) + toluene (\(x_2\)) + solvent (\(x_3\))\}, solvent = \[[\text{MMim}][\text{MeSO}_4]\], \[[\text{EMim}][\text{EtSO}_4]\] and sulfolane at \(T = 313.2\) K were also made [10, 22]. The literature data for the systems heptane(\(x_1\)) + toluene(\(x_2\)) + \[[\text{MMim}][\text{MeSO}_4]\] (\(x_3\)) are similar to our experimental data. The values of selectivity decrease with increasing compositions of the aromatic compound in the hydrocarbon-rich phase. It has been published [18] that the aromatic solutes and the ionic liquids cations interact in an alternating structure through \(\pi - \pi\) interactions with ionic liquid anions. As the aromatic content increases, the distance between toluene and cations becomes larger and therefore the strength of interaction and, consequently, the solute distribution ratio is reduced [18]. As can be observed in Figure 3, the use of ionic liquids leads to selectivity values higher than sulfolane (solvent traditional). The high polarity of the R-SO\(_4\) anion could explain this behavior [22]. The values of the experimental selectivity, always above 1, suggest that the toluene extraction is possible with \[[\text{MMim}][\text{MeSO}_4]\] at 318.15 K.

The literature data at 313.2 K (see Figure 3) show the effect of the nature of the ionic liquid, the selectivity values increase in the form \[[\text{EMim}][\text{EtSO}_4]\] < \[[\text{MMim}][\text{MeSO}_4]\], the alkyl chain length in the R-SO\(_4\) anion and the imidazolium cation grows and the selectivity of toluene decrease, that which would mean lesser polarity of the ionic liquid and more affinity toward heptane [22]. The values of the distribution coefficients (see figure 4) are lower than unity and this implies more complex processes and more quantities of ionic liquid, the low solubility of toluene in ionic liquid-rich phase, Figure 2, explains this behavior and low aromatic recovery from their mixture with heptane.

![Figure 3](image-url)
Figure 4. Distribution coefficient for the ternary systems heptane \((x_1)\) + toluene \((x_2)\) + solvent \((x_3)\). Solvent: ■: [MMim][MeSO_4] at 318.15 K this work, □: [MMim][MeSO_4] at 313.2 K [22], ●: [MMim][MeSO_4] at 313.2 K [10], ▲: [EMim][EtSO_4] at 313.2 K [22], ∆: [EMim][EtSO_4] at 313.2 K [10] and ○: sulfolane at 313.2 K [10].

The reliability of the experimental tie-lines can be ascertained by applying the Othmer-Tobias equation [28]:

\[
\ln \left( \frac{1 - w_3^{LI}}{w_1^{HI}} \right) = a + b \ln \left( \frac{1 - w_3^{LI}}{w_3^{LI}} \right)
\]  

(4)

where \(w_1^{HI}\) is the fraction mass of heptane in the hydrocarbon-rich phase; \(w_3^{LI}\) is the fraction mass of ionic liquid in the ionic liquid-rich phase; \(a\) and \(b\) are the adjusted parameters. The linearity of the Othmer-Tobias plot indicates the degree of consistency of the data. This procedure has been used for many authors [15-18]. The Othmer-Tobias plots for the system heptane \((x_1)\) + toluene \((x_2)\) + [MMim][MeSO_4] \((x_3)\) at 318.15 K is shown in the Figure 5. The regression coefficient is near 1, which indicates a good degree of consistency of the experimental data.

Figure 5. Othmer-Tobias plot for system heptane \((x_1)\) + toluene \((x_2)\) + [MMim][MeSO_4] \((x_3)\), at 318.15 K. Adjusted parameters and regression coefficient.

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Thermodynamic correlation. The experimental data obtained in this work were used to test the ability of NRTL [29] and UNIQUAC [30] models to describe the behavior of LLE of ternary systems containing ionic liquids.

The UNIQUAC model for the excess Gibbs energy, \(g_E\), consists in two parts: a combinatorial, entropic contribution, which accounts the molecules size and shape effects, and a residual, enthalpic contribution, that accounts for the energy interaction effects [35, 36]. The equation has the form:

\[
\frac{g_E}{RT} = \frac{g_{Ecomb}}{RT} + \frac{g_{Eres}}{RT}
\]  

(5)

\[
\frac{g_{Ecomb}}{RT} = \sum_{i=1}^c x_i \ln \phi_i + 5 \sum_{i=1}^c q_i x_i \ln \phi_i
\]  

(6)

\[
\frac{g_{Eres}}{RT} = - \sum_{i=1}^c q_i x_i \ln \left( \sum_{j=1}^c \theta_j \tau_{ij} \right)
\]  

(7)
Where the volume fraction and the area fractions are given by:

\[ \phi_i = \frac{x_i r_i}{\sum x_j r_j} \quad (8) \]

\[ \theta_i = \frac{x_i q_i}{\sum x_j q_j} \quad (9) \]

\[ \tau_{ij} = \exp\left(\frac{a_{ij}}{T}\right) = \exp\left(\frac{\Delta u_{ij}}{RT}\right) \cos \tau_j \quad (10) \]

The parameters \( r \) and \( q \) are molecular structure parameters of the pure component (van der Waals parameters), representing the volume and the surface area. For each binary mixture there are two adjustable parameters, \( \tau_{ij} \) and \( \tau_{ji} \), here \( \Delta u_{ij} \) and \( \Delta u_{ji} \) are characteristics of the energy of the \( i \)-\( j \) interactions and are weakly dependents of temperature.

The required van der Waals parameters of the UNIQUAC model, \( r_i \) and \( q_i \), were taken from literature [35, 36] and they are presented in Table 3.

### Table 3. Structural parameters for the UNIQUAC equation.

<table>
<thead>
<tr>
<th>Component</th>
<th>( r_i )</th>
<th>( q_i )</th>
</tr>
</thead>
<tbody>
<tr>
<td>heptane</td>
<td>5.174</td>
<td>4.396</td>
</tr>
<tr>
<td>toluene</td>
<td>3.923</td>
<td>2.968</td>
</tr>
<tr>
<td>[MMim][MeSO₄]</td>
<td>6.903</td>
<td>5.501</td>
</tr>
</tbody>
</table>

The NRTL model is based on local composition and is applicable to partially miscible systems. The equations of NRTL for the activity coefficient in multicomponent systems are:

\[ \ln y_i = \frac{\tau_{ij} G_{ij}}{\sum_k G_{ik} x_k} + \sum_j x_j G_{ij} \left[ \tau_{ij} - \frac{\sum_k x_k \tau_{kj} G_{ki}}{\sum_k G_{kj} x_k} \right] \quad (11) \]

\[ \tau_{ij} = \frac{\Delta g_{ij}}{RT} \quad (\tau_{ij} \neq \tau_{ji}) \quad (12) \]

\[ G_{ij} = \exp(-\alpha_{ij} \tau_{ij}) \quad (\alpha_{ij} = \alpha_{ji}) \quad (13) \]

This models has three adjustable parameters for each binary pair \( \tau_{ij}, \tau_{ji} \) and \( \alpha_{ij} \). The parameters \( \tau_{ij} \) and \( \tau_{ji} \) are related to the characteristic energy of interaction between the molecules of type \( i \) and \( j \), while the parameter \( \alpha_{ij} \) is related to the nonrandomness of the mixture. Following a common procedure, the nonrandomness parameter in the NRTL equation, \( \alpha_{ij} \), was set to different values between 0.05 and 0.2 during calculations, and the best results were achieved for the values presented in Table 4.

**Parameter estimation:** The estimation of the parameters of interaction \( \Delta u_{ij} \) and \( \Delta u_{ji} \) of UNIQUAC and \( \Delta g_{ij} \) and \( \Delta g_{ji} \) of NRTL is based on the simplex method [37] and consist in the minimization of a concentration-base objective function, \( F \) [38]:

\[ F = \sum_{k=1}^{3} \sum_{j=1}^{M} \left( \left( x_{i,j}^{\exp} - x_{i,j}^{\text{cal}} \right)^2 + \left( x_{i,j}^{\exp} - x_{i,j}^{\text{cal}} \right)^2 \right) \quad (14) \]

Here, \( D \) is number of data sets, \( N \) and \( M \) are the number of components and tie-lines in each data set; the superscripts I and II refer to the two liquid phases in equilibrium, while superscripts “exp” and “cal” refer to the experimental and calculated values of the liquid phase concentration.

The fitting parameters of the correlation models, together with the values of the root-mean-square deviation of the composition, \( \sigma_x \), are listed in Table 4. This deviation gives an idea of the quality of the correlation and they were calculated as follows:

\[ \sigma_x = 100 \left[ \sum_k \sum_{i=1}^{3} \sum_{j} \left( x_{i,j,k}^{\exp} - x_{i,j,k}^{\text{cal}} \right)^2 / 6n \right]^{1/2} \quad (15) \]
Where $x_{ijk}^{\text{exp}}$ is the fraction molar of component $i$ in the phase $j$ and in the tie-line $k$, $x_{ijk}^{\text{cal}}$ is the fraction molar calculated with NRTL or UNIQUAC and $n$ is the number of tie-lines.

After the inspection of the values of $\sigma$ presented in Table 4, it can be inferred that the NRTL and UNIQUAC models gives slightly lower deviations and although both models fit satisfactorily the experimental data. Table 5 shows the composition of the phases calculated from NRTL and UNIQUAC models for ternary system heptane ($x_1$) + toluene ($x_2$) + [MMim][MeSO₄]($x_3$) at 318.15 K.

Error of the distribution coefficient, $\Delta \beta$, is calculated with the following expression:

$$
\Delta \beta = 100 \left( \frac{1}{M} \sum_{k=1}^{M} \left( \frac{\beta_k - \beta_k^{\text{cal}}}{\beta_k} \right)^2 \right)^{1/2}
$$

where $M$ is the number of tie-lines, $\beta_k$ is experimental distribution coefficient (see Table 2) and $\beta_k^{\text{cal}}$ is distribution coefficient calculated with NRTL and UNIQUAC models, values of $\Delta \beta$ are presented in Table 5. In Figures 6 and 7 the experimental tie-lines and those obtained from the correlation with NRTL and UNIQUAC models are plotted for system heptane ($x_1$) + toluene ($x_2$) + [MMim][MeSO₄]($x_3$) at 318.15 K, and the goodness of the correlation can be visually confirmed.

In this work, (liquid+liquid) equilibria for the ternary system heptane ($x_1$) + toluene ($x_2$) + [MMim][MeSO₄]($x_3$) was determined at $T = 318.15$ K and atmospheric pressure. Selectivity and distribution coefficient were calculated from experimental data. The selectivity was higher than those of sulfolane on the whole range of compositions. However, the distribution coefficients of toluene were lower. It can be concluded that [MMim][MeSO₄] can be used to separate toluene from heptane since the

![Figure 6. Ternary phase diagram of heptane ($x_1$) + toluene ($x_2$) + [MMim][MeSO₄]($x_3$) at 318.15 K. ■: experimental data, □: calculated data from NRTL model with $\alpha_y = 0.20$.](image-url)
selectivity values are higher than unity, but taken into account that distribution coefficient values are less than unity, a high number of stages would be necessary. Regarding the planning for a process, it is important to consider that ionic liquids are easily recovered.

The consistency of the LLE data was proved by the linearity of Othmer-Tobias equation and the NRTL and UNIQUAC models satisfactorily correlate the experimental LLE data.

Acknowledgements

The financial support the University of Pamplona (project 191-2010) is gratefully acknowledged.

Figure 7. Ternary phase diagram of heptane(x₁) + toluene(x₂) + [MMim][MeSO₄](x₃) at 318.15 K. ■: experimental data, □: calculated data from UNIQUAC model.

Table 5. Composition of the phases calculated from NRTL and UNIQUAC models for ternary system heptane (x₁) + toluene (x₂) + [MMim][MeSO₄](x₃) at 318.15 K.

<table>
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<tr>
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<th>Hydrocarbon-rich phase</th>
<th>Ionic liquid-rich phase</th>
<th>β_k</th>
<th>Δβ</th>
</tr>
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<tr>
<td></td>
<td>x₁^{cal}</td>
<td>x₂^{cal}</td>
<td>x₃^{cal}</td>
<td>x₁^{cal}</td>
</tr>
<tr>
<td>heptane(x₁) + toluene(x₂) + [MMim]<a href="x%E2%82%83">MeSO₄</a> with NRTL model (xᵢ = 0.20)</td>
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<th>Δβ</th>
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<td>x₁^{cal}</td>
<td>x₂^{cal}</td>
<td>x₃^{cal}</td>
<td>x₁^{cal}</td>
</tr>
<tr>
<td>heptane(x₁) + toluene(x₂) + [MMim]<a href="x%E2%82%83">MeSO₄</a> with UNIQUAC model</td>
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