Synthesis, characterization and spectroscopic studies of two new 1-acetyl-3-alkylimidazolium ionic liquids

Abstract: Two new functionalized ionic liquids, 1-acetyl-3-alkylimidazolium iodides, were synthesized by the reactions of 1-acetylimidazole with alkyl iodides under solvent-free condition. Their structures were confirmed by \(^1\)H NMR, ESI-MS, IR, UV-Vis and elemental analysis. The 1-acetyl-3-ethylimidazolium iodide (3a) is a solid and 1-acetyl-3-hexylimidazolium iodide (3b) is a viscous liquid at room temperature.

Keywords: 1-acetylimidazole; iodides; ionic liquids; synthesis.

Results and discussion

The synthesis of 1-acetyl-3-alkylimidazolium-based ionic liquids is outlined in Scheme 1.

It should be noted that only the use of iodoalkanes provided the final products 3. Owing to the presence of an electron-withdrawing acetyl group in 2, the attempted reactions with less reactive bromides and chlorides were not successful [18]. Compared with previous methods in the literatures [16, 17], this new synthetic route appears to be more convenient, is faster and is more efficient.

The structures of these new 1-acetyl-3-alkylimidazolium compounds were confirmed by IR, UV-Vis, \(^1\)H NMR, ESI-MS and elemental analysis. Figure 1 shows the UV-Vis absorption spectra of 3a and 3b compared with that of 1-acetylimidazole. The lowest energy electronic transition at 247 nm for 3a and 248 nm for 3b, which is...
UV-Vis spectra were measured on an Analytikjena Specord 210 Plus instrument. Elemental analysis was performed on a Vario EL analysis system.

1-Acetyl-3-ethylimidazolium iodide (3a)

A mixture of 1-acetylimidazole (11.0 g, 0.1 mol) and iodoethane (32.7 g, 0.3 mol) was heated under reflux for approximately 7 h at 50–60°C. Upon completion of the reaction, the yellow mixture was cooled to room temperature and washed with ether several times to remove the excess iodoethane. After crystallization from acetone/trifluoroacetate ether the compound was obtained as a light yellow solid: yield 17.2 g (65%); mp 97–99°C; IR: 3152, 3098, 2986, 1757, 1530, 1478, 1388, 961, 790 cm⁻¹; ¹H NMR: δ 10.00 (s, 1H), 8.26 (d, 1H, J = 1.6 Hz), 7.99 (d, 1H, J = 1.6 Hz), 4.30 (q, 2H, J = 7 Hz), 2.76 (s, 3H), 1.47 (t, 3H, J = 7 Hz); ESI-MS: m/z 139.11 (M⁺, 60), 97.06 (85), 126.60 (100); UV-Vis (CH₃CN): λmax 207 nm (ε 4.3 × 10³), 247 nm (ε 2.9 × 10³). Anal. Calcd for C₇H₁₁N₂OI: C, 31.60; H, 4.17; N, 10.53. Found: C, 31.50; H, 4.12; N, 10.69.

1-Acetyl-3-hexylimidazolium iodide (3b)

A mixture of 1-acetylimidazole (1.33 g, 0.01 mol) and 1-iodohexane (6.49 g, 0.03 mol) was heated to 50–60°C for 7 h. Upon completion of the reaction, the yellow mixture was cooled to room temperature and washed with ether and acetone to remove 1-iodohexane. This compound was obtained as a light yellow liquid: yield 1.76 g (55%); IR: 3092, 2931, 2858, 1718, 1668, 1544, 1409, 940, 762 cm⁻¹; ¹H NMR: δ 10.00 (s, 1H), 8.26 (d, 1H, J = 2.3 Hz), 8.01 (d, 1H, J = 2.3 Hz), 4.16 (t, 2H, J = 7 Hz), 1.72 (m, 8H), 1.24 (t, 3H, J = 7 Hz); ESI-MS: m/z 195.10 (M⁺, 57), 153.00 (88), 126.60 (100); UV-Vis (CH₃CN): λmax 208 nm (ε 4.8 × 10³), 248 nm (ε 3.1 × 10³). Anal. Calcd for C₁₁H₁₉N₂OІ: C, 41.01; H, 5.94; N, 8.69. Found: C, 40.71; H, 5.65; N, 10.12.

Acknowledgments: This work was supported by the project of Key Technology Researches on Desiccant Evaporation Cooling System with Medium Temperature Solar Energy of MOHURD and Beijing Forestry University Young Scientist Fund (2008XJS16).

Received March 12, 2013; accepted April 19, 2013; previously published online May 25, 2013

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