Studies on the synthesis of substituted 2-amino-4*H*-benzo[*h*]chromene and 3-amino-1*H*-benzo[*f*]chromene derivatives using base supported ionic liquid like-phase (SILLP) as an efficient green catalyst

Leila Kheirkhah^a, Manouchehr Mamaghani^a*, Nosrat Ollah Mahmoodi^a, Asieh Yahyazadeh^a and Seyedeh Saeedeh Mirnezami Ziabari^b

^aDepartment of Chemistry, Faculty of Sciences, University of Guilan, PO Box 41335-1914, Rasht, Iran ^bDepartment of Chemistry, Faculty of Sciences, Islamic Azad University, Rasht Branch, PO Box 41335-3516, Rasht, Iran

The synthesis of several substituted 2-amino-4*H*-benzo[*h*]chromene and 3-amino-1*H*-benzo[*f*]chromene derivatives was carried out using a one-pot three-component reaction of an arylaldehyde, malononitrile and a naphthol in H_2O and in the presence of recyclable base supported ionic liquid like-phase as an efficient green catalyst.

Keywords: 2-aminobenzo[h]chromene, 3-aminobenzo[f]chromene, three-component reaction, base supported ionic liquid like-phase (SILLP), naphthol

2-Aminochromenes are an important class of heterocyclic compounds with important biological activities and pharmacological properties. Fused-ring chromenes are biologically interesting compounds showing a wide spectrum of activities, such as antimicrobial,¹ mutagenicitical,² mitogenactivated protein kinase-activated protein kinase 2 inhibitory,³ antiproliferative,⁴ sex pheromonal,⁵ antitumoural⁶ and antioxidant activities.⁷

Traditional methods have been reported for the synthesis of 2-aminobenzo[*h*]chromene 3-aminobenzo[*f*]chromene and derivatives by condensation of α -naphthol or β -naphthol, an aldehyde and malononitrile using different homogeneous or heterogeneous catalysts, such as cetyltrimethyl-ammonium chloride/bromide,8,9 triethylbenzylammonium chloride,10 tetrabutylammonium bromide¹¹ and benzyl[2-(dimethylamino) ethyl]dimethylammonium chloride.12 Although each of these procedures has its own merits, some methods require the use of a hazardous amine-based catalyst and expensive catalysts, and have long reaction times, harsh reaction conditions, low yields, tedious work-up processes, environmental disposal problems or use organic solvents. Thus, we report a more convenient and practical method for the syntheses of 2-aminobenzo[h]chromene and 3-aminobenzo[f]chromene derivatives using base supported ionic liquid like-phase (SILLP)13 as a recyclable solid supported heterogeneous catalyst in water. Water is the cheapest and most abundant non-toxic chemical in nature. Aqua-mediated reactions have received considerable attention in organic synthesis due to environmental safety reasons. Reactions in aqueous media offer many advantages, such as simple operation and high efficiency, in many organic reactions that involve water soluble substrates and reagents.^{14,15} These advantages become even more attractive if such reactions can be conducted using ionic liquids in aqueous media.

Results and discussion

Ionic liquids have attracted increasing interest in the context of green synthesis in recent years. They have a unique combination of chemical and physical properties, including nonvolatility, nonflammability, thermal stability and controlled miscibility. They are the solvents of choice for a large array of organic reactions and can perform as a catalyst at the same time.^{16–22}

With respect to our continued interest in developing benign methods for the synthesis of biologically important products,^{23–28} we report a simple and high-yielding protocol for the synthesis of 2-amino-2-chromenes using SILLP as an efficient and recyclable heterogeneous base catalyst (Scheme 1).

In a preliminary experiment, to optimise the reaction conditions, SILLP was synthesised using Merrifield resin (1% cross linked, 200–400 mesh, 1.0–1.3 mmol g⁻¹) and the procedure employed by Luis and coworkers.¹³ A one-pot three-component reaction of equimolar amounts of



 $A_{1} = 4 - C1C_{6}H_{4}, 4 - B1C_{6}H_{4}, 5 - B1C_{6}H_{4}, 4 - MeC_{6}H_{4}, 4 - MeC_{6}H_{4}, 2 - Unleyl, 2 - Unlyl, 2 - O_{2}NC_{6}H_{4}, 3 - O_{2}NC_{6}H_{4}, 4 - O_{2}NC_{6}H_{4}, 4 - FC_{6}H_{4}, ... R^{1} = H, 4 - C1; R^{2} = H, 6 - Br$

Scheme 1 Synthesis of 2-amino- and 3-aminochromene derivatives using SILLP.

^{*} Correspondent. E-mail: m-chem41@guilan.ac.ir; mchem41@gmail.com

4-chlorobenzaldehyde (1), malononitrile and α -naphthol in the presence of SILLP (0.1 g mmol⁻¹ substrate) in refluxing water was selected as the model reaction. The reaction furnished the desired 2-aminochromene **5a** (Table 1, entry 1) in 80% yield. The effect of various solvents, such as H₂O, EtOH, THF, CH₃CN, CH₂Cl₂, DMF and 1,4-dioxane, and different temperatures was also examined, with water selected as the solvent of choice at refluxing temperature. Under optimised conditions, various arylaldehydes and naphthol derivatives were evaluated and the results are presented in Table 1. As

shown in Table 1, aromatic aldehydes with electron-donating and electron-withdrawing groups were rapidly condensed with malononitrile and various naphthols in the presence of SILLP and afforded the corresponding aminochromenes in high yield (80–97%) and short reaction time (10–20 min).

A plausible mechanism for the formation of products 5 and 6 is presented in Scheme 2, which proceeds through a Knoevenagel condensation of an arylaldehyde (1) and malononitrile (2) followed by Michael addition of a naphthol (3 or 4) to furnish the desired products.

Table 1 2-Aminochromene derivatives using SILLP under optimised conditions

-	4 010 11			rine/min	Field/ 70	Menning point/ C
1	$4-\text{CIC}_6\text{H}_4$	lpha-naphthol	5a	18ª	80	233-235 (lit. ²⁹ 231-233)
2	4-0 ₂ NC ₆ H ₄	lpha-naphthol	5b	20 ^a	82	239-241(lit.29 239.5-241)
3	$4-BrC_6H_4$	lpha-naphthol	5c	15ª	85	255–257
4	4-CH ₃ OC ₆ H ₄	lpha-naphthol	5d	10ª	87	195–197 (lit.30 195–196)
5	$4-CH_3C_6H_4$	lpha-naphthol	5e	15ª	86	206-208 (lit.31 205-207)
6	C_6H_5	lpha-naphthol	5f	10ª	97	222-224 (lit.32 218-219)
7	2-NO ₂ C ₆ H ₄	lpha-naphthol	5g	20 ^a	94	232-234 (lit.32 230-232)
8	3-0 ₂ NC ₆ H ₄	lpha-naphthol	5h	15ª	93	215-217 (lit.29 214.5-216)
9	$3-BrC_6H_4$	lpha-naphthol	5i	10ª	90	236-238
10	$4-FC_6H_4$	lpha-naphthol	5j	20 ^a	93	231-233 (lit.32 234-236)
11	3-0 ₂ NC ₆ H ₄	4-chloro- α -naphthol	5k	10 ^a	91	220–221
12	$4-0_{2}NC_{6}H_{4}$	4-chloro- α -naphthol	51	15ª	96	229–231
13	2-thienyl	4-chloro- α -naphthol	5m	10 ^a	91	235–236
14	2-furyl	4-chloro- α -naphthol	5n	10 ^a	90	227–229
15	2-furyl	lpha-naphthol	50	12	87	170-171 (lit.32 169-172)
16	C_6H_5	β -naphthol	6a	10 ^a	93	285-287 (lit.32 288-289)
17	$4-FC_6H_4$	β -naphthol	6b	12ª	90	230-232 (lit.32 232-233)
18	$4-CH_3C_6H_4$	β -naphthol	6c	15ª	85	268-270 (lit.31 270-272)
19	2-furyl	β -naphthol	6d	10 ^a	85	224-226 (lit.32 225-226)
20	$4-\text{CIC}_6\text{H}_4$	β -naphthol	6e	15	83	206-208 (lit.32 207-208.5)
21	4-0 ₂ NC ₆ H ₄	6-bromo-β-naphthol	6f	13	81	197–199

^alsolated yield.

^bRatio of aldehyde (1 mmol)/malononitrile (1 mmol)/naphthol (1 mmol)/SILLP (0.1 g); the reaction was accomplished in H₂O at 100 °C.



Scheme 2 A plausible mechanism for the synthesis of 5a-o and 6a-f.

Table 2 Comparison of the efficiency of different catalysts in the synthesis of $\rm 5f$ in refluxing $\rm H_2O$

-		- 2		
Entry	Ar	Catalyst	Time/min	Yield/%ª
1	C ₆ H ₅	SILLP	10	97 (this work)
2	C ₆ H ₅	[bmim]0H ^₅	60	90 (lit. ³³)
3	C ₆ H ₅	[bmim][PF ₆]°	120	86 (lit. ³⁴)
4	C_6H_5	CuSO ₄ ·5H ₂ 0	60	95 (lit. ³⁴)
2 3 4	С ₆ Н ₅ С ₆ Н ₅ С ₆ Н ₅	[bmim][PF ₆]⁰ CuSO₄·5H₂O	120 60	96 (lit. ³⁴) 95 (lit. ³⁴)

^alsolated yield.

^b1-Butyl-3-methylimidazolium hydroxide.

°1-Butyl-3-methylimidazolium hexafluorophosphate.

The reusability of the catalyst in the abovementioned reactions was also rechecked by using the preparation of **5a** and **6a** as model reactions. After five consecutive runs, the activity of the catalyst remained almost unchanged. After each run, the product mixture was dissolved in EtOH, the catalyst simply filtered off, washed with EtOH, dried and reused for the next four runs without appreciable loss of its catalytic activity.

To show the efficiency of the present method, we have compared our results for the synthesis of 2-aminobenzo[h] chromene **5f**, catalysed by SILLP, with other reported protocols in the literature. It is evident from the results in Table 2 that the reaction in the presence of SILLP produces the product in a much shorter reaction time and higher yield.

Conclusion

We have developed an efficient and eco-friendly protocol for the one-pot three-component synthesis of 2-aminobenzo[h] chromene and 3-aminobenzo[f]chromene derivatives using SILLP. The catalyst is recoverable and the efficiency of the catalyst remains almost unaltered after five cycles. Excellent yields, simple workup, short reaction times, easy recovery of the catalyst and the green nature of the procedure make this methodology attractive for large-scale synthesis.

Experimental

Melting points were measured on an Electrothermal 9100 apparatus. IR spectra were determined on a Shimadzo IR-470 spectrometer. ¹H NMR and ¹³C NMR spectra were recorded on a 400 and 500 MHz Bruker DRX-400 with DMSO- d_6 as solvent and TMS as an internal standard. Chemical shifts are expressed in ppm downfield from TMS. Elemental analyses were performed on a Carlo-Erba EA1110CNNO-S analyser and agreed (within 0.2%) with the calculated values. All chemicals were purchased from Merck and used without further purification. All solvents used were dried and distilled according to standard procedures.

Synthesis of 2-amino-4H-benzo[h]chromene and 3-amino-1Hbenzo[f]chromene derivatives (**5a–0**, **6a–f**); general procedure

A mixture of benzaldehyde 1 (1 mmol), malononitrile 2 (1 mmol), α -naphthol 3 (1 mmol) or β -naphthol (4) and SILLP (0.1 g) in water (5 mL) were refluxed for a certain time (monitored by TLC), and the solid product was dissolved in EtOH and filtered off to remove the catalyst. The catalyst was washed with EtOH and the combined organic solution was evaporated under reduced pressure to produce the desired products. The crude products were purified by recrystallisation from DMF-H₂O and washing with cold ethanol. The catalyst was dried and reused for four runs without appreciable loss of its catalytic activity.

2-*Amino-4-(4-bromophenyl)-3-cyano-4*H-*benzo[h]chromene* (**5c**): White powder; IR (KBr) (v_{max} cm⁻¹): 3450, 3333, 3198 (N–H), 2179 (C=N), 1662 (C=C), 1012 (C–Br), 806, 758 (aromatic C–H bend); ¹H NMR (500 MHz, DMSO- d_6): δ 8.25 (1H, d, J = 8.32 Hz, ArH), 7.88 (1H, d, J = 8.08 Hz, ArH), 7.63 (1H, t, J = 7.58 Hz, ArH), 7.59 (1H, d, J = 8.52 Hz, ArH), 7.56 (1H, t, J = 7.02 Hz, ArH), 7.50 (2H, d, J = 8.34 Hz, ArH), 7.21 (2H, d, J = 8.34 Hz, ArH), 7.20 (2H, s, NH₂), 7.08 (1H, d, J = 8.52 Hz, ArH), 4.93 (1H, s, C–H); ¹³C NMR (100 MHz,

DMSO- d_6): δ 161.0, 145.9, 143.7, 133.6, 132.5, 130.8, 128.6, 127.7, 127.6, 126.9, 124.9, 123.6, 121.6, 121.2, 121.0 118.2, 56.7, 41.2. Anal. calcd for C₂₀H₁₃BrN₂O (377.23): C, 63.68; H, 3.47; N, 7.43; found: C, 63.49; H, 3.32; N, 7.28%.

2-*Amino-4-(3-bromophenyl)-3-cyano-4*H-*benzo[h]chromene* (5i): White powder; IR (KBr) (v_{max} cm⁻¹): 3445, 3334, 3190 (N−H), 2189 (C≡N), 1656 (C=C), 1016 (C−Br), 810, 756 (aromatic C−H bend); ¹H NMR (500 MHz, DMSO-*d*₆): δ 8.25 (1H, d, *J* = 8.4 Hz, ArH), 7.92 (d, *J* = 8.0 Hz, ArH), 7.68–7.58 (3H, m, ArH), 7.47–7.44 (2H, m, ArH), 7.33–7.25 (2H, m, ArH), 7.15 (1H, d, *J* = 8.8 Hz, ArH), 4.98 (1H, s, C−H); ¹³C NMR (100 MHz, DMSO-*d*₆): δ 160.77, 160.73, 148.87, 143.26, 133.27, 131.51, 130.74, 130.40, 128.19, 127.39, 127.25, 126.50, 124.58, 123.22, 122.45, 121.23, 120.81, 117.68, 56.15, 40.88. Anal. calcd for C₂₀H₁₃BrN₂O (377.23): C, 63.68; H, 3.47; N, 7.43; found: C, 63.51; H, 3.28; N, 7.24%.

2-*Amino*-6-*chloro*-4-(3-*nitrophenyl*)-3-*cyano*-4H-*benzo*[h] *carbonitrile* (**5k**): Yellow powder; IR (KBr) (v_{max} cm⁻¹): 3369, 3315 (N−H), 2196 (C≡N), 1660 (C=C), 1529, 1352 (NO₂), 857, 811, 762 (aromatic C−H bend); ¹H NMR (400 MHz, DMSO-*d*₆): δ 8.36 (1H, m, ArH), 8.19 (1H, t, *J* =1.8, ArH), 8.16–8.12 (2H, m, ArH), 7.82–7.77 (3H, m, Ar–H), 7.67 (1H, t, *J* = 8.0 Hz, ArH), 7.44 (s, NH₂), 7.40 (1H, s, ArH), 5.22 (1H, s, C–H); ¹³C NMR (100 MHz, DMSO-*d*₆): δ 160.7, 160.65, 148.5, 147.7, 142.7, 135.1, 131.0, 129.9, 128.9, 128.3, 126.4, 126.1, 124.4, 124.3, 122.8, 122.6, 122, 120.5, 117.9, 55.7. Anal. calcd for C₂₀H₁₂ClN₃O₃ (377.78): C, 63.59; H, 3.20; N, 11.12; found: C, 63.43; H, 3.09; N, 11.04%.

2-Amino-6-chloro-4- (4-nitrophenyl)-3-cyano-4H-benzo[h] carbonitrile (**5**I): Yellow powder; IR (KBr) (v_{max} cm⁻¹): 3445, 3325, 3202 (N–H), 2191 (C≡N), 1661 (C=C), 1514, 1348 (NO₂), 1109 (C–Cl), 874, 812, 758, 698 (aromatic C–H bend); ¹H NMR (500 MHz, DMSO- d_6): δ 8.33 (d, *J* = 7.87 Hz, 1H, ArH), 8.17 (d, *J* = 8.80 Hz, 2H, ArH), 8.03 (d, *J* = 8.50 Hz, 1H, ArH), 7.75–7.68 (m, 2H, ArH), 7.55 (d, *J* = 8.80 Hz, 2H, ArH), 7.42 (s, 2H, NH₂), 7.27 (s, 1H, ArH), 5.13 (s, 1H, C–H); ¹³C NMR (125 MHz, DMSO- d_6): δ 161.0, 153.2, 147.5, 143.2, 130.4, 129.9, 129.3, 128.7, 126.8, 126.5, 125.0, 124.8, 124.7, 122.4, 120.8, 118.0, 56.0, 41.2. Anal. calcd for C₂₀H₁₂CIN₃O₃ (377.78): C, 63.59; H, 3.20; N, 11.12; found: C, 63.40; H, 3.02; N, 11.01%.

2-*Amino*-6-*chloro*-4-(*thiophen*-2-*yl*)-4H-*benzo*[h]*chromene*-3*carbonitrile* (**5m**): White powder; IR (KBr) (v_{max} cm⁻¹): 3445, 3325 (N–H), 2195 (C≡N), 1664 (C=C), 761 (C–S), 802, 761, 705 (aromatic C–H bend); ¹H NMR (400 MHz, DMSO-*d*₆): δ 8.32 (1H, m, ArH), 8.14 (1H, m, ArH), 7.79 (2H, m, ArH), 7.49 (1H, s, ArH), 7.43 (1H, dd, *J* = 1.2, 5.2 Hz, ArH), 7.44 (1H, s, ArH), 7.36 (s, NH₂), 7.16 (1H, dd, , *J* = 0.8, 3.6 Hz, ArH), 6.99 (1H, dd, *J* = 3.6, 5.2 Hz, ArH), 5.32 (1H, s,); ¹³C NMR (100 MHz, DMSO-*d*₆): δ 160.5, 160.4, 150.6, 142.1, 129.8, 128.8, 128.2, 127.5, 126.3, 126.2, 126.16, 125.4, 124.33 124.3, 121.9, 120.6, 118.8, 56.8. Anal. calcd for C₁₈H₁₁ClN₂OS (338.81): C, 63.81; H, 3.27; N, 8.27; found: C, 63.72; H, 3.11; N, 8.09.

2-*Amino*-6-*chloro*-4- (*furan*-2-*yl*)-4H-*benzo*[h]*chromene*-3*car*bonitrile (**5n**): White powder; IR (KBr) (v_{max} cm⁻¹): 3449, 3330 (N–H), 2186 (C=N), 1658 (C=C), 1257 (C–O), 857, 758, 734 (aromatic C–H bend); ¹H NMR (400 MHz, DMSO-*d*₀): δ 8.31 (1H, m, ArH), 8.15 (1H, m, ArH), 7.78 (2H, m, ArH), 7.57 (1H, dd, *J* = 1.0, 1.8 Hz, ArH), 7.44 (1H, s, ArH), 7.34 (s, NH₂), 6.41 (1H, dd, *J* = 1.6, 3.2 Hz, ArH), 6.31 (1H, d, *J* = 3.2 Hz, ArH), 5.11 (1H, s); ¹³C NMR (100 MHz, DMSO-*d*₀): δ 161.1, 161.1, 156.2, 143.4, 142.9, 129.9, 128.8, 126.1, 124.4, 124.3, 121.8, 120.5, 116.5, 111.0, 107.0, 53.5. Anal. calcd for C₁₈H₁₁ClN₂O₂ (322.75): C, 66.99; H, 3.44; N, 8.68; found: C, 66.85; H, 3.31; N, 8.52%.

3-Amino-8-bromo-2-cyano-1-(4-nitrophenyl)-1H-benzo[f] chromene (6f): Yellow powder; IR (KBr) (ν_{max} cm⁻¹): 3391, 3325, 3200 (N−H), 2191 (C≡N), 1653 (C=C), 1520, 1346 (NO₂), 1086 (C−Br), 840, 827 (aromatic C−H bend); ¹H NMR (500 MHz, CDCl₃): δ 7.69 (2H, d, *J* = 8.28 Hz, ArH), 7.55 (1H, s, ArH), 7.33 (1H, d, *J* = 8.96 Hz ArH), 7.03 (1H, d, *J* = 8.88 Hz, ArH), 6.96 (1H, d, *J* = 8.96 Hz, ArH), 6.90−6.84 (3H, m, ArH), 4.87 (1H, s, C−H), 4.50 (2H, s, NH₂); ¹³C NMR (100 MHz, CDCl₃): δ 163.1, 159.3, 151.4, 147.8, 147.4, 133.0, 131.3, 131.2, 129.8, 129.4, 128.5, 125.3, 124.9, 119.9, 118.4, 114.2, 60.7, 39.1. Anal. calcd for C₂₀H₁₂BrN₃O₃ (422.23): C, 56.89; H, 2.86; N, 9.95; found: C, 56.74; H, 2.75; N, 9.82.

Acknowledgements

The authors are grateful to the Research Council of University of Guilan.

Received 10 November 2016; accepted 9 December 2016 Paper 1604421 doi: 10.3184/174751917X14815427219202 Published online: 22 January 2017

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