

Sonochemical Synthesis and Characterization of Some Alkoxyl-Functionalized Ionic Liquids Derived from 1-Butoxyl-3-butyl Imidazolium Bromide

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Abstract Non-conventional techniques, such as power ultrasound (US) has been used to promote one-pot synthesis of second generation ionic liquids (ILs), reducing reaction times and improving yields. Because of the emerging importance of the ILs as green materials with wide ranging applications and our general interests in green processes such as sonication, 1-butoxyl-3-butyl imidazolium bromide (alkoxyl-functionalized) and their derivatives were synthesized using a facile and green US assisted procedure. Their structures were characterized by FT–IR, ¹H-NMR, ¹³C-NMR and mass spectroscopy.

Keywords Green synthesis · Ultrasonic irradiation · Imidazolium based ionic liquids · Functionalization · Spectroscopy

1 Introduction

A large excess of a conventional volatile solvent is generally required to carry out a chemical reaction, raising ecological and economic concerns. Ultrasound assisted organic synthesis is a powerful technique and it is a green approach employed for organic reactions, leading to higher yields, shorter reaction times, milder conditions and high analytical purity [1–4]. It is considered as a processing aid in terms of energy conservation and waste minimization compared to other, traditional, methods. Sonochemistry offers synthetic chemists the advantage of chemical activation through cavitation and provides a source of energy which can be used to enhance a variety of chemical reactions. The strong acceleration of reaction rate by ultrasonic irradiation is based on the cavity effect and hot spot formation [5, 6].

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Over the past two decades, the use of ILs as reaction media have attracted considerable attention as eco-friendly substitutes for volatile organic solvents [7, 8]. These solvents, commonly defined as a class of salts with low melting points (less than 100 °C) [9], have attracted intense attention of chemists. ILs present some unique properties, which are often not available in traditional solvents; these include negligible vapor pressure, high chemical and electrochemical stabilities, high polarity, easy recyclability, no flammability, and high ionic conductivity, among others [9–16]. They are widely used as reaction media these days and new approaches involving ILs have been proposed for aspects of energy chemistry [17], preparation of materials [18], biomass valorization [19], lignocellulosic biomass fractionation [20], analytical chemistry [21], microextraction [22], organic and pharmaceutical chemistry [23], electrodeposition of metals [24–26], food chemical science [27], the nuclear industry [28] and many others.

Preparation of ionic liquids by classical methods involves long reaction times and low yields [29, 30]. An efficient microwave assisted synthesis of ionic liquids has reduced the reaction time from several hours to a few minutes [31, 32]. However, as a result of the exothermic nature of the reaction, continuous microwave heating may result in overheating that leads to the formation of colored products. To overcome these problems, ionic liquids have been prepared using ultrasound as an energy source [33–35], which reduces the reaction time and improves the yield.

Herein, we report the sonochemical synthesis (green synthesis) and characterization of some new, task specific, alkoxyl functionalized ionic liquids derived from 1-butoxyl-3-butyl imidazolium bromide.

2 Materials and Methods

Imidazole (99%, Himedia, India), *n*-BuLi (98%, Sigma–Aldrich, India), 1-bromobutane (99%, CDH, India), H_2O_2 (30%, Thomas Baker, India), dichloromethane (99%, Fisher Scientific, India), sodium tetrafluoroborate (99%), silver sulfate (99%), sodium trifluoromethane sulphonate (98%) and *p*-toluene sulphonic acid (99%) (Himedia) were used as received. Ultrasonic cleaner 392 (Systronics) was used for ultrasonic irradiation with a frequency of 40 kHz and a nominal power of 115 W.

3 Experimental

All reactions were carried out in an ultrasonic bath. The synthesis of ionic liquids involves four steps (Scheme 1).

- Step I 1-Butylimidazole was synthesized by N-alkylation of imidazole (13.6 g, 0.2 mol) with *n*-butyl lithium (12.8 g, 0.2 mol) in ethanol (50 mL) under the influence of ultrasound for 3 h at room temperature. The progress of the reaction was monitored by TLC.
- Step II 1-Butylimidazole-3-N-oxide was prepared by the reaction of 1-butylimidazole (12.4 g, 0.1 mol) with H_2O_2 (6.8 mL, 0.2 mol) in acetone (30 mL), in the presence of ultrasonic waves for 30 min at room temperature. The light yellow reaction mixture was dried under vacuum.
- Step III Reaction of 1-butyl imidazolium 3-N-oxide (14 mL, 0.1 mol) with 1-bromobutane (13.7 mL, 0.1 mol) for 2 h at room temperature under the influence



Scheme 1 Ultrasound-assisted synthesis of 1-butoxyl-3-butylimidazolium based ionic liquids

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of ultrasonic waves yields 1-butoxyl-3-butyl imidazolium bromide (ionic liquid).

Step IV In the presence of ultrasound, the reaction of 1-butoxyl-3-butylimidazolium bromide ([BO-BIm] Br) (5.5 mL, 0.02 mol) was carried out either with sodium tetrafluoroborate (2.2 g, 0.02 mol), silver sulfate (6 g, 0.02 mol), sodium trifluoromethyl sulphonate (sodium triflate) (3.4 mL, 0.02 mol) or *p*-toluene sulphonic acid (3.4 g, 0.02 mol) to give 1-butoxyl-3-butylimidazolium tetrafluoroborate (Step IV a), 1-butoxyl-3-butylimidazolium sulfate (Step IV b), 1-butoxyl-3-butylimidazolium trifluoromethyl sulphonate (triflate) (Step IV c) and 1-butoxyl-3-butylimidazolium *p*-toluene sulphonate (tosylate) (Step IV d) ionic liquids, respectively, in methanol (30 mL).

These ionic liquids were dissolved in dichloromethane, which were further extracted with ether for purification. The muddy solution was kept at room temperature untill it became transparent. The solutions were then filtered and washed three times with ether.

The alkylating agents are quantitatively extracted with the ether. After work-up, the products were dried under 80 °C for 12 h at high vacuum (0.01 mbar) to remove any volatiles remaining from the preparation. Water may still be present in trace amounts. Table 1 shows impurities present in ionic liquids.

4 Results and Discussion

A Perkin-Elmer spectrum–2000 Fourier transform IR spectrophotometer (USA) was used to obtain the IR spectra between 400 and 4000 cm⁻¹. A NaCl cell was used for liquid samples by dissolving a sample in dichloromethane (DCM). ¹H-NMR and ¹³C-NMR spectra were recorded using a multinuclear FT–NMR spectrometer (Model Avance-II, Bruker). The instrument was equipped with a cryomagnet of field strength 9.4 T. Its ¹H frequency was 400 MHz, while for ¹³C the frequency was 100 MHz. Waters, Q-Tof micro Mass spectrometer (LC–MS) was used for mass spectral analysis of the synthesized ionic liquids.

S. no.	Ionic liquid	Reagent used for preparation	[Na ⁺] (ICP-MS)	[Br ⁻] (ICP- MS) wt%	H ₂ O (Karl- Fischer titration) wt%
1	1-Butoxyl-3-butylimidazolium bromide	1-Bromobutane		0.097	0.021
2	1-Butoxyl-3-butylimidazolium tetrafluoroborate	Sodium tetrafluoroborate	0.080 wt%	0.090	0.031
3	1-Butoxyl-3-butylimidazolium sulfate	Silver sulfate	-	0.028	0.009
4	1-Butoxyl-3-butylimidazolium trifluoromethane sulphonate	Sodium trifluoromethane sulphonate	0.072 wt%	0.071	0.015
5	1-butoxyl-3-butylimidazolium <i>p</i> -toluene sulphonate	<i>p</i> -toluene sulphonic acid	-	0.069	0.019

Table 1 Impurities present in the synthesized ionic liquids

5 Characterizations

5.1 1-Butoxyl-3-Butylimidazolium Bromide ([BO-BIm] Br)

IR (NaCl cell, cm⁻¹): C–H stretching, 2874; CH₂ bending, 1464; CH₃ bending, 1378; =CH stretching, 3137; N–C stretching, 1260 and 3080, N–O stretching, 1512 and 1323, C–O stretching, 1285, C=C stretching, 1645 and C=N stretching, 1632 cm⁻¹.

¹H-NMR (CDCl₃, 400 MHz): δ ppm = 0.92 (t, ¹⁰CH), 1.47 (m, ⁹CH), 1.50 (m, ⁸CH), 3.45 (t, ⁷CH), 7.50 (t, ⁵CH), 7.86 (t, ⁴CH), 8.84 (s, ²CH), 3.87 (t, ¹¹CH), 1.91 (m, ¹²CH), 1.22 (m, ¹³CH), 0.86 (t, ¹⁴CH).

¹³C-NMR (CDCl₃, 100 MHz) : (δ ppm): 13.98 (¹⁰CH), 17.49 (⁹CH), 31.23 (⁸CH), 64.46 (⁷CH), 122.84 (⁴CH), 122.65 (⁵CH), 135.29 (²CH), 45.91 (¹¹CH), 32.56 (¹²CH), 20.04 (¹³CH) and 13.86 (¹⁴CH).

Mass peaks: 197.3 (C₁₁H₂₁N₂O), 140.1 (C₇H₁₂N₂O), 124.1 (C₇H₁₂N₂).

5.2 1-Butoxyl-3-Butylimidazolium Tetrafluoroborate ([BO-BIm] BF₄)

IR (NaCl cell, cm⁻¹): C–H stretching, 2850; CH₂ bending, 1446; CH₃ bending, 1368; =CH stretching, 3061; N–C stretching, 1287 and 3095, N–O stretching, 1534 and 1326, C–O stretching, 1213, C=C stretching, 1653; C=N stretching, 1664 and B–F stretching, 1453, 712 and 481 cm⁻¹.

¹H-NMR (CDCl₃, 400 MHz): δ ppm = 0.93 (t, ¹⁰CH), 1.40 (m, ⁹CH), 1.48 (m, ⁸CH), 3.39 (t, ⁷CH), 7.74 (t, ⁵CH), 7.87 (t, ⁴CH), 8.79 (s, ²CH), 3.91 (t, ¹¹CH), 1.94 (m, ¹²CH), 1.21 (m, ¹³CH), 0.88 (t, ¹⁴CH).

¹³C-NMR (CDCl₃, 100 MHz): (δ ppm) : 13.88 (¹⁰CH), 19.19 (⁹CH), 31.32 (⁸CH), 63.37 (⁷CH), 122.53 (⁴CH), 122.10 (⁵CH), 136.13 (²CH), 46.88 (¹¹CH), 32.51 (¹²CH), 19.93 (¹³CH) and 13.04 (¹⁴CH).

Mass peaks: 197.3 (C11H21N2O), 140.1 (C7H12N2O), 124.1 (C7H12N2), 67.8 (BF3).

5.3 1-Butoxyl-3-Butylimidazolium Sulfate ([BO-BIm] SO₄)

IR (NaCl cell, cm⁻¹): C–H stretching, 2868; CH₂ bending, 1462; CH₃ bending, 1380; =CH stretching, 3133; N–C stretching, 1272 and 3051, N–O stretching, 1545 and 1365, C–O stretching, 1145; C=C stretching, 1646; C=N stretching, 1639; S=O stretching, 1167 and S–O stretching, 949 cm⁻¹.

¹H-NMR (CDCl₃, 400 MHz): δ ppm = 0.93 (t, ¹⁰CH), 1.36 (m, ⁹CH), 1.50 (m, ⁸CH), 3.39 (t, ⁷CH), 7.68 (t, ⁵CH), 7.97 (t, ⁴CH), 8.85 (s, ²CH), 4.01 (t, ¹¹CH), 1.93 (m, ¹²CH), 1.24 (m, ¹³CH), 0.84 (t, ¹⁴CH).

¹³C-NMR (CDCl₃, 100 MHz): δ ppm = 13.80 (¹⁰CH), 17.56 (⁹CH), 28.98 (⁸CH), 63.82 (⁷CH), 123.12 (⁴CH), 120.97 (⁵CH), 138.14 (²CH), 47.21 (¹¹CH), 32.47 (¹²CH), 20.11 (¹³CH) and 13.54 (¹⁴CH).

Mass peaks: 197.3 (C₁₁H₂₁N₂O), 140.1 (C₇H₁₂N₂O), 124.1 (C₇H₁₂N₂).

5.4 1-Butoxyl-3-Butylimidazolium Trifluoromethane Sulphonate ([BO-BIm] TfO)

IR (NaCl cell, cm⁻¹): C–H stretching, 2939; CH₂ bending, 1466; CH₃ bending, 1371; =CH stretching, 3112; N–C stretching, 1256 and 2966; N–O stretching, 1556 and 1351; C–O

stretching, 1166; C=C stretching, 1639; C=N stretching, 1632; S=O, 1371; S-O, 851 and C-F stretching (trifloromethyl- 2 strong broad bands), 1166 and 1256 cm⁻¹.

¹H-NMR (CDCl₃, 400 MHz): δ ppm = 0.91 (t, ¹⁰CH), 1.37 (m, ⁹CH), 1.54 (m, ⁸CH), 3.42 (t, ⁷CH), 7.71 (t, ⁵CH), 7.78 (t, ⁴CH), 8.95 (s, ²CH), 4.02 (t, ¹¹CH), 1.94 (m, ¹²CH), 1.21 (m, ¹³CH), 0.85 (t, ¹⁴CH).

¹³C-NMR (CDCl₃, 100 MHz): (δ ppm) : 13.64 (¹⁰CH), 18.21 (⁹CH), 31.35 (⁸CH), 64.96 (⁷CH), 122.68 (⁵CH), 122.09 (⁴CH), 136.66 (²CH), 46.56 (¹¹CH), 32.32 (¹²CH), 20.02 (¹³CH) and 13.32 (¹⁴CH) and 135.02 (^aCH).

Mass peaks: 197.3 (C₁₁H₂₁N₂O), 140.1 (C₇H₁₂N₂O), 124.1 (C₇H₁₂N₂), 149 (CF₃O₃S).

5.5 1-Butoxyl-3-Butylimidazolium *p*-Toluene Sulphonate ([BO-BIm] Tos)

IR (NaCl cell, cm⁻¹): C–H stretching, 2875; CH₂ bending, 1461; CH₃ bending, 1383; =CH stretching, 3109; N–C stretching, 1333 and 2963; N–O stretching, 1543 and 1326; C–O stretching, 1185; C=C stretching,1601; C=N stretching, 1719; C=C stretching (conjugation), 1629; C–H in plane, 1399; C–H bend (para), 817; S=O stretching, 1383 and S–O stretching, 753 cm⁻¹.

¹H-NMR (CDCl₃, 400 MHz): δ ppm = 0.94 (t, ¹⁰CH), 1.42 (m, ⁹CH), 1.50 (m, ⁸CH), 3.45 (t, ⁷CH), 7.69 (t, ⁵CH), 7.88 (t, ⁴CH), 8.85(s, ²CH), 3.99 (t, ¹¹CH), 1.95 (m, ¹²CH), 1.23 (m, ¹³CH), 0.87 (t, ¹⁴CH) 2.39 (^eCH), 7.41 (^eCH), 7.69 (^bCH).

¹³C-NMR (CDCl₃, 100 MHz): (δ ppm) : 14.28 (¹⁰CH), 19.15 (⁹CH), 30.41 (⁸CH), 64.52 (⁷CH), 122.76 (⁴CH), 122.55 (⁵CH), 135.83 (²CH), 47.12 (¹¹CH), 32.78 (¹²CH), 19.47 (¹³CH) and 13.45 (¹⁴CH) 21.32 (^eCH), 138.06 (^dCH), 129.07 (^eCH), 126.83 (^bCH) and 140.93 (^aCH).

Mass peaks: 197.3 ($C_{11}H_{21}N_2O$), 140.1 ($C_7H_{12}N_2O$), 124.1 ($C_7H_{12}N_2$), 171.2 ($C_7H_7O_3S$), 156.1 ($C_6H_4O_3S$), 15 (CH_3), 91 (C_7H_7).

5.6 Viscosity

Ionic liquids are viscous liquids as compared to the conventional organic solvents, being 1–3 orders of magnitude more viscous. Alkyl chain lengthening or fluorination makes the salt more viscous, due to an increase in Van der Waals interactions and hydrogen bonds (Fig. 1). Alkyl chain ramification also increases the viscosity due to reduced rotational freedom. The viscosity measurement was carried out with Brookfield DV-I (II) viscometer (1% accuracy, 0.2% repeatability) from 25 to 95 °C at an interval of 5 °C under nitrogen gas.

The calibration was made by comparison with reference oils (Canon S20, S60 and Paragon N10). The estimated overall uncertainty of the temperature readings was ± 0.5 °C. Three sets of each measurement were performed, having deviations from the mean smaller than $\pm 0.1\%$. It has been observed that on increase in temperature, viscosity of an ionic liquid decreases. Viscosities of the synthesized ionic liquids at 25 °C are given in Table 2.



Fig. 1 The chemical structure of the 1-butoxyl-3-butylimidazolium cation [BO-BIm]⁺ with different anions

S. no.	Structural formula	Reaction time in ultrasound (h)	Yield (%)	Viscosity η/cp at 25 °C	Conductivity (S·m ⁻¹) at 27 °C	
1	Bu N Bu Br	2.0	91	551	0.31	
	1-Butoxyl-3-butyl imidazolium bromide					
	[BO-BIm] Br					
2		2.5	80	305	0.25	
	1-Butoxyl-3-butyl imidazolium tetrafluoro borate					
	[BO-BIm] BF ₄					
3	Bu O Bu]2 SO42-	4.0	75	151	0.09	
	1-Butoxyl-3-butyl imidazolium sulfate					
	[BO-BIm] SO ₄					
4	$\begin{bmatrix} & & & & \\ & & & & \\ & & & & \\ & & & & $	3.0	91	85	0.39	
	1-Butoxyl-3-butyl imidazolium trifluoromethyl sulphonate					
	[BO-BIm] TfO					
5	$\begin{bmatrix} & & & \\ & & & \\ & & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & $	3.5	86	73	0.41	
	I-butoxyi-3-butyi imidazoilum p-i oluene sulphonate					
	[105-Dill] 105					

Table 2 Properties and reaction data for 1-butoxyl-3-butylimidazolium based ionic liquids

5.7 Conductivity

Ionic liquids are composed of only ions; therefore, they show very high ionic conductivity, thermal stability and nonflammability. Nonflammable liquids with high ionic conductivity are practical materials for use in electrochemistry. Conductivity measurements were carried out using conductivity meter (Systronics Model 305). The instrument conductivity measuring range is 0 to 2000 μ S and 0 to 200.0 mS. The instrument has a full scale resolution of 0.5% and an accuracy of ±1%. Before and after measurements, the instrument was calibrated with aqueous KCl solution. The temperature of the sample was kept at 27 ± ±0.1 °C. Conductivities of the synthesized ionic liquids are also reported in Table 2.

6 Conclusion

It is clear from these observations that a large number of ionic liquids can be synthesized by simple combination of different cations and anions, which can be used for a particular task. Ultrasound assisted synthesis of ionic liquids has the advantages over classical methods (conventional, thermal, chemical, etc.) of being ecofriendly, simplying product isolation, increasing yield and shortening reaction times. Therefore, introduction of such efficient synthetic protocols should lower the cost of ionic liquids; thus, encouraging a wider use of these neoteric solvents.

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