Synthesis and Characterization of Antibacterial Ionic Liquids Moieties under Multiple Routes and their Catalytic Responses

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Supplementary data

Experimental section:

General procedure for N-alkylation reaction:

2-Methyl-4-(5)-nitro-1H-imidazole (1.573 × 10⁻² mmol; 1.0 equiv.) mixed with slight excess of benzyl bromide/4-nitro benzyl bromide (1.652 × 10⁻² mmol; 1.0 equiv.) in presence of 30 mL of the MeCN under refluxing condition for about 9-13 hours afforded the N-alkylated products of (1a-b) in quantitative yield after the purification.

General procedure for solvent free Muffle furnace condition:

Required equivalent as mentioned in conventional method except solvent, we have used 5 g of (80-120 mesh) silica gel with fine grinding using mortar and pestle. The reaction mixture is kept in Muffle furnace at 100°C for required period.

General procedure for anion exchange reaction:

N-alkylated product of imidazolium bromide 1a,1b (1.0 equiv.) is treated with NaBF₄/KPF₆/LiCF₃SO₃ (1.05 equiv.) in the presence of 10 mL of deionized water at room temperature stirring for about 1 h afforded the anion exchanged product of imidazolium cation with different anion. After the anion exchanged reaction, we have used Soxhlet extraction to remove metal bromide from di/trimeric imidazolium salts using 100 mL of dry THF for about 1 h refluxation to give respective imidazolium salts in quantitative yield.

General procedure for imidazolium salt assisted Pechmann reaction:

Phenol/4-nitrophenol/4-chlorophenol (2.126 × 10⁻³ mmol; 1.0 eq.), EAA (2.126 × 10⁻³ mmol; 1.0 eq.), solvent (5 mL) are mixed along with optimized concentration of imidazolium salt (2.021 × 10⁻⁴ mmol) at room temperature stirring, after disappearance of starting material monitored by TLC, the reaction mixture was poured into 5 mL of ice cold water and 100 mL of diethyl ether stirred for 1 h. Two layer are formed; the organic layer which was then dried over anhydrous Na₂SO₄.

General procedure for solvent free Muffle furnace condition:

Required equivalent as mentioned in conventional method except solvent, we have used 5 g of (80-120 mesh) silica gel with fine grinding using mortar and pestle. The reaction mixture is kept in Muffle furnace at 100°C for required period.
NMR (100 MHz, CDCl₃): δ; 19.1, 116, 117.9, 122, 124.1, 124.6, 132.6, 154.5, 161.6; MS: m/z: 160.05 Elemental analysis: Molecular formula (C₆H₅O₂) Calculated: C: 74.99; H: 5.03; Found: C: 74.94; H: 4.99.

4-Methyl-6-nitro-2H-chromen-2-one (4): 0.34 g, 80%, Mp: 153-155°C, ¹H NMR (400 MHz, CDCl₃): δ; 2.43 (s, 3H), 6.14 (s, 1H), 7.30 (d, 1H), 8.10-8.15 (d, 1H), 8.21-8.24 (d, 1H); ¹³C NMR (100 MHz, CDCl₃): δ; 20.3, 111.4, 120.9, 129.5, 122.0, 144.3, 151.7, 155.9, 161.8; MS: m/z: 205.03. Elemental analysis: Molecular formula (C₁₀H₇NO₄) Calculated: C: 58.84, H: 3.44, N: 6.83, Found: C: 58.80, H: 3.40, N: 6.80.

6-Chloro-4-methyl-2H-chromen-2-one (5): 0.35 g, 86%, Mp: 179-182°C, ¹H NMR (400 MHz, CDCl₃): δ; 2.05 (s, 3H), 6.18 (s, 1H), 7.12-7.16 (d, 1H), 7.19-7.22 (d, 1H), 7.29 (s, 1H); ¹³C NMR (100 MHz, CDCl₃): δ; 21.4, 115.2, 122.4, 123.4, 127.3, 129.8, 132.3, 149.2, 154.5, 160.9; MS: m/z: 194.01 Elemental analysis: Molecular formula (C₆H₅ClO₂) Calculated: C: 61.72, H: 3.63, Found: C: 61.67, H: 3.58.

1-Methyl-3H-benzo[f]chromen-3-one (6): 0.39 g, 89%, Mp: 182-185°C, ¹H NMR (400 MHz, CDCl₃): δ; 2.45 (s, 3H), 6.32 (s, 1H), 7.32-7.60 (m, 4H), 7.90-7.94 (d, 1H), 8.54-8.57 (d, 1H); ¹³C NMR (100 MHz, CDCl₃): δ; 24.10, 109.47, 115.70, 116.80, 117.76, 123.60, 126.53, 129.84, 134.58, 153.40, 154.90, 160.30. MS: m/z: 210.06 Elemental analysis: Molecular formula (C₁₄H₁₀O₂) Calculated: C: 79.98, H: 4.79, Found: C: 79.92, H: 4.74.

¹H NMR spectrum of 2-Methyl-4(5)-nitro(3-methylenebenzene)-imidazoliumbromide 1a

¹³C NMR spectrum of 2-Methyl-4(5)-nitro(3-methylenebenzene)-imidazoliumbromide 1a
Mass spectrum of 2-Methyl-5-nitro(3-methylenebenzene)-imidazoliumbromide 1a

$^1$H NMR spectrum of 2-Methyl-4(5)-nitro(3-methylene4'-nitrobenzene)-imidazoliumbromide 1b