Micellization Behavior of Ionic Liquid Surfactants with Two Hydrophobic Tail Chains in Aqueous Solution

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ABSTRACT: The drop volume method was used to determine the surface tensions of two series of polymerizable ionic liquid surfactants, namely, 1-acryloyloxypropyl-3-alkylimidazolium chloride [APC_nim][Cl] (n = 8, 10, 12) containing two hydrophobic tail chains and 1-propionyloxypropyl-3-alkylimidazolium chloride [PPC_nim][Cl] (n = 8, 10, 12), which showed similar structures with [APCnim][Cl] (n = 8, 10, 12) at 25°C. The surface properties of the surfactants were also compared. Furthermore, the thermodynamic behavior of [PPC_nim][Cl] (n = 10, 12, 14) micellization in aqueous solution and the enthalpy–entropy compensation phenomenon were studied. The two kinds of surfactants had high surface activity. Double bonds had almost no effect on the surface properties of [APC_nim][Cl]. The micellization of [PPC_nim][Cl] in aqueous solution was spontaneous and entropy-driven. For the surfactant with specific structure, micellization ability initially increased and then decreased with temperature increase. The entropic contribution to Gibbs free energy change tended to decrease, whereas the enthalpic contribution increased. In addition, the enthalpy–entropy compensation phenomenon occurred during the micellization process. For all [PPC_nim][Cl], the compensation temperature was (314 ± 1) K, which remained unaffected by the surfactant molecular structure. However, when the hydrophobic carbon chain length increased, both micelle formation ability and stability increased. © 2013 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 129: 2057–2062, 2013

KEYWORDS: colloids; ionic liquids; micelles; surfaces and interfaces; surfactants

Received 20 June 2012; accepted 3 December 2012; published online 7 January 2013 DOI: 10.1002/app.38914

INTRODUCTION

Ionic liquid^{1,2} consists entirely of ions, most commonly liquid organic molten salts at room temperature or near room temperature. The specific functional ionic liquid could be obtained³ by changing the anion or cation structure of the ionic liquid. Ionic liquid surfactants are one type of functional ionic liquids with hydrophobic side chains and hydrophilic head, which confer amphiphilic properties. The ionic liquid surfactant structure is thus similar to that of a traditional surfactant. The long-chain imidazolium ionic liquid, an ionic liquid surfactant, possesses higher adsorption efficiency and efficacy than traditional cationic surfactants with the same hydrophobic carbon chain and the same efficiency and efficacy as anionic surfactants, such as alkyl sodium sulfate.^{4,5} Ionic liquid surfactants have good surface activity and low toxicity. They are easy to prepare, and their structures are easy to control.^{6,7} Thus far, reports on long chain imidazolium ionic liquid have mainly focused on the synthesis,^{8,9} application,^{10,11} and micellization behavior^{12,13} in aqueous solution of the 1alkyl-3-methyl imidazolium ionic liquid surfactants and Gemini imidazolium ionic liquid surfactants.^{14–16} No reports on imidazolium ionic liquid with double hydrophobic chains have yet been published.

Two series of cationic polymerizable ionic liquid surfactants were synthesized in this article. Their surface properties and enthalpy– entropy compensation phenomenon were discussed. We found that these surfactants were more surface active than Gemini imidazolium ionic liquid surfactants which was agreed with the theory proposed by Zheng et al.¹⁷ and El-Sherbiny.¹⁸ They found that excellent surface activities of the new surfactants, which may be due to their special structure can be measured in terms of their surface tensions. The properties for different functional requirements of these polymerizable ionic liquid surfactants could be controlled by changing their anion, cation, and alkyl substituent. In this research, the ionic liquid surfactants were synthesized via molecular self-assembly process, which was a new topic in recent years. For larger charge-density of the cation in the molecular structure, these polymerizable ionic liquid surfactants represented

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Figure 1. FTIR (KBr coating) spectrum of [APC₁₀im][Cl]. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

a good ability of carbon dioxide adsorption. And the polymers polymerized via these polymerizable ionic liquid surfactants showed well ionic conducting properties. The conductivity could be greatly improved after adding with lithium salt. These polymers could be expected as solid electrolyte and be applied to the production of solid battery. Therefore, the relationship between structure and properties of ionic liquid and surfactant should be paid more attention in the future work. And more meaningful polymerizable ionic liquid surfactants would be synthesized and be applied in every field. This study would bring a new theoretical research foundation in green chemistry, supramolecular chemistry, and life science.

EXPERIMENTAL

Materials

1-acryloyloxypropyl-3-alkylimidazolium chloride $[APC_nim][Cl]$ (n = 8, 10, 12) and 1-propionyloxypropyl-3-alkylimidazolium chloride $[PPC_nim][Cl]$ (n = 8, 10, 12, 14) were synthesized as described previously.^{19,20} Double-distilled deionized water obtained from a Milli-Q Academic water purification system with ≥ 18 M Ω cm resistivity was used throughout the investigation.

Characterizations

The ¹H-NMR spectrum of purified ionic liquid surfactant was recorded on a Bruker DRX 400 MHz nuclear magnetic resonance instrument at 30°C, with CDCl₃ as solvent and tetramethylsilane as internal standard. The Fourier Transform infrared (FTIR) spectra of the purified ionic liquid surfactants were recorded on a Bruker-Tensor 27 FTIR spectrophotometer (KBr coating).

Determining the Solution Surface Tension by Drop Volume Method

Using secondary distilled water as solvent, a series of different aqueous solution concentrations of $[PPC_nim][Cl]$ (n = 8, 10, 12) and $[APC_nim][Cl]$ (n = 8, 10, 12) were prepared. After allowing the solutions to stand for 2 h, the solution surface tensions were determined at different temperatures using the drop

volume method.²¹ Drawing the curve of surface tensions and concentration logarithmic of surfactants (curve of γ -lgc), the critical micelle concentration (cmc) values were obtained by each turning point of curve. The surface tension using drop volume method was calculated via Formula (1).

$$\gamma = FV\rho g/R \ \left(\mathrm{mN}\cdot\mathrm{m}^{-1}\right) \tag{1}$$

where V was the volume of liquid droplets, ρ was the density of liquid, g was the acceleration of gravity (980.7 cm m⁻²), R was the radius of dripper. F was the correction coefficient, introducing to correct the deformation and some residual in the process of dripping of liquid droplets, F was function of V/R³ after measured by experiments, regardless of surface tension, density, viscosity of testing liquids, and dropper materials. F can be obtained through the look-up table.

The Formula (1) was only function of *V* because of ρ , *g*, and *R* were known. Thus, the measurement procedures were as follows.

- a. Determining the volume of droplets.
- b. Choosing appropriate F value through calculating V/R^3 .
- c. Calculating surface tension using Formula (1).

RESULTS AND DISCUSSION

Characterization of Synthetic Products

The structures of ionic liquid surfactants $[PPC_nim][Cl]$ and $[APC_nim][Cl]$ were characterized by FTIR and ¹H-NMR. The $[APC_{10}im][Cl]$ FTIR spectrum is shown in Figure 1. The C=C absorption peak was at 1636.6 cm⁻¹, the C=O absorption peak at 1722.2 cm⁻¹, the imidazole ring absorption peak at 1563.8 cm⁻¹, the C–N of imidazole ring absorption peaks at 1466.7 cm⁻¹, the C–H of imidazole ring absorption peaks at 1166.1 and 1192.0 cm⁻¹, and the C–H of long alkyl chain absorption peaks at 3385.2 cm⁻¹ appeared because ionic liquid has a strong hygroscopicity.



Figure 2. ¹H-NMR (400 MHz) spectrum of [APC₁₀im][Cl] in CDCl₃.

The ¹H-NMR spectrum of $[APC_{10}im][Cl]$ in CDCl₃ was shown in Figure 2. ¹H-NMR (CDCl₃, 400 MHz) δ (ppm): 0.87 ppm to 0.89 ppm (t, 3H, H_a), 1.19 ppm to 1.33 ppm (m, 14H, H_b), 1.90 ppm to 1.92 ppm (m, 2H, H_c), 2.38 ppm to 2.44 ppm (m, 2H, H_f), 4.25 ppm to 4.38 ppm (m, 4H, H_d, and H_e), 4.52 ppm to 4.56 ppm (t, 2H, H_g), 5.87 ppm to 5.90 ppm (d, 1H, H_i), 6.08 ppm to 6.15 ppm (t, 1H, H_h), 6.42 ppm to 6.46 ppm (d, 1H, H_j), 7.22 ppm to 7.27 ppm (d, 2H, H_m, and H_n), and 7.37 ppm (s, 1H, H_k).

Surface Activities of [PPC_nim][Cl] and [APC_nim][Cl]

The surface tensions of the ionic liquid surfactant series [APC- $_n$ im][Cl] (n = 8, 10, 12) and [PPC $_n$ im][Cl] (n = 8, 10, 12) were determined at 25°C. The relationships between the surface tension (γ) and logarithm of concentration (lgc) are shown in Figures 3 and 4. The abscissa and ordinate of the intersection point of the two tangent lines of the γ -lgc curve (Figure 3) were cmc and lowest surface tension (γ_{cmc}), respectively. The results by comparing with established imidazolium ionic liquid surfactants were shown in Table I.



Figure 3. γ -lgc curves of [APC_nim][Cl] (n = 8, 10, 12) in aqueous solution at 25°C. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

As shown in Table I, the four series of surfactants cmc value decreased as the length of carbon chain increased. Gemini imidazolium ionic liquid surfactants cmc value were lower than imidazolium ionic liquid surfactants, which was due to the electrostatic interaction of two ion head groups in Gemini imidazolium ionic liquid surfactant molecules. This led to the distance of two ion head groups in Gemini imidazolium ionic liquid surfactant molecules was narrower than the distance of polar groups in imidazolium ionic liquid surfactant molecules. Thus, the hydrophobic chains were arranged closely, and the oblique arrangement and bent assemble of hydrophobic chains were reduced. The surfactant molecules were arranged vertically at the gas-liquid interface and the surface tension was decreased effectively. [PPC_nim][Cl] and [APC_nim][Cl] ionic liquid surfactants cmc value were lower than imidazolium ionic liquid surfactants which was due to an hydrophobic group of acrylic acid propyl base that was added into the [PPCnim][Cl] and [APC-_nim][Cl] ionic liquid surfactant molecules. So that the hydrophobicity of the molecules and the arrangement of the molecules at the gas-liquid interface increased. Therefore, the surface tension was decreased. Gemini imidazolium ionoc liquid surfactants, [PPC_nim][Cl] and [APC_nim][Cl] ionic liquid surfactants cmc value were in the same magnitude maybe the acrylic acid propyl base was added into the [PPC_nim][Cl] and [APC-"im][Cl] ionic liquid surfactant molecules. The interaction of the two-tails in the [PPC_nim][Cl] and [APC_nim][Cl] ionic liquid surfactant molecules increased. Thereby, the hydrophobic chains were arranged tightly, and the surface tension was decreased. The potential advantages of the [PPC_nim][Cl] and [APC_nim][Cl] ionic liquid surfactant were their polymerizability. They could be copolymerized with acrylamide. The copolymers may be used as a good flocculant and could be applied at water treatment, etc.

The cmc of [PPC_nim][Cl] in Aqueous Solution

The surface tensions of $[PPC_nim][Cl]$ (n = 10, 12, 14) in aqueous solution at different temperatures were determined. The γ -lgc curves of $[PPC_{14}im][Cl]$ are shown in Figure 5



Figure 4. γ -lgc curves of [PPC_nim][Cl] (n = 8, 10, 12) in aqueous solution at 25°C. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

	[APC _n im][CI]		[PPC _n im][CI]		$[C_n-4-C_n im]Cl_2$		[C _n mim][Cl]					
n	8	10	12	8	10	12	8ª	10 ^a	12ª	8 ^b	10 ^b	12°
γcmc (mN⋅m ⁻¹)	28.36	27.33	25.31	28.24	27.37	24.95	35.0	35.2	35.7	32.2	33.7	42.0
cmc (mmol·L ⁻¹)	18.4	0.0622	0.0322	20.9	0.0697	0.0216	12.9	4.5	0.72	190	45	13.2

Table I. Surface Property Parameters of $[PPC_nim][Cl]$ and $[APC_nim][Cl]$ (n = 8, 10, 12)

^aReported in Ref. 22.

^bReported in Ref. 23.

°Reported in Ref. 12.

([PPC₁₀im][Cl] and [PPC₁₂im][Cl] γ -lgc curves show similar trends). The cmc values of [PPC_nim][Cl] at different temperatures are shown in Table II.

Comparing the data in Table II, when the temperature increased within the experimental temperatures, the cmc value initially decreased and then increased thereafter for all [PPC_nim][Cl]. At low temperature, increasing the temperature weakened the hydration of hydrophilic groups and enhanced the dehydration of hydrophobic chains, which were beneficial to micelle formation. When the temperature was higher, the hydrophobic interaction weakened because of the aggravation of molecular thermodynamic movement.²¹⁻²⁴ Increasing the temperature would separate the ionic liquid surfactant micelles, thus forming a looser aggregation structure,²⁵ which is not beneficial to micelle formation. At a specific temperature, as the length of the hydrophobic alkyl chain increased, the cmc value of the ionic liquid surfactant [PPC_nim][Cl] in aqueous solution decreased, indicating that increasing the length of the alkyl chain could improve micellization ability.

Thermodynamic Parameters of [PPC_nim][Cl] Micellization in Aqueous Solution

The ln $X_{cmc} - T$ curves of the [PPC_nim][Cl] ionic liquid surfactant series were obtained from the data in Table II (X_{cmc} was the molality of the surfactant in aqueous solution), and the curves are shown in Figure 6.



Figure 5. γ -lgc curves of [PPC₁₄im][Cl] in aqueous solution at different temperatures. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

The gradient of $\ln X_{\rm cmc} - T$ curves was obtained at each temperature from Figure 6 (cmc was the molality). The parameters of the Gibbs micellization free energy change ΔG_m^0 , micellization enthalpy change ΔH_m^0 , and micellization entropy change ΔS_m^0 were calculated using Formulas (1)–(³). The [PPC₁₄im][Cl] thermodynamic data are shown in Table III ([PPC₁₀im][Cl] and [PPC₁₂im][Cl] show similar thermodynamic data).

$$\Delta G_m^0 = 2RT \ln(cmc) \tag{2}$$

$$\Delta H_m^0 = -2RT^2 \left(\frac{\partial \ln(cmc)}{\partial T}\right) \tag{3}$$

$$\Delta S_m^0 = \left(\Delta H_m^0 - \Delta G_m^0\right) / T \tag{4}$$

At different temperatures, the ΔG_m^0 values were always negative because micelle formation was a spontaneous process. The ΔH_m^0 values were more variable (positive or negative), indicating that micelle formation was an endothermic process at low temperatures and exothermic at high temperatures. This finding is attributed to the loss of translational energy of the single ion. The heat released by the interaction between C-H bonds was less than the heat required to damage the iceberg structure during the micelle formation process at low temperatures (the opposite is true for high temperatures). The ΔS_m^0 values were always positive because of the disruption of the iceberg structure around the long alkyl chain of the surfactants during the micelle formation process. Additional water molecules became disorganized, and the entropy of the system increased. The $-T\Delta S_m^0$ values were obviously lesser than the ΔH_m^0 values. The ΔG_m^0 values were contributed by the $-T\Delta S_m^0$. Therefore, the micellization process is an entropy-driven process. During a temperature increase, although ΔG_m^0 , ΔH_m^0 , and ΔS_m^0 value all

Table II. Critical Micelle Concentration (cmc) of the [PPC_nim][Cl] Series

		cmc (mol·L ⁻¹)	
T (K)	[PPC10im][CI]	[PPC ₁₂ im][CI]	[PPC ₁₄ im][CI]
288.15	7.79×10^{-4}	2.58×10^{-4}	7.76×10^{-5}
298.15	6.97×10^{-4}	2.16×10^{-4}	7.08×10^{-5}
308.15	6.06×10^{-4}	1.72×10^{-4}	6.67×10^{-5}
318.15	5.77×10^{-4}	1.64×10^{-4}	6.38×10^{-5}
328.15	$6.03 imes 10^{-4}$	$1.73 imes 10^{-4}$	6.55×10^{-5}



Figure 6. $\ln X_{cmc} - T$ curves of the [PPC_nim][Cl] series in aqueous solution. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

decreased, $-T\Delta S_m^0$ value increased. Therefore, the entropic contribution to the Gibbs free energy change decreased, whereas the enthalpic contribution increased.

The Enthalpy–Entropy Compensation Phenomenon of [PPC,,im][Cl] Micellization in Aqueous Solution

The compensation phenomenon between enthalpy change (ΔH_m^0) and entropy change (ΔS_m^0) of surfactant micellization can be described via Formula (4):

$$\Delta H_m^0 = \Delta H_m^* + T_c \Delta S_m^0 \tag{5}$$

According to Sugihara and Hisatomi²⁶ and Lumry and Rajender,²⁷ in the compensation phenomenon, the micellization of a surfactant can be described by two processes: (a) the "desolvation" part, meaning the dehydration of the hydrocarbon chain and (b) the "chemical" part, indicating the aggregation of the hydrocarbon chain to form a micelle. The slope of the compensation plot, T_o , named the compensation temperature, can be explained as a characteristic of solute–solvent interactions, considered as a measure of the "desolvation" part. The intercept of that plot, ΔH_m^* , characterizes the solute–solute interactions and is considered to represent the "chemical" part belonging to the $\Delta S_m^0 = 0$. The smaller the ΔH_m^* value, the higher the stability of the micelle structure will be.²⁸

Table III. Thermodynamic Parameters of $[{\rm PPC}_{14}{\rm im}][{\rm Cl}]$ Micellization in Aqueous Solution

T (K)	ΔG_m^0 (kJ·mol ⁻¹)	ΔH_m^0 (kJ·mol $^{-1}$)	ΔS^0_m (kJ [mol·K] $^{-1}$)	$-T\Delta S_m^0$ (kJ·mol ⁻¹)
288.15	-64.59	12.72	0.27	-77.31
298.15	-67.29	11.23	0.26	-78.52
308.15	-69.85	8.18	0.25	-78.03
318.15	-72.35	1.55	0.23	-73.90
328.15	-74.49	-4.54	0.21	-69.95



Figure 7. $\Delta H_m^0 - \Delta S_m^0$ plots of [PPC_nim][Cl] micellization in aqueous solution. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

The $\Delta H_m^0 - \Delta S_m^0$ curves of the micellization process of the [PPC_nim][Cl] ionic liquid surfactant series in aqueous solution are shown in Figure 7. The curves showed a good linear relationship and fitted well with Formula (4). An enthalpy–entropy compensation phenomenon is thus indicated in the micellization process of surfactants in aqueous solution. The T_c and ΔH_m^* values were obtained from Figure 7, and the results are shown in Table IV.

Table IV shows that the compensation temperature T_c was (314) \pm 1) K and almost remained unchanged with increasing alkyl chain length. This meaning the "desolvation" part of $[PPC_nim][Cl]$ may follow the same mechanism. But whether the T_c can be used to deduce a process that follows the same mechanism is controversial. Ranatunga et al.²⁹ suggested that the same compensation temperatures just indicate the relative contributions of ΔH_m^0 and ΔS_m^0 to the free energy are equal and cannot be adopted as evidence. Therefore, further studies are necessary to resolve this problem. The ΔH_m^* values decreased with increasing alkyl chain length, indicating that with increasing alkyl chain length, both the micelle formation ability and micelle stability improved. This result coincided with the influence of controlling the [PPC_nim][Cl] micellization process on molecular structure at a particular temperature. That is the hydrophobic interaction of surfactant molecules increased with increasing the alkyl chain length, and the aggregation structure was more easier to be formed. Thus, the micelle formation ability and micelle stability both improved.

Table IV. Fitting Results for $\Delta H_m^0 - \Delta S_m^0$ Plots of the [PPC_nim][Cl] Series

	R^2	$T_c(K)$	ΔH_m^* (kJ·mol $^{-1}$)
[PPC ₁₀ im][CI]	0.99994	315.56	-60.25
[PPC12im][CI]	0.99990	314.43	-66.65
[PPC14im][CI]	0.99986	313.39	-71.29



CONCLUSIONS

In this article, two series of cationic polymerizable ionic liquid surfactants were first synthesized. The drop volume method was used to determine the surface tensions of novel polymerizable ionic liquid surfactants 1-acryloyloxypropyl-3-alkylimidazolium chloride [APC_nim][Cl] and 1-propionyloxypropyl-3-alkylimidazolium chloride [PPC_nim][Cl] (n = 8, 10, 12) in aqueous solution at different temperatures. Their surface properties were also compared. The results showed that (a) double bonds in molecular structure had little effect on surface activity of polymerizable ionic liquid surfactant $[APC_n im][Cl]$. (b) The micellization process of surfactants [PPC_nim][Cl] in aqueous solution was a spontaneous process. The micellization process is an entropy-driven process. Surfactant with determined structure, the micelle formation ability increased at first, then decreased with the increment of temperature. And the entropic contribution to the Gibbs free energy change decreased, whereas the enthalpic contribution increased. (c) The enthalpy-entropy compensation phenomenon existed in the micellization process of surfactants $[PPC_n im][Cl]$ in aqueous solution. And the compensation temperature T_c was (314 \pm 1) K and almost remained unchanged with increasing alkyl chain length. With the alkyl chain length increasing, the micelle formation ability and micelle stability both being improved. It is important for further research on aggregation behavior and practical application of these ionic liquid surfactants in aqueous solution. These surfactants exhibiting the characteristics of ionic liquid and surfactant, combining surfactant and ionic liquid. This provided a new direction for research on polymerization of new functional monomers and expanded the application of ionic liquid surfactant in the polymer field.

ACKNOWLEDGMENTS

This work was financially supported by National Science and Technology fund major project of P.R.C. (2008ZX05011), Heilongjiang Province Natural Science Foundation (ZJG0507), Youth Fund of Heilongjiang Province Natural Science Foundation (QC08C33), and Graduate funding innovative research in Heilongjiang Province (YJSCX2008-044HLJ).

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