

Synthesis and Optical Properties of 1-Alkyl-3-Methylimidazolium Lauryl Sulfate Ionic Liquids

Judy M. Obliosca · Susan D. Arco · Michael H. Huang

Received: 13 December 2006 / Accepted: 7 August 2007 / Published online: 21 August 2007
© Springer Science + Business Media, LLC 2007

Abstract We report the synthesis of a new series of imidazolium-based halogen-free ionic liquids 1-alkyl-3-methylimidazolium lauryl sulfates. By reacting 1-methylimidazole (MIM) with butyl, hexyl, octyl, and decyl bromides and exchanging bromide ion with lauryl sulfate anion, a series of ionic liquids [RMIM][C₁₂H₂₅OSO₃] were produced. The high purity of these ionic liquids was verified with ¹H-NMR, ¹³C-NMR, FT-IR and mass spectrometry (MS), demonstrating the effectiveness of this synthetic approach. Solubility test of these ionic liquids showed that they are soluble in most organic solvents except nonpolar solvents such as hexane and cyclohexane. The optical properties of [BMIM]Br and [BMIM][C₁₂H₂₅OSO₃], where B refers to butyl, were examined. Both ionic liquids absorbed light in the UV region, yet essentially no absorption was recorded beyond 450 nm. Furthermore, both ionic liquids showed excitation wavelength-dependent fluorescence behavior. As an example, with an excitation wavelength of 360 nm, [BMIM][C₁₂H₂₅OSO₃] showed an emission band maximum at 447 nm. Increasing the excitation wavelength to 440 nm, the emission band maximum was shifted to ~500 nm.

Keywords Ionic liquids · Excitation wavelength-dependent fluorescence

Introduction

Ionic liquids (ILs) are a class of solvents that are receiving a great deal of attention for their wide range of applications [1]. As ‘designer solvents’, ILs are capable of dissolving almost anything including coal, plastics, metals and even rock. They are used as solvents for organic synthesis [1, 2], catalysis [2–4], extraction of gold nanorods [5], sulfur removal from fuels [6], and the synthesis of gold nanoparticles and sheets [7–9]. They have the potential to make ideal green solvents as they have negligible vapor pressure and do not evaporate into the atmosphere, making them more environmentally responsible materials than traditional organic solvents.

Typical ILs consist of *N,N'*-dialkylimidazolium cations, and a wide range of halide-based anions such as [AlCl₄][−], [PF₆][−], [BF₄][−] and [CF₃SO₃][−] [10, 11]. However, the presence of halides may cause serious concerns if the hydrolysis stability of the anion is poor (e.g., for [AlCl₄][−] and [PF₆][−]) or if thermal treatment of the utilized ILs is desired [11]. In view of the emerging importance of benign and environmental friendly ILs, this study explores the synthesis of a series of ILs based on 1-alkyl-3-methylimidazolium cations with lauryl sulfate as the halogen-free anion. Sodium lauryl sulfate (SLS) is of great advantage because unlike other alkyl sulfates which are made from synthetic alcohol, SLS is a naturally derived surfactant made from the whole natural coconut. In this study, ILs containing 1-alkyl-3-methylimidazolium cations were synthesized by reacting 1-methylimidazole with alkyl bromides ranging from C₄ to C₁₀ hydrocarbon chain groups and exchanging bromide ions with sodium lauryl sulfate to afford the viscous to solid products, [RMIM][C₁₂H₂₅OSO₃]. The formation of the products was verified with ¹H-NMR, ¹³C-NMR, FT-IR and MS. All the products were found to show very high purity, demonstrating the success of the

J. M. Obliosca · M. H. Huang (✉)
Department of Chemistry, National Tsing Hua University,
Hsinchu 30013, Taiwan
e-mail: hyhuang@mx.nthu.edu.tw

J. M. Obliosca · S. D. Arco
Institute of Chemistry, University of the Philippines, Diliman,
Quezon City, Philippines

synthetic procedure adopted. With the consideration of the applications of these SLS-based ILs in the synthesis and phase transfer of nanoscale materials, the solubility of the synthesized ILs in various organic solvents was examined. In addition, absorption and photoluminescence spectra of the [BMIM]Br and [BMIM][C₁₂H₂₅OSO₃] ILs were taken to study their optical properties. Both ILs were found to exhibit an excitation wavelength-dependent fluorescence behavior.

Experimental

Synthesis of ionic liquids

A range of ILs containing 1-alkyl-3-methylimidazolium cations was synthesized. In the first step, 1-methylimidazole was used as the starting material and reacted with alkyl bromides containing C₄ to C₁₀ hydrocarbon chain groups. Sodium lauryl sulfate was then added to replace the bromide ions with lauryl sulfate anions forming the desired ILs.

First, 1 mol of 1-methylimidazole (79.7 mL) and 1 mol of 1-bromoalkane (107.4 mL for 1-bromobutane, all purchased from Fluka) were placed in a two-necked round-bottom flask, stirred thoroughly and heated to 70°C for 48 h under N₂. The resulting viscous liquid was cooled to room temperature, washed several times with small portions of ethyl acetate (purchased from Aldrich) by shaking the mixture using a separatory funnel and then draining away the solvent to remove unreacted starting material (that is, excess of 1-bromoalkane), and then dried under vacuum for 5–7 h.

For the synthesis of 1-alkyl-3-methylimidazolium lauryl sulfates, 48.4 mmol of the freshly synthesized and dried [RMIM]Br (10.6 g for [BMIM]Br) and 43.6 mmol (12.6 g) of sodium lauryl sulfate CH₃(CH₂)₁₁OSO₃Na (purchased

from Aldrich) were dissolved in 20 mL of hot water (60°C). The water was slowly removed under vacuum at 70–80°C and a white solid precipitated. By using a separatory funnel, 45 mL of CH₂Cl₂ (purchased from Fluka) was added to extract the product from the mixture. The ionic liquid was in the lower layer, while NaBr was in the upper layer. The clear but slightly yellow viscous extract was then washed several times with water and the white solid (accounted for NaBr as by-product) was separated out. The washing was repeated until it was bromide-free; two to three drops of 0.1 M AgNO₃ were added to the washings to confirm the presence/absence of bromide ions. The extract was distilled to get rid of the CH₂Cl₂ solvent and finally dried under N₂ and vacuum for 5–7 h to afford the viscous gel product, [RMIM][C₁₂H₂₅OSO₃].

The solubility of the synthesized ionic liquids in various organic solvents having different dielectric constants has been determined. Approximately 0.1 g or 0.2 mL (two to three drops) of ionic liquid in a vial was added to 1 mL of organic solvents listed in Table 1. The mixture was then shaken vigorously after addition.

Five to ten mg of ionic liquid was placed in an aluminum hermetic pan, covered and measured by a differential scanning calorimeter (DSC) from 30 to 150°C at a heating rate of 10°C/min. The DSC was purged with N₂ at a flow rate of 50 mL/min.

Instrumentation

¹H-NMR and ¹³C-NMR spectra taken in d₆-DMSO solvent were recorded on a Varian Mercury 400 and 500 MHz spectrometer, respectively. FT-IR spectra were measured on a Perkin Elmer Spectrum RX I spectrometer. Mass spectra were recorded on a liquid chromatograph mass spectrometer (JEOL JMS-SX102A). Photoluminescence spectra

Table 1 Solubility of the synthesized ionic liquids in various organic solvents

Solvent	ϵ	[BMIM] Br (1a)	[HMIM]Br (1b)	[OMIM] Br (1c)	[DMIM] Br (1d)	BLS (2a)	HLS (2b)	OLS (2c)	DLS (2d)
Water	80.0	✓	✓	✓	✓	✓	✓	o	×
Methanol	32.6	✓	✓	✓	✓	✓	✓	✓	✓
Ethanol	24.3	✓	✓	✓	✓	✓	✓	✓	✓
Acetone	20.7	✓	✓	✓	✓	o	✓	✓	✓
CH ₂ Cl ₂	9.1	✓	✓	✓	✓	✓	✓	✓	✓
THF	7.6	×	×	×	✓	✓	✓	✓	✓
Ethyl acetate	6.0	×	×	×	×	o	✓	✓	×
CS ₂	2.6	×	×	×	×	×	✓	✓	✓
Toluene	2.4	×	×	×	×	o	✓	✓	✓
Cyclohexane	2.0	×	×	×	×	×	o	o	×
Hexane	2.0	×	×	×	×	×	o	×	×

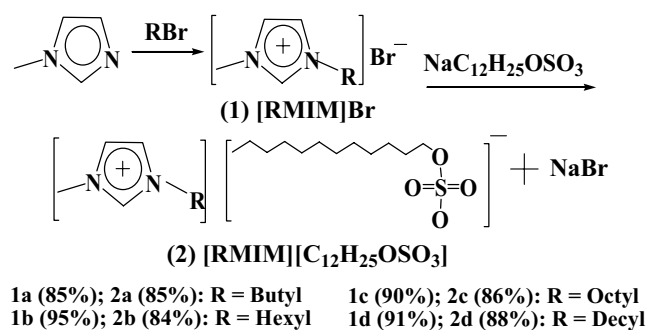
✓ Soluble, × insoluble, o partially soluble

were taken with excitation wavelengths ranging from 300 to 440 nm using a fluorescence spectrophotometer (Hitachi F-4500). UV-vis absorption spectra were recorded on a spectrophotometer (JASCO V-570). DSC curves were collected using a differential scanning calorimeter (SDC Q10 V9.0 Build 275).

Results and discussion

The preparation of 1-alkyl-3-methylimidazolium ILs with bromide ions and the desired ILs with lauryl sulfate anions follows the general reaction presented in Fig. 1. This simple synthesis was carried out under nitrogen atmosphere and dried under vacuum since 1-methylimidazole is a hygroscopic compound. The products are white solid compounds for ILs **2a**, **2c**, and **2d**, and colorless gel for IL **2b** (see Fig. 1). Despite their solid form at room temperature, these ILs can turn into the liquid state at slightly higher temperatures. DSC measurements showed that ILs **2a**, **2c**, and **2d** exhibit an endothermic peak due to the melting of the ILs at about 48, 39, and 44–48°C, respectively. Above these temperatures, these ILs bearing the lauryl sulfate anion can serve as solvents or media for various reactions. IL **2b**, however, displayed a more gradual endothermic behavior at higher temperatures and then a sharp endothermic peak at 92°C, possibly as a result of its gel-like state at room temperature.

This one-pot synthetic route allowed us to obtain the imidazolium-based halogen-free ILs in good yields and high purity. The purity of the products was identified based on several spectroscopic data. Results of ¹H-NMR and ¹³C-NMR spectra indicated high purity of these ionic liquids without unexpected signals from unreacted starting



2a **2b** **2c** **2d**

Fig. 1 General schematic diagram for the synthesis of ILs and their percent yields. Images show the physical appearances of the products

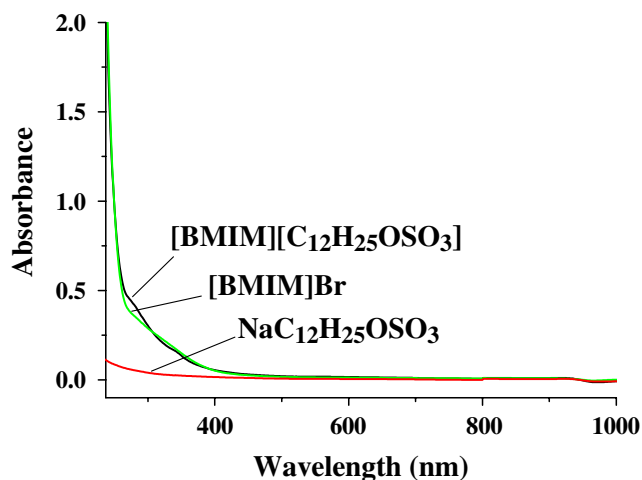


Fig. 2 Absorption spectra of 0.35 M [BMIM]Br (**1a**), sodium lauryl sulfate, and [BMIM][C₁₂H₂₅OSO₃] (**2a**) in water

materials. Careful examinations of the FT-IR results showed all functional groups expected for the desired ILs. MS analysis revealed all the mass units of the ionic liquids intact. These pure ILs showed 100% cation and anion signals under MS-ES⁺ (electron spray) and MS-ES⁻, respectively.

With the interest on the possible use of these ILs for the synthesis and phase transfer of nanoscale materials, the solubility (or miscibility) of the ILs in various organic solvents having different dielectric constants (ϵ) was determined (Table 1). The solubility of the ILs in bromide form increases in polar protic and aprotic solvents having high dielectric constants (that is, $\epsilon=20.7$ for acetone up to $\epsilon=80$ for water). Organic solvents with dielectric constants lower than 10 are generally not soluble except CH₂Cl₂ because of the polar nature of these ILs. Exchanging bromide with lauryl sulfate ion resulted in possibly less polar products. For this reason, these ILs are generally soluble in solvents having dielectric constant as low as that of toluene ($\epsilon=2.4$). These ILs also exhibit good solubility in polar solvents such as alcohol (that is, CH₃CH₂OH and CH₃OH) due to the presence of the alkyl groups on the alcohols which can readily interact with the long alkyl chain of sulfate via van der Waals attraction. An opposite effect was observed with water which does not have any alkyl group and solubility is poor for ILs **2c** and **2d**.

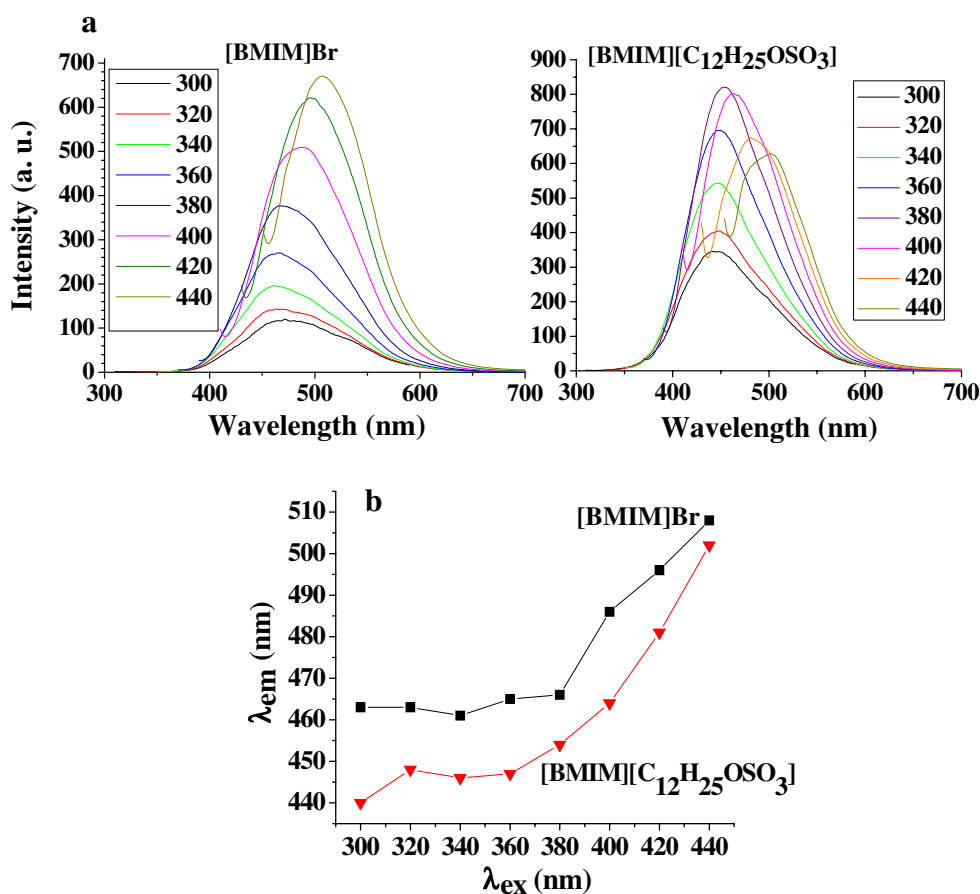
In the quest of understanding the optical properties of the products, the absorbance and fluorescence behavior of the ILs in bromide and sulfate forms were examined. The UV-vis absorption spectra of 0.35 M [BMIM]Br, [BMIM][C₁₂H₂₅OSO₃], and lauryl sulfate in water were measured and shown in Fig. 2. A strong absorption in the deep UV region (230–380 nm) with decreasing trend towards the visible part was observed for both ILs. The absorption tail is considerably long and extends beyond 380 nm. Since both compounds have almost identical absorption pro-

file, the absorption center should be the 1-alkyl-3-methylimidazolium cation. This significant absorption prompted us to further examine the photoluminescence spectra of [BMIM]Br and [BMIM][C₁₂H₂₅OSO₃]. With an excitation wavelength of 300 nm, [BMIM]Br and [BMIM][C₁₂H₂₅OSO₃] showed emission maxima at 463 and 440 nm, respectively. When subjected to an increasing excitation wavelength, both ILs exhibited fluorescence maxima which started to shift progressively toward the red region with increasing intensity for [BMIM]Br and increasing trend for [BMIM][C₁₂H₂₅OSO₃] up to an excitation wavelength of 380 nm (see Fig. 3a). Thus, there is an excitation wavelength-dependent fluorescence behavior for both ILs. The progressive increase and then decrease in the fluorescence intensity of [BMIM][C₁₂H₂₅OSO₃] is similar to those reported for [BMIM][BF₄] and other ionic liquids examined [12]. At longer excitation wavelengths beyond 440 nm, a decreased fluorescence intensity for [BMIM]Br should be expected, as light absorption by the ionic liquid becomes less efficient at such wavelengths. A plot of the shifts in the emission maxima with different excitation wavelengths (see Fig. 3b) shows that the emission maxima do not differ much with excitation light in the deep UV region (that is, relatively flat profile in the 300–360 nm

region) but has great change towards the blue light region (ascending trend, 380–440 nm).

Results show that by replacing the IL in bromide form with lauryl sulfate anion, significant absorption in the UV region and excitation wavelength-dependent fluorescence characteristics can still be observed. Hence, these unique optical properties are not due to the anion effect but are attributed to the imidazole moiety of the ILs. These findings are consistent with a recent study done by Paul et al. [12]. They examined the imidazolium cation with halogen-based ILs in PF₆⁻ and Cl⁻ forms and found significant electronic absorption in the UV region and dramatic excitation-wavelength dependent photoluminescence. The excitation-wavelength dependent fluorescence behavior has been suggested as due to the presence of different associated structures of the ionic liquids resulting from the spatial correlations of the cation–anion and cation–cation pairs [12]. By varying the excitation wavelengths, the various associated structures of the ionic liquids become excited and emit light at slightly different wavelengths. The high viscosity also contributes to the observed effect. Our work further illustrates that the emission intensities of the ILs in the deep UV region are lower than that in the visible region (see Fig. 3a). The emission

Fig. 3 **a** Excitation wavelength-dependent emission behavior of the ILs [BMIM]Br (**1a**) and [BMIM][C₁₂H₂₅OSO₃] (**2a**) (λ_{ex} =300 to 440 nm). **b** A plot of the shifts in the emission maxima of the ILs [BMIM]Br and [BMIM][C₁₂H₂₅OSO₃] excited at different wavelengths



intensity of these ILs is in general relatively weak, and should not interfere with the fluorescence of dissolved organic molecules having sufficiently strong fluorescence intensities. Fluorescent probes have recently been used to study the local solvent environment of ILs [13, 14]. Thus, the emission of the dissolved fluoroprobes should not present a problem with UV light absorption by the ILs.

Conclusion

In summary, we have successfully synthesized a series of 1-alkyl-3-methylimidazolium lauryl sulfate ILs. All the ILs have been carefully characterized with $^1\text{H-NMR}$, $^{13}\text{C-NMR}$, FT-IR and MS analysis. The results indicated that the synthetic procedure yields highly pure products. Compared to the insoluble nature of the ILs with bromide ion in nonpolar solvents, the ILs with lauryl sulfate anion exhibit good solubility in both polar and nonpolar solvents. The optical properties of [BMIM]Br and [BMIM][C₁₂H₂₅OSO₃] were examined. An interesting excitation wavelength-dependent emission behavior was observed for both ILs, suggesting that such optical property is typical of ILs bearing the 1,3-dialkylimidazole moiety. Because 1-alkyl-3-methylimidazolium lauryl sulfate ILs contain the lauryl sulfate (or dodecyl sulfate) anion, which is a common surfactant, this series of ILs may find application in the synthesis of nanoscale materials as the capping agent is already incorporated into the ILs.

Acknowledgments We thank National Science Council of Taiwan (Grant 93-2113-M-007-049) for the funding, Hsu-Kai Chang and Hao-Chun Li for the assistance in the NMR measurement, Marlon T. Conato for the assistance in the DSC analysis, and Dr. Nelson R. Villarante for his valuable comments.

Appendix

Supporting Information

Synthesis and Optical Properties of 1-Alkyl-3-Methylimidazolium Lauryl Sulfate Ionic Liquids

Judy M. Obliosca, Susan D. Arco and Michael H. Huang*

$^1\text{H-NMR}$, $^{13}\text{C-NMR}$, FT-IR, and MS data for all the ionic liquids synthesized in this study.

1a: 1-Butyl-3-Methylimidazolium Bromide

$^1\text{H-NMR}$ (500 MHz, d₆-DMSO): δ 0.766 (t, $J=7.5$ Hz, 3 H), 1.148 (sxt, $J=7.5$ Hz, 2H), 1.700 (qnt, $J=7.0$ Hz, 2 H), 3.882 (s, 3 H), 4.211 (t, $J=7.5$ Hz, 2H), 7.881 (d, $J=1.5$ Hz, 1H), 7.984 (d, $J=2.0$ Hz, 1H), 9.561 (s, 1H); $^{13}\text{C-NMR}$ (500 MHz, d₆-DMSO): δ 13.111, 18.549, 31.286,

35.739, 48.211, 122.090, 123.279, 136.398; IR (neat) ν_{max} : 3,460, 3,153, 3,091, 2,960, 2,920, 2,853, 1,637, 1,575, 1,472, 1,382, 1,171 cm⁻¹; ESI-MS: (ESI⁺) m/e 139.17 {[C₈H₁₅N₂]⁺}, (ESI⁻) 78.82 (Br⁻), 80.81 (Br⁻).

1b: 1-Hexyl-3-Methylimidazolium Bromide

$^1\text{H-NMR}$ (500 MHz, d₆-DMSO): δ 0.704 (t, $J=6.5$ Hz, 3H), 1.114 (m, 6H), 1.711 (qnt, $J=6.5$ Hz, 2H), 3.888 (s, 3H), 4.212 (t, $J=7.5$ Hz, 2H), 7.911 (d, $J=1.5$ Hz, 1H), 8.012 (s, $J=1.5$ Hz, 1H), 9.613 (s, 1H); $^{13}\text{C-NMR}$ (500 MHz, d₆-DMSO): δ 13.555, 21.680, 24.909, 29.308, 30.364, 35.704, 48.432, 122.055, 123.226, 136.389; IR (neat) ν_{max} : 3,438, 3,148, 3,097, 2,961, 2,926, 2,858, 1,637, 1,575, 1,467, 1,382, 1,166 cm⁻¹; ESI-MS: (ESI⁺) m/e 167.23 {[C₁₀H₁₉N₂]⁺}, (ESI⁻) 78.82 (Br⁻), 80.82 (Br⁻).

1c: 1-Octyl-3-Methylimidazolium Bromide

$^1\text{H-NMR}$ (500 MHz, d₆-DMSO): δ 0.728 (t, $J=6.5$ Hz, 3H), 1.125 (m, 10H), 1.725 (qnt, $J=6.5$ Hz, 2H), 3.889 (s, 3H), 4.215 (t, $J=7.0$ Hz, 2H), 7.906 (d, $J=1.5$ Hz, 1H), 7.998 (d, $J=1.5$ Hz, 1H), 9.607 (s, 1H); $^{13}\text{C-NMR}$ (500 MHz, d₆-DMSO): δ 13.635, 21.875, 25.317, 28.226, 28.350, 29.388, 31.002, 35.677, 48.441, 122.064, 123.252, 136.407; IR (neat) ν_{max} : 3,466, 3,148, 3,097, 2,955, 2,926, 2,858, 1,643, 1,575, 1,472, 1,382, 1,171 cm⁻¹; ESI-MS: (ESI⁺) m/e 195.29 {[C₁₂H₂₃N₂]⁺}, (ESI⁻) 78.81 (Br⁻), 80.81 (Br⁻).

1d: 1-Decyl-3-Methylimidazolium Bromide

$^1\text{H-NMR}$ (500 MHz, d₆-DMSO): δ 0.732 (t, $J=7.5$ Hz, 3H), 1.156 (m, 14H), 1.725 (qnt, $J=7.0$ Hz, 2H), 3.893 (s, 3H), 4.216 (t, $J=7.0$ Hz, 2H), 7.910 (d, $J=1.5$ Hz, 1H), 7.996 (d, $J=2.0$ Hz, 1H), 9.608 (s, 1H); $^{13}\text{C-NMR}$ (500 MHz, d₆-DMSO): δ 13.617, 21.928, 25.352, 28.324, 28.563, 28.758, 28.811, 29.423, 31.144, 35.686, 48.441, 122.055, 123.252, 136.416; IR (neat) ν_{max} : 3,432, 3,148, 3,103, 2,961, 2,926, 2,858, 1,671, 1,580, 1,467, 1,387, 1,183 cm⁻¹; ESI-MS: (ESI⁺) m/e 223.30 {[C₁₄H₂₇N₂]⁺}, (ESI⁻) 78.81 (Br⁻), 80.81 (Br⁻).

2a: 1-Butyl-3-Methylimidazolium Lauryl Sulfate = [BMIM][C₁₂H₂₅OSO₃] = BLS

$^1\text{H-NMR}$ (400 MHz, d₆-DMSO): δ 0.833 (t, $J=6.8$ Hz, 3H), 0.882 (t, $J=7.6$ Hz, 3H), 1.221 (m, $J=7.6$ Hz, 20H), 1.451 (m, $J=6.8$ μz , 2H), 1.752 (qnt, $J=6.8$ Hz, 2H), 3.692 (t, $J=6.0$ Hz, 2H), 3.845 (s, 3H), 4.158 (t, $J=7.2$ μHz , 2H), 7.707 (d, $J=1.6$ Hz, 1H), 7.776 (d, $J=1.2$ Hz, 1H), 9.120 (s, 1H); $^{13}\text{C-NMR}$ (500 MHz, d₆-DMSO): δ 13.226, 13.892, 18.770, 22.106, 25.538, 28.740, 28.811, 29.051 (3C), 29.086 (2C), 31.313, 31.402, 35.677, 48.468, 65.534, 122.268, 123.589, 136.575; IR (neat) ν_{max} : 3,460, 3,153, 3,091, 2,960, 2,920, 2,853, 1,637, 1,575, 1,472, 1,382, 1,274, 1,217, 1,171, 1,069, 990, 814, 762, 723 cm⁻¹; ESI-MS: (ESI⁺) m/e 139.2 {[C₈H₁₅N₂]⁺}, (ESI⁻) 265.13 {[C₁₂H₂₅OSO₃]⁻}.

2b: 1-Hexyl-3-Methylimidazolium Lauryl Sulfate = [HMIM][C₁₂H₂₅OSO₃]=HLS

$^1\text{H-NMR}$ (400 MHz, d_6 -DMSO): δ 0.839 (tt, $J=6.6$ Hz, 6H), 1.223 (m, $J=12.8$ Hz, 20H), 1.250 (m, $J=10.8$ Hz, 4H), 1.458 (qnt, $J=6.8$ Hz, 2H), 1.762 (qnt, $J=7.2$ Hz, 2H), 3.667 (t, $J=6.8$ Hz, 2H), 3.841 (s, 3H), 4.145 (t, $J=7.2$ Hz, 2H), 7.699 (d, $J=1.2$ Hz, 1H), 7.767 (d, $J=1.6$ Hz, 1H), 9.107 (s, 1H); $^{13}\text{C-NMR}$ (500 MHz, d_6 -DMSO): δ 13.803, 13.909, 21.902, 22.106, 25.175, 25.547, 28.740, 28.820, 29.060 (3C), 29.086 (2C), 29.397, 30.594, 31.322, 35.695, 48.743, 65.525, 122.268, 123.598, 136.549; IR (neat) ν_{max} : 3,438, 3,148, 3,097, 2,961, 2,926, 2,858, 1,637, 1,575, 1,467, 1,382, 1,222, 1,166, 1,069, 990, 808, 768, 728 cm^{-1} ; ESI-MS: (ESI $^+$) m/e 167.24 {[C $_{10}$ H $_{19}$ N $_2$] $^+$ }, (ESI $^-$) 265.19 {[C $_{12}$ H $_{25}$ OSO $_3$] $^-$ }.

2c: 1-Octyl-3-Methylimidazolium Lauryl Sulfate = [OMIM][C $_{12}$ H $_{25}$ OSO $_3$]=OLS

$^1\text{H-NMR}$ (400 MHz, d_6 -DMSO): δ 0.830 (tt, $J=7.0$ Hz, 6H), 1.217 (m, $J=12$ Hz, 20H), 1.250 (m, 8H), 1.458 (qnt, $J=6.0$ Hz, 2H), 1.780 (qnt, $J=7.2$ Hz, 2H), 3.677 (t, $J=7.2$ Hz, 2H), 3.847 (s, 3H), 4.150 (t, $J=7.2$ Hz, 2H), 7.709 (d, $J=1.6$ Hz, 1H), 7.777 (d, $J=1.6$ Hz, 1H), 9.138 (s, 1H); $^{13}\text{C-NMR}$ (500 MHz, d_6 -DMSO): δ 13.865 (2C), 22.088, 22.114, 25.556 (2C), 28.430, 28.563, 28.767, 28.856, 29.095 (3C), 29.113 (2C), 29.486, 31.233, 31.339, 35.677, 48.743, 65.552, 122.268, 123.572, 136.567; IR (neat) ν_{max} : 3,466, 3,148, 3,097, 2,955, 2,926, 2,858, 1,643, 1,575, 1,472, 1,382, 1,222, 1,171, 1,063, 995, 808, 723 cm^{-1} ; ESI-MS: (ESI $^+$) m/e 195.27 {[C $_{12}$ H $_{23}$ N $_2$] $^+$ }, (ESI $^-$) 265.19 {[C $_{12}$ H $_{25}$ OSO $_3$] $^-$ }.

2d: 1-Decyl-3-Methylimidazolium Lauryl Sulfate = [DMIM][C $_{12}$ H $_{25}$ OSO $_3$] = DLS

$^1\text{H-NMR}$ (500 MHz, d_6 -DMSO): δ 0.828 (t, $J=5.5$ Hz, 6H), 1.214 (m, 20H), 1.245 (m, 12H), 1.453 (m, $J=6.5$ Hz, 2H), 1.760 (qnt, $J=7.0$ Hz, 2H), 3.675 (t, $J=6.5$ Hz, 2H), 3.852 (s, 3H), 4.155 (t, $J=7.5$ Hz, 2H), 7.728 (d, $J=2.0$ Hz, 1H), 7.794 (s, $J=2.0$ Hz, 1H), 9.187 (s, 1H); $^{13}\text{C-NMR}$ (500 MHz, d_6 -DMSO): δ 13.839 (2C), 22.097 (2C), 25.529, 25.547, 28.457, 28.723, 28.749, 28.856, 28.891, 28.962, 29.060, 29.086 (2C), 29.104 (2C), 29.468, 31.313 (2C), 35.668, 48.698, 65.499, 122.241, 123.545, 136.549;

IR (neat) ν_{max} : 3,432, 3,148, 3,103, 2,961, 2,926, 2,858, 1,671, 1,580, 1,467, 1,387, 1,257, 1,217, 1,183, 1,058, 984, 870, 802, 723 cm^{-1} ; ESI-MS: (ESI $^+$) m/e 223.3 {[C $_{14}$ H $_{27}$ N $_2$] $^+$ }, (ESI $^-$) 265.13 {[C $_{12}$ H $_{25}$ OSO $_3$] $^-$ }.

References

- Holbrey JD, Seddon KR (1999) Ionic liquids. *Clean Prod Processes* 1:223–236
- Welton T (1999) Room-temperature ionic liquids. Solvents for synthesis and catalysis. *Chem Rev* 99(8):2071–2083
- Sheldon R (2001) Catalytic reactions in ionic liquids. *Chem Commun* 2399–2407
- Dupont J, de Souza RF, Suarez PAZ (2002) Ionic liquid (molten salt) phase organometallic catalysis. *Chem Rev* 102(10):3667–3692
- Wei G-T, Yang Z, Lee C-Y, Yang H-Y, Wang CRC (2004) Aqueous-organic phase transfer of gold nanoparticles and gold nanorods using an ionic liquid. *J Am Chem Soc* 126(16):5036–5037
- Zhang S, Zhang ZC (2002) Novel properties of ionic liquids in selective sulfur removal from fuels at room temperature. *Green Chem* 4:376–379
- Tatumi R, Fujihara H (2005) Remarkably stable gold nanoparticles functionalized with a zwitterionic liquid based on imidazolium sulfonate in a high concentration of aqueous electrolyte and ionic liquid. *Chem Commun* 83–85
- Itoh H, Naka K, Chujo Y (2004) Synthesis of gold nanoparticles modified with ionic liquid based on the imidazolium cation. *J Am Chem Soc* 126(10):3026–3027
- Li Z, Liu Z, Zhang J, Han B, Du J, Gao Y, Jiang T (2005) Synthesis of single-crystal gold nanosheets of large size in ionic liquids. *J Phys Chem B* 109(30):14445–14448
- Earle MJ, Seddon KR (2000) Ionic liquids. Green solvents for the future. *Pure Appl Chem* 72(7):1391–1398
- Wasserscheid P, van Hal R, Bösmann A (2002) 1-*n*-Butyl-3-methylimidazolium ([bmim]) octylsulfate—an even ‘greener’ ionic liquid. *Green Chem* 4:400–404
- Paul A, Mandal PK, Samanta A (2005) On the optical properties of the imidazolium ionic liquids. *J Phys Chem B* 109(18):9148–9153
- Samanta A (2006) Dynamic Stokes shift and excitation wavelength dependent fluorescence of dipolar molecules in room temperature ionic liquids. *J Phys Chem B* 110(28):13704–13716
- Hu Z, Margulis CJ (2006) A study of the time-resolved fluorescence spectrum and red edge effect of ANF in a room-temperature ionic liquid. *J Phys Chem B* 110(23):11025–11028