Crown ether complex cation ionic liquids (CECILs) as environmentally benign catalysts for three-component synthesis of 4,5-dihydropyrano[3,2-*c*]chromene and 4,5-dihydropyrano[4,3-*b*]pyran derivatives

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Abstract Crown ether complex cation ionic liquids (CECILs) catalyze threecomponent reaction of aromatic aldehydes, malononitrile, and 4-hydroxycoumarin or 4-hydroxy-6-methylpyrone, in EtOH under reflux, for synthesis of 4,5-dihydropyrano[3,2-*c*]chromene and 4,5-dihydropyrano[4,3-*b*]pyran derivatives. CECILs are environmentally benign, easily obtained, stable catalysts. High conversion, short reaction times, and cleaner reaction profiles are some of the advantages of this method.

Keywords 4,5-Dihydropyrano[3,2-*c*]chromene derivatives · 4,5-Dihydropyrano[4,3-*b*]pyran derivatives · Multicomponent reaction · Crown ether complex cation ionic liquids (CECILs)

Introduction

Compared with other methods, multicomponent reactions (MCRs) have emerged as attractive and powerful strategies for organic synthesis [1-3]. MCRs are highly flexible and enable exceptionally efficient synthesis of complex molecules, frequently with high stereoselectivity, from simple and readily available substrates [4-8]. MCRs have been developed for synthesis of a variety small organic molecules in organic, bioorganic, and medicinal chemistry [9–12] and are regarded

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as vital for synthesis of many important heterocyclic compounds, for example 4,5dihydropyrano[3,2-*c*]chromene and 4,5-dihydropyrano[4,3-*b*]pyran derivatives.

Dihydropyrano[3,2-c]chromenes and their derivatives are of substantial interest because of their wide range of biological properties [13, 14], for example spasmolytic, diuretic, anti-coagulant, anticancer, and anti-anaphylactic activity [15, 16]. 4,5-Dihydropyrano[4,3-*b*]pyran derivatives also have wide range of pharma-cological and biological applications, because of their fungicidal, insecticidal, acaricidal [17], antiviral [18], antileishmanial, and anticonvulsant activity [19].

Ionic liquids (ILs), salts with melting points below 100 °C consisting of ions, have attracted much attention from research groups because of their unique properties and novel applications. ILs have low vapor pressure, excellent thermal stability, desirable solvating properties, and are recyclable [20]. They have been used in many organic reactions, for example, hydrogenation, oxidation [21–25], three-component reactions [26], and carbon–carbon bond-forming reactions, for example Michael addition, the Henry reaction, Knoevenagel condensation, and Heck reaction [27]. Crown ether complex ionic liquids (CECILs), have been synthesized by chelating crown ethers with alkali metal cations [27]. Because electrostatic interactions between the large cations and the anions are relatively weak compared with van der Waals forces, the lattice energy and melting points of the crystals are low.

In the work reported herein, we synthesized a series of crown ether complex cation ionic liquids (CECILs), by chelating crown ethers with alkali metal cations, and used them as environmentally benign catalysts for synthesis of 4,5-dihydropyrano[3,2-*c*]chromene and 4,5-dihydropyrano[4,3-*b*]pyran derivatives by threecomponent reaction of aromatic aldehydes, malononitrile, and 4-hydroxycoumarin or 4-hydroxy-6-methylpyrone, in EtOH, under reflux.

Results and discussion

In this work we dissolved the crown ether (1) and inorganic or organic base (2a-d) in methanol to generate the required CECIL (3a-d) (Scheme 1).

We then used the CECILs as catalysts for three-component reaction of aromatic aldehydes (**4a–p**), malononitrile (**5**), and 4-hydroxycoumarin (**6a**) or 4-hydroxy-6-methylpyrone (**6b**) for synthesis of 4,5-dihydropyrano[3,2-*c*]chromene (**7a–p**) and 4,5-dihydropyrano[4,3-*b*]pyran (**7q–f**') derivatives, in EtOH, under reflux (Scheme 2).

To optimize the reaction conditions, the reaction between benzaldehyde (4a), malononitrile (5), and 4-hydroxycoumarin (6a) was chosen as a model reaction.





Scheme 2 Three-component reaction of aromatic aldehydes (4a-p), malononitrile (5) and 4-hydroxycoumarin (6a) or 4-hydroxy-6-methylpyrone (6b) in the presence of catalytic amounts of [18-C-6 K][OAc]

Table 1 Optimization of the model reaction between benzaldehyde (4a), malononitrile (5) and 4-hydroxycoumarin (6a)

Entry	Solvent	Catalyst	Catalyst (mol%)	Time (min)	Yield (%)
1	EtOH	[18-C-6K][OAc]	30	15	90
2	Ethyl acetate	[18-C-6K][OAc]	30	45	78
3	Acetonitrile	[18-C-6K][OAc]	30	40	80
4	Toluene	[18-C-6K][OAc]	30	80	60
5	EtOH	[18-C-6Na][OAc]	30	20	88
6	EtOH	[18-C-6K][OH]	30	27	85
7	EtOH	[18-C-6Na][OH]	30	30	83
8	EtOH	[18-C-6K][OAc]	10	25	82
9	EtOH	[18-C-6K][OAc]	20	20	88
10	EtOH	[18-C-6K][OAc]	40	15	90

When this three-component reaction was performed in the presence of [18-C-6K][OAc] (30 mol%), in EtOH, under reflux, the 2-amino-5-oxo-4-phenyl-4,5-dihydropyrano[3,2-*c*]chromene-3-carbonitrile **7a** was obtained in 90 % yield within 15 min. This reaction was also carried out in other solvents (ethyl acetate, acetonitrile, and toluene), but the best results in terms of reaction time and yield of the desired product **7a** were obtained when the reaction was conducted in EtOH (Table 1, entries 1–4). Other CECILs were also used as catalysts (Table 1, entries 5–7). We also optimized the quantity of catalysts. The best results were obtained when the reactions were performed in the presence of 30 mol% [18-C-6K][OAc] (Table 1, entries 8–10).

On the basis of these results we decided to use this method for synthesis of 4,5dihydropyrano[3,2-*c*]chromene (**7a**–**p**) and 4,5-dihydropyrano[4,3-*b*]pyran (**7q**–**f**') derivatives by three-component reaction of aromatic aldehydes (**4a**–**p**), malononitrile (**5**) and 4-hydroxycoumarin (**6a**) or 4-hydroxy-6-methylpyrone (**6b**), in EtOH, under reflux, in the presence of [18-C-6K][OAc] (30 mol%) (Table 2).

Conclusion

In summary, we report use of crown ether complex cation ionic liquids (CECILs) as catalysts for three-component reaction of aromatic aldehydes, malononitrile, and 4-hydroxycoumarin or 4-hydroxy-6-methylpyrone, for synthesis of 4,5-dihydropyr-ano[3,2-*c*]chromene and 4,5-dihydropyrano[4,3-*b*]pyran derivatives. These reactions were performed in EtOH under reflux. High yields, operational simplicity, clean reaction conditions are advantages of this procedure that make it a useful practical process for synthesis of these compounds.

Experimental

General Melting points were measured on an Electrothermal-9100 apparatus and are uncorrected. IR spectra were recorded on a Brucker FT-IR Tensor 27 infrared spectrophotometer. ¹H NMR spectra were recorded on a Brucker Avance III 400 MHz spectrometer. ¹³C NMR spectra were recorded on the same instruments at 100 MHz. TMS was used as internal standard for NMR spectroscopy. Elemental analysis was performed with a Heraeus CHN-O-Rapid analyzer.

General procedure for synthesis of CECILs (3a-d)

Organic or inorganic potassium or sodium base (2a-d) (2 mol) and 18-crown-6 (1) (2 mol) in 15 mL methanol was stirred for 1 h at 60 °C. The methanol was removed under reduced pressure. The residue was dried under vacuum to generate the desired CECIL in 100 % yield. Synthesis of CECILs **3b** and **3d** has been reported elsewhere [27].

[18-C-6Na][OH] (**3a**)

Liquid; ¹H NMR (400 MHz, D₂O) δ_{ppm} : 3.92 (s, 24H); ¹³C NMR (100 MHz, D₂O): δ_{ppm} : 69.01. Anal. calcd. for C₁₂H₂₅NaO₇: C, 47.36; H, 8.28 %. Found: C, 47.18; H, 8.12 %.

[18-C-6K][OH] (**3b**)

White powder; M.P. 38 °C; ¹H NMR (400 MHz, D₂O) δ_{ppm} : 3.58 (s, 24H); ¹³C NMR (100 MHz, D₂O): δ_{ppm} : 68.79. Anal. calcd. for C₁₂H₂₅KO₇: C, 44.98; H, 7.86 %. Found: C, 44.80; H, 7.71 %.

Table 2 Three-cc	omponent reaction of aroma	tic aldehydes (4a-p), malo	monitrile (5) and 4-hy	droxycoumarin (6a)	or 4-hydroxy-6-methylpyrone	; ((p)
NH4 NH4 NH4 NH4 NH4 NH4 NH4 NH4 NH4 NH4	Ar CN	² Ar				
7a-p	7q-f'					
Compd. no.	Activated C-H acid	Ar	Time (min)	Yield (%)	M.P. observed (°C)	M.P. reported (°C)
7a	6a	C_6H_5	15	06	259–261	261–263 [28]
Tb	6a	$4-CI-C_6H_4$	13	91	265-268	265–267 [28]
7с	6a	$2-CI-C_6H_4$	13	91	265-267	266–268 [29]
7d	6a	2,4-(CI) ₂ -C ₆ H ₃	12	92	253–255	256-258 [30]
7e	6a	$4-Br-C_6H_4$	13	91	246–248	247-250 [30]
Tf	6a	$2-Br-C_6H_4$	13	90	293–296	295–297 [29]
7g	6a	$4-NO_2-C_6H_4$	12	92	250-251	250-252 [28]
7h	6a	$3-NO_2-C_6H_4$	13	91	264-266	263-265 [28]
Ті	6a	$2-NO_2-C_6H_4$	12	91	256-258	Ι
7j	6a	$4-CH_3-C_6H_4$	18	88	258-260	257-259 [28]
7k	6a	$4-CH_3O-C_6H_4$	20	87	232–234	235–237 [28]
Ц	6a	$2-CH_3O-C_6H_4$	20	88	246–248	247–249 [29]
7m	6a	$4-OH-C_6H_4$	25	85	257-260	258–260 [28]
7n	6a	Furan-2-yl	22	89	250-253	253–255 [28]
70	6a	Thiophen-2-yl	23	88	227–229	226–230 [30]
7p	6a	Pyridin-3-yl	17	90	257-259	Ι
7q	6b	C_6H_5	18	89	233–235	234–236 [31]

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Compd. no.	Activated C-H acid	Ar	Time (min)	Yield (%)	M.P. observed (°C)	M.P. reported (°C)
7r	6b	4-CI-C ₆ H ₄	15	06	230–231	231–232 [32]
7s	6b	2-CI-C ₆ H ₄	15	06	269–272	270–271 [32]
7t	6b	2,4-(Cl) ₂ -C ₆ H ₃	13	16	233–234	234–235 [32]
7u	6b	$4-Br-C_6H_4$	15	06	220-222	223–225 [32]
7v	6b	$2-Br-C_6H_4$	15	89	271–273	I
7w	6b	$4-NO_2-C_6H_4$	13	91	215-217	215-218 [31]
7x	6b	$3-NO_2-C_6H_4$	15	89	229–232	232–233 [32]
7y	6b	$2-NO_2-C_6H_4$	13	06	261-263	I
7z	6b	4-CH ₃ -C ₆ H ₄	22	87	222-223	224–225 [31]
7a′	6b	$4-CH_{3}O-C_{6}H_{4}$	25	85	202-204	202-205 [31]
7b′	6b	$2-CH_{3}O-C_{6}H_{4}$	25	86	243-245	I
7c′	6b	$4-OH-C_6H_4$	30	85	>300	I
7d′	6b	Furan-2-yl	20	88	220-222	223–224 [31]
7e′	6b	Thiophen-2-yl	22	87	240–243	242-244 [31]
Τf	6b	Pyridin-3-yl	17	89	222–224	I

Table 2 continued

[18-C-6Na][OAc] (3c)

White powder; M.P. 56 °C; ¹H NMR (400 MHz, D₂O) δ_{ppm} : 3.79 (s, 24H), 1.81 (s, 3H); ¹³C NMR (100 MHz, D₂O): δ_{ppm} : 179.01, 69.03, 25.12. Anal. calcd. for C₁₄H₂₇NaO₈: C, 48.55; H, 7.86 %. Found: C, 48.36; H, 7.70 %.

[18-C-6K][OAc] (3d)

White powder; M.P. 75 °C; ¹H NMR (400 MHz, D₂O) δ_{ppm} : 3.38 (s, 24H), 1.79 (s, 3H); ¹³C NMR (100 MHz, D₂O): δ_{ppm} : 178.88, 68.26, 24.12. Anal. calcd. for C₁₄H₂₇KO₈: C, 46.39; H, 7.51 %. Found: C, 46.20; H, 7.34 %.

Typical procedure for the of 4,5-dihydropyrano[3,2-c]chromene (7**a**-**p**) and 4,5-dihydropyrano[4,3-b]pyran (7**q**-**f**') derivatives

A mixture of aromatic aldehyde (4a-p) (2 mmol), malononitrile (5) (2 mmol), and 4-hydroxycoumarin (6a) or 4-hydroxy-6-methylpyrone (6b) (2 mmol), and [18-C-6K][OAc] (30 mol%) in EtOH (10 mL) was heated under reflux for the time reported in Table 2 (the progress of the reaction being monitored by TLC with hexane–ethyl acetate as mobile phase). After completion of the reaction, the reaction mixture was poured into ice-cold water; the crude product was isolated by filtration, dried, and recrystallized from ethanol.

2-Amino-4-(2-nitrophenyl)-5-oxo-4,5-dihydropyrano[3,2-c]chromene-3-carbonitrile (7i)

White powder; IR (KBr, v_{max}/cm^{-1}): 3,392, 3,312 (NH₂), 2,192 (CN), 1,670 (C=O), 1,600, 1,577 (C=C); ¹H NMR (400 MHz, DMSO-d₆) δ_{ppm} : 7.89–7.42 (m, 10H, CH-Ar, NH₂), 5.23 (s, 1H, CH); ¹³C NMR (100 MHz, DMSO-d₆) δ_{ppm} : 161.29 (C=O), 160.22 (C6), 155.20 (C2), 153.75, 150.79, 138.95, 135.26, 134.66, 132.77, 130.04, 126.30, 125.57, 124.15, 120.33, 118.18, 114.38 (CN), 104.85 (C5), 57.73 (C3), 33.22 (C4); Anal. calcd. for C₁₉H₁₁N₃O₅: C, 63.16; H, 3.07; N, 11.63 %. Found: C, 62.95; H, 2.91; N, 11.42 %.

2-Amino-5-oxo-4-(pyridin-3-yl)-4,5-dihydropyrano[3,2-c]chromene-3-carbonitrile (7p)

White powder; IR (KBr, v_{max}/cm^{-1}): 3,376, 3,280 (NH₂), 2,192 (CN), 1,670 (C=O), 1,603, 1,571 (C=C); ¹H NMR (400 MHz, DMSO-d₆) δ_{ppm} : 8.52–7.33 (m, 10H, CH–Ar, NH₂), 4.53 (s, 1H, CH); ¹³C NMR (100 MHz, DMSO-d₆) δ_{ppm} : 161.16 (C=O), 159.70 (C6), 155.40 (C2), 153.81, 150.64, 149.83, 140.37, 137.13, 134.58, 126.22, 125.43, 124.14, 120.66, 118.13, 114.52 (CN), 104.52 (C5), 58.65 (C3), 36.33 (C4); Anal. calcd. for C₁₈H₁₁N₃O₃: C, 68.14; H, 3.49; N, 13.24 %. Found: C, 67.93; H, 3.30; N, 13.05 %.

2-Amino-4-(2-bromophenyl)-7-methyl-5-oxo-4,5-dihydropyrano[4,3-b]pyran-3-carbonitrile (7v)

Yellow powder; IR (KBr, v_{max}/cm^{-1}): 3,456, 3,344 (NH₂), 2,192 (CN), 1,664 (C=O), 1,603, 1,574 (C=C); ¹H NMR (400 MHz, DMSO-d₆) δ_{ppm} : 7.54–7.15 (m, 6H, CH–Ar, NH₂), 6.26 (s, 1H, CH), 4.78 (s, 1H, CH), 2.20 (s, 3H, CH₃); ¹³C NMR (100 MHz, DMSO-d₆) δ_{ppm} : 162.75 (C=O), 160.36 (C6), 159.77 (C2), 143.69, 134.38, 132.18, 130.52, 129.79, 124.47, 120.42, 113.23 (CN), 101.44 (C5), 99.48 (CH), 58.29 (C3), 37.56 (C4), 20.95 (CH₃); Anal. calcd. for C₁₆H₁₁BrN₂O₃: C, 53.50; H, 3.09; N, 7.80 %. Found: C, 53.23; H, 2.92; N, 7.62 %.

2-Amino-7-methyl-4-(2-nitrophenyl)-5-oxo-4,5-dihydropyrano[4,3-b]pyran-3-carbonitrile (**7y**)

White powder; IR (KBr, v_{max}/cm^{-1}): 3,472, 3,360 (NH₂), 2,192 (CN), 1,670 (C=O), 1,606, 1,574 (C=C); ¹H NMR (400 MHz, DMSO-d₆) δ_{ppm} : 7.84–7.35 (m, 6H, CH–Ar, NH₂), 6.27 (s, 1H, CH), 5.05 (s, 1H, CH), 2.18 (s, 3H, CH₃); ¹³C NMR (100 MHz, DMSO-d₆) δ_{ppm} : 163.05 (C=O), 160.36 (C6), 159.90 (C2), 150.85, 139.15, 135.16, 132.55, 129.92, 125.47, 120.41, 114.04 (CN), 101.67 (C5), 99.57 (CH), 57.56 (C3), 32.57 (C4), 20.91 (CH₃); Anal. calcd. for C₁₆H₁₁N₃O₅: C, 59.08; H, 3.41; N, 12.92 %. Found: C, 58.89; H, 3.23; N, 12.74 %.

2-Amino-4-(2-methoxyphenyl)-7-methyl-5-oxo-4,5-dihydropyrano[4,3-b]pyran-3-carbonitrile (**7b**')

Yellow powder; IR (KBr, v_{max}/cm^{-1}): 3,488, 3,344 (NH₂), 2,192 (CN), 1,670 (C=O), 1,600, 1,574 (C=C); ¹H NMR (400 MHz, DMSO-d₆) δ_{ppm} : 7.17–6.84 (m, 6H, CH–Ar, NH₂), 6.22 (s, 1H, CH), 4.48 (s, 1H, CH), 3.69 (s, 3H, OCH₃), 2.18 (s, 3H, CH₃); ¹³C NMR (100 MHz, DMSO-d₆) δ_{ppm} : 162.97 (C=O), 160.44 (C6), 160.28 (C2), 158.78, 132.60, 130.67, 129.93, 127.25, 122.05, 121.11, 113.34 (CN), 101.64 (C5), 99.48 (CH), 58.29 (C3), 57.29 (OCH₃), 33.26 (C4), 20.89 (CH₃); Anal. calcd. for C₁₄H₁₄N₂O₄: C, 65.80; H, 4.55; N, 9.03 %. Found: C, 65.62; H, 4.37; N, 8.84 %.

2-Amino-4-(4-hydroxyphenyl)-7-methyl-5-oxo-4,5-dihydropyrano[4,3-b]pyran-3-carbonitrile (7c')

Brown powder; IR (KBr, v_{max}/cm^{-1}): 3,728–3,024 (broad peak, OH, NH₂), 2,192 (CN), 1,664 (C=O), 1,574, 1,552 (C=C); ¹H NMR (400 MHz, DMSO-d₆) δ_{ppm} : 9.12 (s, 1H, OH), 7.02–6.73 (m, 6H, CH–Ar, NH₂), 6.21 (s, 1H, CH), 4.52 (s, 1H, CH), 2.20 (s, 3H, CH₃); ¹³C NMR (100 MHz, DMSO-d₆) δ_{ppm} : 162.65 (C=O), 160.36 (C6), 159.74 (C2), 156.43, 132.46, 130.40, 129.16, 126.44, 122.14, 120.21, 113.58 (CN), 101.24 (C5), 99.53 (CH), 58.29 (C3), 37.49 (C4), 20.92 (CH₃); Anal. calcd. for C₁₆H₁₂N₂O₄: C, 64.86; H, 4.08; N, 9.46 %. Found: C, 64.68; H, 3.91; N, 9.27 %.

2-Amino-7-methyl-5-oxo-4-(pyridin-3-yl)-4,5-dihydropyrano[4,3-b]pyran-3carbonitrile (7f)

Yellow powder; IR (KBr, v_{max}/cm^{-1}): 3,408, 3,392 (NH₂), 2,192 (CN), 1,664 (C=O), 1,587, 1,523 (C=C); ¹H NMR (400 MHz, DMSO-d₆) δ_{ppm} : 8.43 (s, 2H, NH₂), 7.59–7.28 (m, 4H, CH–Ar), 6.27 (s, 1H, CH), 4.35 (s, 1H, CH), 2.20 (s, 3H, CH₃); ¹³C NMR (100 MHz, DMSO-d₆) δ_{ppm} : 162.96 (C=O), 160.10 (C6), 159.80 (C2), 150.61, 149.83, 140.52, 136.82, 125.34, 120.75, 113.21 (CN), 101.34 (C5), 99.61 (CH), 58.53 (C3), 35.61 (C4), 20.91 (CH₃); Anal. calcd. for C₁₅H₁₁N₃O₃: C, 64.05; H, 3.94; N, 14.94 %. Found: C, 63.88; H, 3.78; N, 14.76 %.

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