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Interfacial Tensions of Imidazolium-Based Ionic Liquids with *N*-Alkanes and Cyclohexane

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ABSTRACT: The interfacial tensions of the liquid−liquid phase boundary of four 1-alkyl-3-methylimidazolium ($[C_nmim]BF_4$, n = 3, 4, 5, 6) based ionic liquids (ILs), namely, 1-propyl-3-methylimidazolium tetrafluoroborate $[C_3mim]BF_4$, 1-butyl-3-methylimidazolium tetrafluoroborate $[C_4mim]BF_4$, 1-pentyl-3-methylimidazolium tetrafluoroborate $[C_5mim]BF_4$, and 1-hexyl-3-methylimidazolium tetrafluoroborate $[C_6mim]BF_4$ with hexane, heptane, and cyclohexane, have been measured using the platinum ring method in the temperature range from (288.15 to 328.15) K at atmospheric pressure. A linear decrease of interfacial tensions with increasing temperature was observed, and the obtained data were fitted by an empirical equation. The influence of alkyl chain length of the cation of ionic liquid on interfacial tension is discussed.

INTRODUCTION

In recent years, ionic liquids (ILs) are attracting considerable attention as potentially benign solvents in a variety of industrial fields such as chemical reaction, separation process, and electrochemistry. In spite of all this attention, there are still very few known values for the physical properties required to design and develop processes that use ILs and for their applications in engineering processes.[1] Physical properties such as interfacial tension are closely related to chemical industrial processes involving contact of phases for mass transfer purposes. For instance, the design of industrial extraction contactors requires knowledge of parameters such as mass transfer coefficients and the liquid−liquid interfacial area of the corresponding process.[2]

Moreover, to expand our knowledge on the thermodynamics and kinetics of separations in liquid−liquid extractions involving ILs, it is necessary to know the interfacial tension of ILs and immiscible molecular solvent. An exhaustive literature survey reveals that a large amount of air−liquid interfacial tension data for pure ILs system are available in the literature, while the report on the liquid−liquid interfacial tension data involving ionic liquids are scarce or absent. Hardacre reported some experimental results for the interfacial tension of 1,3-dialkyl imidazolium-based ionic liquids ($[C_2mim][NTf_2]$, $[C_4mim][NTf_2]$, $[C_6mim][NTf_2]$, $[C_8mim][NTf_2]$, $[C_4mim][CF_3SO_3]$, and $[C_4mim][CF_3COO]$) with *n*-hexane, *n*-octane, and *n*-decane as a function of temperature at atmospheric pressure.[3] Scurto et al. studied the interfacial tension of the saturated 1-octene + $[HMIm][Tf_2N]$.[4] Fei and co-workers investigated the liquid−liquid interfacial tension for $[BMIM][PF_6]$ with 21 organic compounds at 298.15 K.[5] Our previous report demonstrated that the liquid−liquid interfacial tension of four 1-alkyl-3-methylimidazolium hexafluorophosphate ($[C_nmim][PF_6]$, n = 5, 6, 7, 8) and two *n*-alkanes (n = 6, 7) show a linear decrease with increasing temperature.[6]

To extend and complete a comprehensive study of interfacial tension of several closely related imidazolium-based ILs with alkane (or cyclohexane) that our research group is developing, we present now the interfacial tension of four ILs, 1-propyl-3-methylimidazolium tetrafluoroborate $[C_3mim]BF_4$, 1-butyl-3-methylimidazolium tetrafluoroborate $[C_4mim]BF_4$, 1-pentyl-3-methylimidazolium tetrafluoroborate $[C_5mim]BF_4$, and 1-hexyl-3-methylimidazolium tetrafluoroborate $[C_6mim]BF_4$, with hexane, heptane, and cyclohexane in the temperature range from (288.15 to 328.15) K. Results for interfacial tension of $[C_nmim]BF_4$/alkane and $[C_nmim]PF_6$/alkane have been compared to analyze the influence of the anion on their interfacial tension.

EXPERIMENTAL SECTION

Materials. Four ionic liquids, $[C_3mim]BF_4$ (>99 mass %), $[C_4mim]BF_4$ (>99 mass %), $[C_5mim]BF_4$ (>99 mass %), and $[C_6mim]BF_4$ (>99 mass %) were purchased from Shanghai Chengjie Chemical Co. To remove the small amount of water, all of the ILs were dehydrated under vacuum at 80 °C for 48 h prior to experimentation. The water content in the ILs, measured by Karl Fisher titration, was lower than 0.03 %. Heptane (>99 mass %), hexane (>99 mass %), and cyclohexane (>99 mass %) were obtained from Beijing Chemical Reagent Co. and were used as received.

Measurements. Surface and interfacial tension measurements were performed on a model DCAT21 tensiometer using a ring method at different temperatures from (288.15 to 328.15) K. To avoid surface contamination, all surface tension measurements were conducted in nitrogen atmosphere. Samples were measured in our laboratory by the procedure reported in our previous literature.[6]

Each surface and interfacial tension value reported was the mean of 10 replicate measurements. The estimated uncertainty of the experimental surface or interfacial tension was $\pm$ 0.1 $mN \cdot m^{-1}$. The surface tension was calibrated with double-distilled water. The measured liquid interfacial tension value of the immiscible water/hexane system at 293.15 K was 49.9 $mN \cdot m^{-1}$, which is consistent with the value of 49.4 $mN \cdot m^{-1}$ in the literature.[7]

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Table 1. Density of Alkane-Saturated ILs at Different Temperatures

	T/K				
	288.15	298.15	308.15	318.15	328.15
alkane-saturated IL	ρ/g·cm^{-1}				
hexane-saturated [C$_3$mim]BF$_4$	1.2459	1.2388	1.2302	1.2213	1.2156
hexane-saturated [C$_4$mim]BF$_4$	1.1876	1.1816	1.1737	1.1656	1.1589
hexane-saturated [C$_5$mim]BF$_4$	1.1759	1.1698	1.1621	1.1559	1.151
hexane-saturated [C$_6$mim]BF$_4$	1.1361	1.129	1.1225	1.1106	1.0989
heptane-saturated [C$_3$mim]BF$_4$	1.2398	1.2369	1.2311	1.2289	1.2263
heptane-saturated [C$_4$mim]BF$_4$	1.1896	1.1841	1.1776	1.1724	1.1623
heptane-saturated [C$_5$mim]BF$_4$	1.1699	1.1611	1.1563	1.1462	1.1386
heptane-saturated [C$_6$mim]BF$_4$	1.1349	1.1316	1.1308	1.1258	1.1221
cyclohexane-saturated [C$_3$mim]BF$_4$	1.2349	1.2319	1.2259	1.2229	1.2203
cyclohexane-saturated [C$_4$mim]BF$_4$	1.1859	1.1812	1.1781	1.174	1.1665
cyclohexane-saturated [C$_5$mim]BF$_4$	1.1649	1.1568	1.1461	1.139	1.1337
cyclohexane-saturated [C$_6$mim]BF$_4$	1.1249	1.1208	1.1161	1.1149	1.1117
IL-saturated alkane	ρ/g·cm^{-1}				
[C$_3$mim]BF$_4$ -saturated hexane	0.6467	0.6418	0.6379	0.632	0.6235
[C$_4$mim]BF$_4$-saturated hexane	0.6497	0.6455	0.6355	0.6302	0.6263
[C$_5$mim]BF$_4$ -saturated hexane	0.6506	0.6459	0.6401	0.6298	0.6259
[C$_6$mim]BF$_4$ -saturated hexane	0.6497	0.6433	0.6405	0.6289	0.6251
[C$_3$mim]BF$_4$ -saturated heptane	0.6726	0.6679	0.664	0.6578	0.6534
[C$_4$mim]BF$_4$-saturated heptane	0.6703	0.6678	0.6645	0.6606	0.6558
[C$_5$mim]BF$_4$-saturated heptane	0.6739	0.6700	0.6653	0.6591	0.6548
[C$_6$mim]BF$_4$-saturated heptane	0.6722	0.6684	0.6642	0.6569	0.6541
[C$_3$mim]BF$_4$-saturated cyclohexane	0.7703	0.7654	0.7566	0.7523	0.7501
[C$_4$mim]BF$_4$-saturated cyclohexane	0.7632	0.7601	0.7567	0.7542	0.7489
[C$_5$mim]BF$_4$-saturated cyclohexane	0.7733	0.7622	0.7561	0.7522	0.7398
[C$_6$mim]BF$_4$-saturated cyclohexane	0.7733	0.765	0.7579	0.7539	0.7497

Table 2. Interfacial Tension for Ionic Liquids/Alkane Systems at Different Temperatures

	T/K				
IL or alkane	288.15	298.15	308.15	318.15	328.15
	γ/mN·m^{-1}				
[C$_3$mim]BF$_4$/hexane	19.08	18.77	18.38	18.13	17.90
[C$_4$mim]BF$_4$/hexane	13.49	13.26	12.69	12.26	12.19
[C$_5$mim]BF$_4$/hexane	12.00	11.99	11.68	11.62	11.60
[C$_6$mim]BF$_4$/hexane	8.56	8.54	8.28	8.26	8.23
[C$_3$mim]BF$_4$/heptane	18.53	18.52	18.49	18.38	18.30
[C$_4$mim]BF$_4$/heptane	14.53	14.34	14.08	14.01	13.96
[C$_5$mim]BF$_4$/heptane	11.85	11.83	11.82	11.80	11.79
[C$_6$mim]BF$_4$/heptane	8.63	8.60	8.55	8.45	8.34
[C$_3$mim]BF$_4$/cyclohexane	17.83	17.70	17.52	17.02	16.77
[C$_4$mim]BF$_4$/cyclohexane	13.60	13.39	13.28	13.22	13.03
[C$_5$mim]BF$_4$/cyclohexane	10.27	10.00	9.46	8.98	8.81
[C$_6$mim]BF$_4$/cyclohexane	7.04	7.02	6.70	6.69	6.59

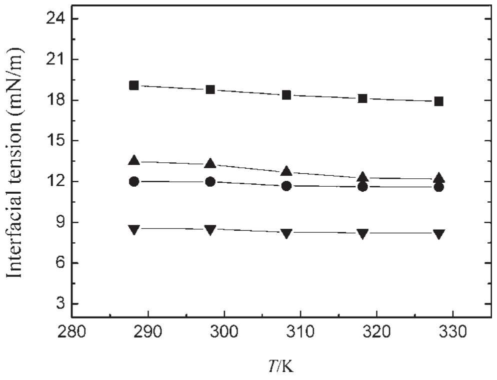

Figure 1. Experimental interfacial tension data of 1,3-dialkyl imidazolium-based ionic liquids studied with hexane as a function of temperature at atmospheric pressure: ■, [C$_3$mim]BF$_4$; ▲, [C$_4$mim]BF$_4$; ●, [C$_5$mim]BF$_4$; ▼, [C$_6$mim]BF$_4$.

The density measurement of the two liquid phases was determined using a model MYX-1 densimeter with a precision of ± 0.0002 g·cm^{-3}. Circulating water from a thermostatically regulated bath was around the measuring cell to maintain the temperature with a temperature stability of ± 0.02 K.

For the measurements of liquid–liquid interfacial tension, the mixture of alkane and ionic liquid was placed in a conical flask maintaining the required constant temperature and vigorously stirred for at least 12 h to reach the equilibrium. Afterward, the mixture was allowed to stand for several hours to completely separate into two transparent phases. The density of each liquid phase was measured separately. The measured densities of alkane-saturated ionic liquid and the ionic liquid-saturated alkane are listed in Table 1. The surface tension of the separated two liquid phase was also measured separately.

RESULTS AND DISCUSSION

The liquid–liquid interfacial tension data of the studied [C$_n$mim]BF$_4$/alkane systems were measured at (288.15 to 328.15) K and are shown in Table 2 and Figure 1. For all of the ILs/alkane systems studied in the current work, the interfacial tension data are found to slightly decrease with an increase in temperature range from (288.15 to 328.15) K. A similar trend was observed for the series of [C$_n$mim]PF$_6$/alkane reported by our previous work.[6] Ahosseini and co-workers[4] have measured the interfacial tension of the [HMIm][Tf$_2$N]/1-octane system and demonstrated that the interfacial tension value decreases with temperature to a small extent at (288.15 to 328.15) K. This phenomenon is similar to our results. Hardacre and co-workers[3] also measured the interfacial tension of [C$_n$mim][NTf$_2$]/n-alkanes systems and showed the same decreasing trend of these data with temperature.

For a given alkane or cyclohexane, a general trend is observed: the longer the cation's alkyl chain, the higher the interfacial tension ILs/alkane (or ILs/heptane) system. For example, when the carbon number in alkyl chain on the imidazolium cation increases from [C$_3$mim]$^+$ to [C$_6$mim]$^+$ for ILs/hexane system, the interfacial tension value falls from (18.77 to 8.54) mN·m^{-1} at 298.15 K. This reflects the increased van der Waals interactions between the alkyl chains of the ionic liquid and the alkane. This behavior follows the results obtained by our previous report.[6]

The anions have also a significant influence on the interfacial tension of the ILs/hexane system. By considering the interfacial tension of [C$_4$mim][NTf$_2$]/hexane, [C$_4$mim][CF$_3$SO$_3$]/hexane, and [C$_4$mim][CF$_3$COO]/hexane from Hardacre et al.[3] and the experimental interfacial tension data of [C$_4$mim]

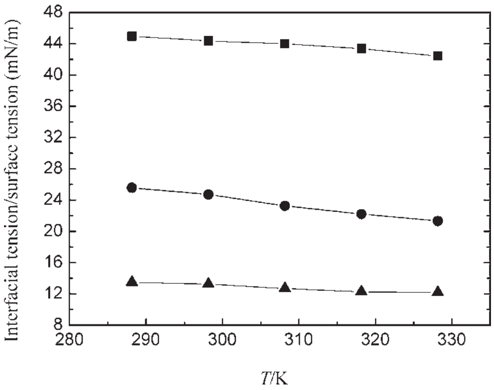

Figure 2. Interfacial tension of the $[C_4mim]BF_4$/hexane system with temperature compared to the pure component surface tensions: ■, $[C_4mim]BF_4$; ●, hexane; ▲, $[C_4mim]BF_4$/hexane.

Table 3. Surface Tension for Pure Ionic Liquids and Alkanes at Different Temperatures

	T/K				
pure substances	288.15	298.15	308.15	318.15	328.15
	γ/mN·m^{-1}				
$[C_3mim]BF_4$	50.68	50.31	49.36	48.73	48.38
$[C_4mim]BF_4$	44.97	44.37	43.99	43.36	42.43
$[C_5mim]BF_4$	42.21	41.63	41.43	40.80	40.34
$[C_6mim]BF_4$	37.89	37.34	37.03	36.78	36.26
hexane	19.01	18.03	17.12	16.23	15.23
heptane	20.43	19.68	18.89	17.95	17.02
cyclohexane	25.58	24.73	23.26	22.22	21.32

BF_4/hexane in the current work, the experimental interfacial tension data are found to decrease in the order $[BF_4]^- > [CF_3SO_3]^- > [CF_3COO]^- > [NTf_2]^-$. In the case of $[C_6mim]BF_4$/hexane, $[C_6mim]PF_6$/hexane,[6] and the $[C_6mim][NTf_2]$/hexane[3] systems, interfacial tension follows the order: $[PF_6]^- > [BF_4]^- > [NTf_2]^-$.

Interfacial tension values of alkane/ILs are much lower than the surface tension of pure ILs and the pure alkane. As shown in Figure 2 and Table 3, the interfacial tension value of $[C_4mim]BF_4$/hexane is 13.26 mN·m^{-1} at 298.15 K, while that of the pure $[C_4mim]BF_4$ is 44.37 mN·m^{-1} and hexane is 18.03 mN·m^{-1}.

The variation of the interfacial surface tension of the ILs/alkane system studied here with temperature was linear. The temperature dependence of the interfacial tension is presented typically by Figure 1. The experimental interfacial tension value can be written as[8−12]

$$\gamma/\text{mN}\cdot\text{m}^{-1} = K_1 - K_2(T/\text{K}) \tag{1}$$

where K_1 and K_2 are the interfacial tension fitting coefficients, and T is the absolute temperature. These coefficients for the series of ILs/alkane systems are given in Table 4. The absolute average deviation (AAD) is expressed as

$$\text{AAD} = \left[\frac{1}{c}\sum_{i=1}^{c}\frac{|\gamma_{i,\text{exp}} - \gamma_{i,\text{cal}}|}{\gamma_{i,\text{exp}}}\right]\cdot 100 \tag{2}$$

In this equation, c represents the number of data points. $\gamma_{i,\text{exp}}$ is the experimental interfacial tension value, and $\gamma_{i,\text{cal}}$ is the calculated interfacial tension value. The AADs obtained from the experimental and calculated values are listed in Table 4. The calculated interfacial tensions display a good agreement with the corresponding experimental surface tensions. For all of the studied system, the overall AAD is 0.43 % with a maximum deviation inferior to 0.85 %.

Table 4. Interfacial Tension Parameters K_1 and K_2 for the System

	K_1	$K_2\cdot 10^{-2}$	AAD (%)
$[C_3mim]BF_4$/hexane	27.7	3.00	0.20
$[C_4mim]BF_4$/hexane	23.87	3.60	0.81
$[C_5mim]BF_4$/hexane	15.38	1.17	0.51
$[C_6mim]BF_4$/hexane	11.27	0.94	0.55
$[C_3mim]BF_4$/heptane	20.29	0.60	0.13
$[C_4mim]BF_4$/heptane	18.71	1.47	0.38
$[C_5mim]BF_4$/heptane	12.28	0.15	0.021
$[C_6mim]BF_4$/heptane	10.76	0.73	0.28
$[C_3mim]BF_4$/cyclohexane	26.00	2.80	0.47
$[C_4mim]BF_4$/cyclohexane	17.34	1.31	0.24
$[C_5mim]BF_4$/cyclohexane	21.65	3.94	0.85
$[C_6mim]BF_4$/cyclohexane	10.60	1.23	0.71

Table 5. Model Interaction Parameters for ILs/Alkanes at Different Temperatures

	T/K				
IL/alkane	288.15	298.15	308.15	318.15	328.15
	ϕ				
$[C_3mim]BF_4$/hexane	0.8153	0.8229	0.8273	0.8326	0.8420
$[C_4mim]BF_4$/hexane	0.8634	0.8687	0.8822	0.8921	0.8944
$[C_5mim]BF_4$/hexane	0.8688	0.8700	0.8799	0.8823	0.8870
$[C_6mim]BF_4$/hexane	0.9006	0.9024	0.9109	0.9158	0.9204
H_2O/hexane		0.55[a]			
$[C_3mim]BF_4$/heptane	0.8170	0.8179	0.8148	0.8166	0.8207
$[C_4mim]BF_4$/heptane	0.8391	0.8411	0.8464	0.8477	0.8464
$[C_5mim]BF_4$/heptane	0.8648	0.8643	0.8668	0.8674	0.8696
$[C_6mim]BF_4$/heptane	0.8930	0.8931	0.8955	0.9006	0.9045
$[C_3mim]BF_4$/cyclohexane	0.8114	0.8128	0.8131	0.8195	0.8240
$[C_4mim]BF_4$/cyclohexane	0.8396	0.8409	0.8436	0.8434	0.8432
$[C_5mim]BF_4$/cyclohexane	0.8752	0.8783	0.8896	0.8974	0.9011
$[C_6mim]BF_4$/cyclohexane	0.9062	0.9058	0.9130	0.9149	0.9169

[a] Obtained from ref 16.

The liquid−liquid interfacial tension data of IL and alkane has been related to their surface tension of the pure substance. An expression was proposed by van Oss,[13] as a function of the corresponding surface tension of γ_{IL} and γ_{alk}

$$\gamma^{\text{IL-alk}} = \gamma^{\text{IL}} + \gamma^{\text{alk}} - 2\phi\sqrt{\gamma^{\text{IL}}\gamma^{\text{alk}}} \tag{3}$$

where ϕ is the interaction parameter, which is near zero for nonpolar/nonpolar systems, less than one for nonpolar/polar interfaces, and larger than one for polar/polar system. The ϕ value at different temperatures is shown in Table 5. The fact that all of the interaction parameters ϕ of ILs/alkane systems in the current work are smaller than one is in accordance with a nonpolar/polar interface. It can be seen that the ϕ value increases slightly with the alkyl chain length on the imidazolium cation from [C$_3$mim]$^+$ to [C$_6$mim]$^+$ for the series of [C$_n$mim]BF$_4$/heptane systems; a similar trend is observed for the [C$_n$mim]BF$_4$/cyclohexane systems. For example, when the alkyl chain of ionic liquid on the cation increases from 3 to 6, the interaction parameter value increases from 0.8179 to 0.8931 at 298.15 K for [C$_n$mim]BF$_4$/heptane systems. However, there is a little difference in the case of [C$_n$mim]BF$_4$/hexane; for example, [C$_5$mim]BF$_4$/hexane exhibits a slightly lower ϕ value than that of the [C$_4$mim]BF$_4$/hexane at temperatures of (308.15, 318.15, and 328.15) K. The interaction parameter ϕ is closely correlated to the contribution from polar interaction to the interfacial tension.[4] In comparison with the water/hexane system ($\phi = 0.55$, Table 5), the [C$_n$mim]BF$_4$/hexane system has a higher ϕ value, whereas the polarity of water are higher than that of the 1-alkyl-3-methylimidazolium-based ionic liquid.[14,15] The polarity of 1-alkyl-3-methylimidazolium-based ionic liquid decreases with the increase of chain length.[14,15] Therefore, for a fixed IL/alkane system at 298.15 K, the longer the chain length, the higher the polar contribution to the interfacial tension.

CONCLUSIONS

The interfacial tensions of the liquid−liquid phase boundary of four 1-alkyl-3-methylimidazolium ([C$_n$mim]BF$_4$, n = 3, 4, 5, 6) based ionic liquids (ILs) with hexane, heptane, and cyclohexane were measured in the temperature range from (288.15 to 328.15) K at atmospheric pressure. The interfacial tension data are found to slightly decrease with an increase in temperature range from (288.15 to 328.15) K. For all the ILs/alkane system, the interfacial tension value decreases with the increase of alkyl chain length on the cation at the same temperature. A linear decrease of interfacial tension with increasing temperature was observed, and the obtained data were fitted by an empirical equation. The absolute average deviations obtained from the calculated interfacial tension and the experimental values are in the range of (0.021 to 0.85) %. The interfacial tension was correlated using the air−liquid interfacial tension of pure ILs and alkanes, and the corresponding interaction parameter is just less than 1 in agreement with a nonpolar/polar interface.

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