Journal of the Iranian Chemical Society (2018) 15:2147–2157
https://doi.org/10.1007/s13738-018-1408-x

ORIGINAL PAPER

An affordable DABCO-based ionic liquid efficiency in the synthesis of 3-amino-1-aryl-1*H*-benzo[*f*] chromene-2-carbonitrile, 1-(benzothiazolylamino)phenylmethyl-2-naphthol, and 1-(benzoimidazolylamino)phenylmethyl-2-naphthol derivatives

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Received: 13 February 2018 / Accepted: 21 May 2018 / Published online: 26 May 2018

Abstract
In this study, $[H_2\text{-DABCO}][HSO_4]_2$ is applied as an acidic ionic liquid to exclude some disadvantages of former methods reported for the synthesis of 3-amino-1-aryl-1*H*-benzo[*f*]chromene-2-carbonitrile, 1-(benzothiazolylamino)phenylmethyl-2-naphthol, and 1-(benzoimidazolylamino)phenylmethyl-2-naphthol derivatives. In the presence of this catalyst, all reactions are performed under neat conditions during short reaction times in good-to-high yields. Ease of preparation and recyclability of the catalyst are the other important advantages of this method.

Keywords Acidic ionic liquid · 1,4-Diazobicyclo[2,2,2] octane (DABCO) · 3-Amino-1-aryl-1*H*-benzo[*f*] chromene-2-carbonitrile derivatives · 1-(Benzothiazolylamino)phenylmethyl-2-naphthol · 1-(Benzoimidazolylamino)phenylmethyl-2-naphthol derivatives · Multi-component reaction

Introduction

According to global environmental concern and green chemistry roles, its important issue to using less hazardous chemical solvents, reduce waste materials, and apply catalysis in organic reactions. One of the powerful synthetic tools that use to develop preparation of some organic compounds is multi-component reactions (MCRs). The importance of this category of reactions is that they can facilitate synthesis of some important heterocyclic and bioactive compounds in just one straightforward step; moreover, these reactions are in correlation with some green chemistry principles [1]. 2-Amino-4*H*-chromene, and benzimidazole and benzothiazole derivatives are two important classes of heterocyclic compounds that can be synthesized via this strategy.

Compounds, which possess 2-amino-4*H*-chromene structure moiety, are important heterocycles because of their remarkable biological and pharmacological properties such as antibacterial [2], anticancer [3–5], antimicrobial [6, 7], antiviral [8, 9], and central nervous system (CNS) [10] activities.

Benzimidazole and benzothiazole derivatives are a group of attractive heterocyclic pharmacophores which demonstrate a wide range of biological properties, including antimicrobial, antibacterial, antitumor, anticancer, anti-inflammatory, and analgesic [11–25] activities. These compounds also are known in drugs designed for targeting DNA and DNA-associated processes [26]. In addition, benzimidazole nucleus exists in some natural bioactive compounds like vitamin B_{12}.

Because of the above-mentioned important properties many of methods have been reported for the synthesis of 2-amino-4*H*-chromene, benzimidazole and benzothiazole derivatives [27–33]. These methods are also useful, but most of them suffer from disadvantages such as harsh reaction conditions, waste primary materials, use of toxic solvents, and long reaction conditions.

Nowadays, due to the significant properties of ionic liquids, scientists have developed the applications of these compounds as green solvents and potential catalysts in various fields. Ionic liquids have attracted attentions because that their unique

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physical properties such as low viscosity, non-volatile, high solubility capability, and ease of preparation made them different from ordinary solvents [34, 35]. In addition, they can be prepared easily from commercially available materials. In this study and in continuation of our research group successive efforts in the synthesis of novel ionic liquid catalysts [36–41] and to solve the restrictions which have been observed in the synthesis of 3-amino-1-aryl-1*H*-benzo[*f*] chromene-2-carbonitrile, 1-(benzothiazolylamino)phenylmethyl-2-naphthol, and 1-(benzoimidazolylamino)phenylmethyl-2-naphthol derivatives, we wish to report the applicability of our recently introduced acidic DABCO-based ionic liquid [42] in the acceleration of the synthesis of these types of compounds.

Experimental

General

All the chemicals were used in this procedure including aldehydes, 2-naphthol, malononitrile, 2-aminobenzimidazole, and 2-aminobenzothiazole were purchased from Merck Chemical Company (Munich) and were used without further purification. The purity determination of the substrates and reactions was monitored by thin-layer chromatography (TLC) on silica-gel polygram SILG/UV 254 Plates.

DABCO (CAS: 280-57-9, MW: 112.17 g/gmol, assay ≥ 99% w/w %) and sulfuric acid (CAS: 7664-93-9, MW: 98.08 g/gmol, assay ≥ 99.99 v/v %) were purchased from Sigma-Aldrich Company (Mumbai). Both assays were reported by company and were not examined more. All solvents were prepared from Merck Chemical Company (Munich) and were kept in isolated conditions as well to minimize the absorption of atmosphere moisture. Moreover, they had gotten distilled before being used. Products were characterized by their physical constants, comparison with reported in the literature samples and IR and NMR spectroscopy.

Melting points were measured by electrothermal IA9100 melting point apparatus in capillary tubes. The starting temperature of the approximate melting range was input *via* the keyboard and the melting point range was spotted visually. FT-IR spectra were recorded on a Perkin-Elmer spectrum BX series and KBr pellets were used for solid samples. ^{1}H NMR and ^{13}C NMR spectra were determined on Bruker AV-400 using TMS (0.00 ppm) as internal standard and DMSO-d_6 as solvent.

Preparation of 1,4-diazabicyclo[2.2.2] octane-1,4-diium hydrogen sulfate {[H_2-DABCO][HSO_4]$_2$} [39]

In a 100 mL round-bottomed flask, 1,4-diazabicyclo[2.2.2] octane (DABCO) (1.05 g, 9.4 mmol) was dissolved in 50 mL dry dichloromethane. Then, this mixture was stirred in an ice-bath for 1 min and a stoichiometric amount of sulfuric acid (99.99%, 1 mL, 18.8 mmol) was added drop-wise to it within 25 min. Then, the mixture was heated to reflux with constant stirring for 24 h. In continue, the solvent was decanted and the resulting white solid was frequently washed with diethyl ether (3 × 15 ml_2) to obliterate non-ionic

Table 1 Optimization of the amount of the catalyst, temperature, and solvent in the synthesis of 3-amino-1-(4-chlorophenyl)-1*H*-benzo[*f*] chromene-2-carbonitrile (5b) [entries 1–8] and 1-benzothiazolylamino-(4-chlorophenylmethyl)-2-naphthol (6b) [entries 9–18]

Entry	Catalyst (mol%)	Solvent	Temp. (°C)	Time (min.)	Conversion [Yield (%)][a]
1	–	–	100	90	Trace
2	–	H_2O	Reflux	90	Trace
3	0.0048	–	100	60	Not completed
4	0.0048	–	120	40	100(75)
5	0.0048	H_2O	Reflux	90	Not completed
6	0.0065	H_2O	Reflux	90	Not completed
7	0.0065	–	100	25	100(93)
8	0.0097	–	100	5	100(96)
9	–	–	100	90	Trace
10	–	H_2O	Reflux	90	Trace
11	–	H_2O:C_2H_5OH (1:1)	80	90	Trace
12	0.0065	C_2H_5OH	Reflux	90	Not completed
13	0.0065	CH_3CN	Reflux	90	Not completed
14	0.0065	H_2O	Reflux	90	100(73)
15	0.0065	H_2O:C_2H_5OH (1:1)	80	90	100(70)
16	0.0065	–	100	50	100(85)
17	0.0065	–	120	30	100(90)
18	0.0097	–	120	15	100(95)

[a]Isolated yields

residues. After that, the obtained ionic liquid was dried under vacuum to achieve $[H_2\text{-DABCO}][HSO_4]_2$ in 95% yield (2.74 g) (Scheme 1). The purity of the product was determined by melting point, Mass, FT-IR, ^{1}H NMR, and ^{13}C NMR.

Spectral data for $[H_2\text{-DABCO}][HSO_4]_2$: white solid; M.p. 65 °C; MS: $m/z = 308(M^+)$; FT-IR (KBr, cm^{-1}) ν_{max}: 3400–2400, 1229, 1165, 849, 576; ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 3.59 (s, 6H), 6.64 (s, 1H, NH), 13.88 (s, 1H, HSO_4); ^{13}C NMR (100 MHz, DMSO-d_6): δ (ppm) 43.4.

General procedure for the synthesis of 3-amino-1-aryl-1*H*-benzo[*f*] chromene-2-carbonitrile derivatives

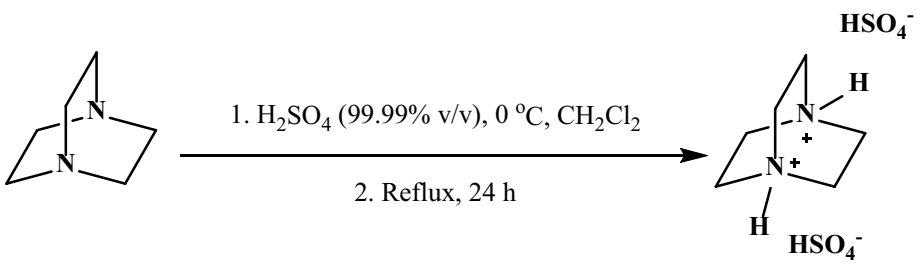
In a 25 mL round-bottom flask, a mixture of the requested aromatic aldehyde 1 (1 mmol), 2-naphthol 2 (1 mmol), malononitrile 3 (1 mmol), and $[H_2\text{-DABCO}][HSO_4]_2$ (0.030 g, 0.097 mmol) was stirred under solvent-free conditions at 100 °C for a certain time. After completion of the reaction, as identified by TLC [ethyl acetate:*n*-hexane (3:7)], the solid product was filtered and washed with water to separate the catalyst. Then, the precipitate was recrystallized from ethanol to obtain the pure product (5a–5l). The spectral data of the new compound are as follows.

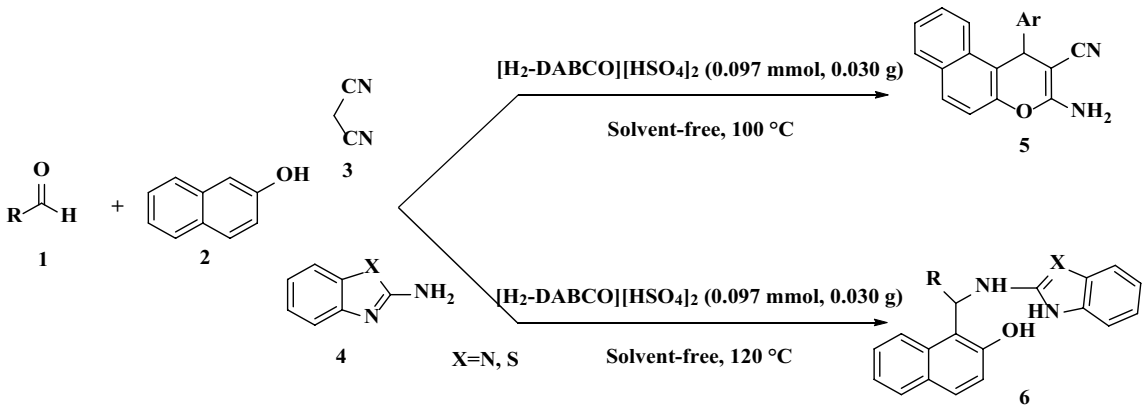
3-Amino-1-(2-fluorophenyl)-1*H*-benzo[*f*] chromene-2-carbonitrile (5 l). Mp = 286–288 °C; IR (KBr) ν_{max}/cm^{-1}: 3440, 3342, 3089, 2921, 2180, 1645,1586, 1482, 1410, 1228, 1178, 810, 751; ^{1}H NMR (400 MHz, DMSO-d_6) $\delta = 7.95$ (d, $J = 9.2$ Hz, 1H), 7.93 (d, $J = 9.2$ Hz, 1H), 7.77 (d, $J = 8$ Hz, 1H), 7.41–7.51 (td, $J_1 = 7.6$ Hz, $J_2 = 1.2$ Hz, 2H), 7.34 (d, $J = 9.2$ Hz, 1H), 7.16–7.25 (m, 3H), 7.09 (ddd, $J_1 = 13.4$ Hz, $J_2 = 12$ Hz, $J_3 = 1.2$ Hz, 3H), 5.54 (s, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 160.6, 159.8 (d, $^1J_{CF} = 244$ Hz), 147.5, 132.4, 132.3, 131.2, 130.5, 130.2, 130.1, 129.3(d, $^3J_{CF} = 7$ Hz), 129.1, 127.8, 125.4, 123.1, 120.7, 117.2, 116.2 (d, $^2J_{CF} = 21$ Hz), 114.7, 56.4, 32.9 ppm.

General procedure for the synthesis of 1-(benzothiazolylamino)phenylmethyl-2-naphthol and 1-(benzoimidazolylamino)phenylmethyl-2-naphthol derivatives.

To a mixture of the requested aromatic aldehyde 1 (1 mmol), 2-naphthol 2 (1 mmol) and 2-aminobenzimidazole/or 2-aminobenzothiazole 4 (1 mmol), $[H_2\text{-DABCO}][HSO_4]_2$ (0.030 g, 0.097 mmol) was added, and then, the mixture was stirred at 120 °C for an appropriate time. The progress of the reaction was monitored by TLC [ethyl acetate: *n*-hexane (3:7)]. After completion of the reaction, the precipitate was filtered and washed with water to separate the catalyst. Finally, the crude products were purified by recrystallization from ethanol to obtain the pure product (6a–6m).

Scheme 1 Preparation of $[H_2\text{-DABCO}][HSO_4]_2$





Scheme 2 Synthesis of 3-amino-1-aryl-1*H*-benzo[*f*] chromene-2-carbonitrile (5), 1-(benzothiazolylamino)phenylmethyl-2-naphthol, and 1-(benzoimidazolylamino)phenylmethyl-2-naphthol (6) derivatives in the presence of $[H_2\text{-DABCO}][HSO_4]_2$ as the catalyst

Table 2 Synthesis of 3-amino-1-aryl-1*H*-benzo[*f*] chromene-2-carbonitrile, 1-(benzothiazolylamino)phenylmethyl-2-naphthol and 1-(benzoimidazolylamino)phenylmethyl-2-naphthol derivatives using $[H_2\text{-DABCO}][HSO_4]_2$ as the catalyst

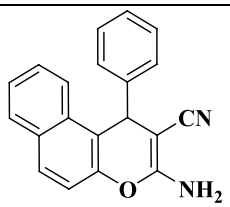
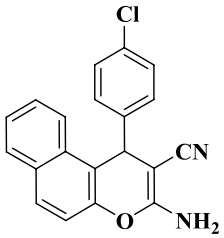
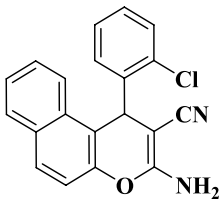
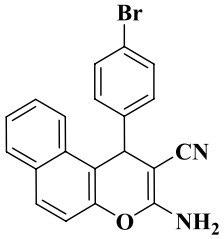
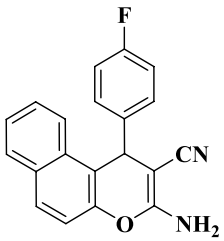
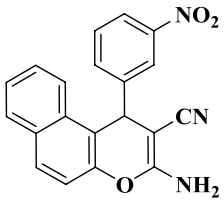
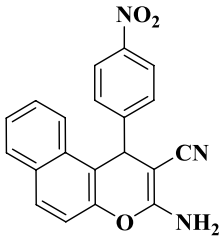
Entry	Aldehyde	Product		Time (min.)	Yield (%)[a]	M.P. (°C)		References
						Found	Reported	
1	C_6H_5CHO		5a	5	93	275–277	279–280	[43]
2	$4\text{-}ClC_6H_4CHO$		5b	5	96	208–210	207–208	[44]
3	$2\text{-}ClC_6H_4CHO$		5c	5	95	268–270	271–272	[43]
4	$4\text{-}BrC_6H_4CHO$		5d	8	94	237–239	238–240	[44]
5	$4\text{-}FC_6H_4CHO$		5e	12	91	227–229	228–229	[43]
6	$3\text{-}NO_2C_6H_4CHO$		5f	10	91	235–237	235–236	[43]
7	$4\text{-}NO_2C_6H_4CHO$		5g	8	93	170–172	176–178	[43]

Table 2 (continued)

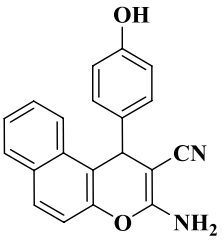
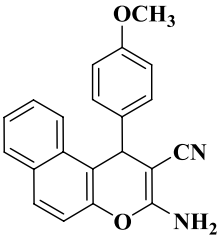
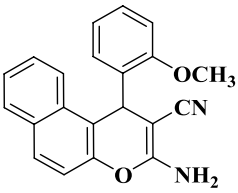
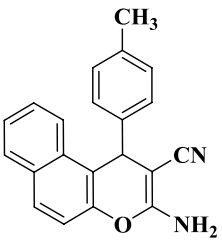
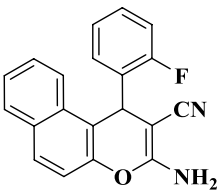
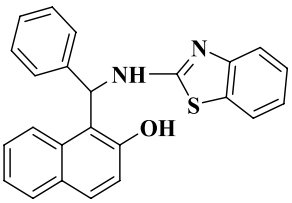
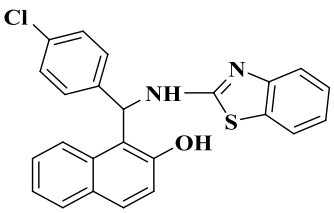
Entry	Aldehyde	Product		Time (min.)	Yield (%)[a]	M.P. (°C)		References
						Found	Reported	
8	4-OHC_6H_4CHO		5h	15	90	283–285	288–290	[45]
9	4-$OCH_3C_6H_4CHO$		5i	15	90	183–185	185–187	[44]
10	2-$OCH_3C_6H_4CHO$		5j	20	88	218–220	219–222	[46]
11	4-$CH_3C_6H_4CHO$		5k	12	92	270–272	273–274	[43]
12	2-FC_6H_4CHO		5l	15	96	286–288	New	–
13	C_6H_5CHO		6a	20	93	200–202	201–203	[47]
14	4-ClC_6H_4CHO		6b	15	95	205–207	208–210	[47]

Table 2 (continued)

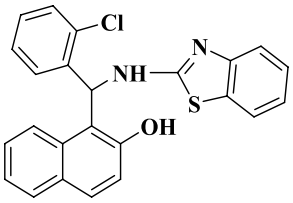
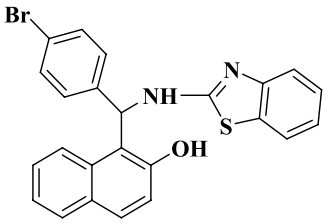
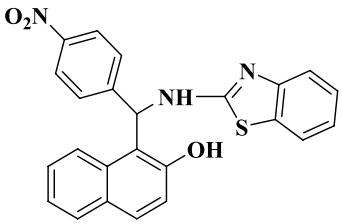
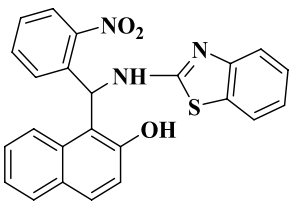
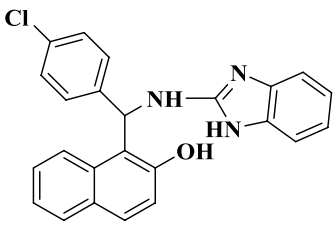
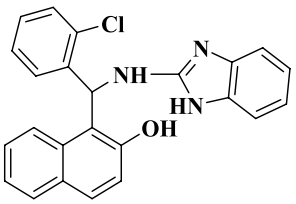
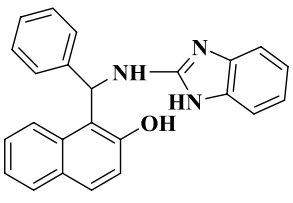
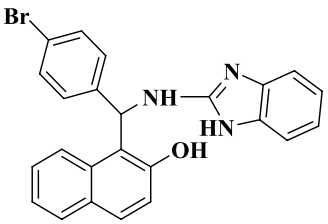
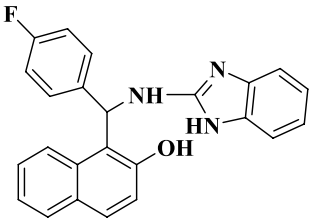
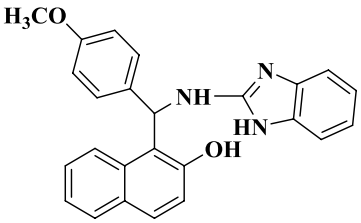
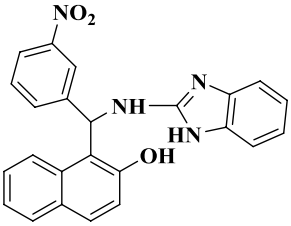
Entry	Aldehyde	Product		Time (min.)	Yield (%)[a]	M.P. (°C)		References
						Found	Reported	
15	2-ClC_6H_4CHO		6c	18	91	187–189	188–190	[47]
16	4-BrC_6H_4CHO		6d	10	94	204–206	211–213	[47]
17	4-$NO_2C_6H_4CHO$		6e	20	92	188–190	186–188	[47]
18	2-$NO_2C_6H_4CHO$		6f	20	90	220–222	218–220	[47]
19	4-ClC_6H_4CHO		6g	23	92	188–190	195–197	[48]
20	2-ClC_6H_4CHO		6h	30	89	146–148	144–146	[48]
21	C_6H_5CHO		6i	25	88	191–193	195–196	[48]

Table 2 (continued)

Entry	Aldehyde	Product		Time (min.)	Yield (%)[a]	M.P. (°C)		References
						Found	Reported	
22	4-BrC_6H_4CHO		6j	20	93	200–202	199–201	[48]
23	4-FC_6H_4CHO		6k	35	90	197–199	195–196	[48]
24	4-$OCH_3C_6H_4CHO$		6l	35	88	170–172	171–173	[48]
25	3-$NO_2C_6H_4CHO$		6 m	30	89	198–200	197–198	[48]

[a]Isolated yields

Results and discussion

At the first step and to achieve the optimum reaction conditions, the synthesis of 3-amino-1-(4-chlorophenyl)-1*H*-benzo[*f*] chromene-2-carbonitrile (5b) and 1-benzothiazolylamino-(4-chlorophenylmethyl)-2-naphthol (6b) was selected as two model reactions in various conditions such as different amounts of the catalyst [[H_2-DABCO][HSO_4]$_2$], and various solvents and temperatures. The results are summarized in Table 1. As it is clear from this table, the best yield and time for the synthesis of the compound 5b can be resulted in the presence of 0.030 g of the catalyst, in the absence of solvent at 100 °C, while, in the case of the compound 6b, the best results can be obtained in the presence of 0.030 g of the catalyst, under solvent-free conditions at 120 °C (Scheme 2). As shown in Table 1, for both of the reactions, a trace amount of the product is obtained when the reactions were proceeded in the absence of the catalyst.

After optimization of the reactions conditions and to improve these protocols, different types of aromatic aldehydes with electron-donating and electron-withdrawing functional groups were used for the synthesis of 3-amino-1-aryl-1*H*-benzo[*f*] chromene-2-carbonitrile, 1-(benzothiazolylamino)phenylmethyl-2-naphthol, and 1-(benzoimidazolylamino)phenylmethyl-2-naphthol derivatives under the optimized conditions. As represented in Table 2, various aldehydes with both electron-donating and electron-withdrawing substituents are converted to the corresponding products in short times and good-to-excellent yields.

Table 3 Compared performance of various catalysts with $[H_2\text{-DABCO}][HSO_4]_2$ in the synthesis of 3-amino-1-aryl-1*H*-benzo[*f*]chromene-2-carbonitrile [entries 1–9] and 1-(benzothiazolylamino)phenylmethyl-2-naphthol [entries 10–16] derivatives of 4-chlorobenzaldehyde

Entry	Catalyst	Amount (mol%)	Conditions	Time (min.)	Yield (%)	TON	TOF (min^{-1})	References
1	Morpholine	30	H_2O/r.t	180	87	290	1.61	[28]
2	$CuSO_4{\cdot}5H_2O$	5	H_2O/reflux	80	85	17	21.25	[46]
3	$H_{14}[NaP_5W_{30}O_{110}]$	0.030 g	H_2O/reflux	240	91	–	–	[27]
4	Na_2CO_3	0.01	Solvent-free/125 °C	60	100	1×10^6	16666.67	[49]
5	$Fe(HSO_4)_3$	0.01	CH_3CN/reflux	240	85	85×10^4	3541.67	[50]
6	DABCO	30	C_2H_5OH/r. t	120	72	240	0.02	[51]
7	Nanozeolite CP	0.01 g	H_2O/reflux	20	92	–	–	[52]
8	Methanesulfonic acid	0.1	CH_3CN/reflux	240	91	91×10^3	397.16	[53]
9	$[H_2\text{-DABCO}][HSO_4]_2$	0.0097	Solvent-free/100 °C	5	96	989690.72	197938.14	This work
10	HPA	3	H_2O/45 °C with ultrasonic irradiation	110	90	3000	27.27	[54]
11	LiCl	1.1	H_2O/90 °C	360	92	8363.63	23.23	[55]
12	TCCA	10	Solvent-free/80 °C	20	96	960	48	[56]
13	SDS	20	H_2O/100 °C	60	88	440	7.33	[57]
14	KOH/MgO	0.050 g	Solvent-free/70 °C	180	92	–	–	[30]
15	Oxalic acid	30	Solvent-free/80 °C	4	91	303.33	75.83	[29]
16	$[H_2\text{-DABCO}][HSO_4]_2$	0.0097	Solvent-free/120 °C	15	95	979381.44	65292.09	This work

To show the merits of our catalyst activity, in Table 3, the results obtained from the synthesis of 3-amino-1-aryl-1*H*-benzo[*f*]chromene-2-carbonitriles and 1-(benzothiazolylamino)phenylmethyl-2-naphthols catalyzed by $[H_2\text{-DABCO}][HSO_4]_2$ are compared with other reported results in the literatures. As shown in this table, this method avoids from some of disadvantages associated with the other procedures such as low yields, long reaction times, hard conditions for the catalyst preparation, using ultrasonic irradiation, and high catalyst loading.

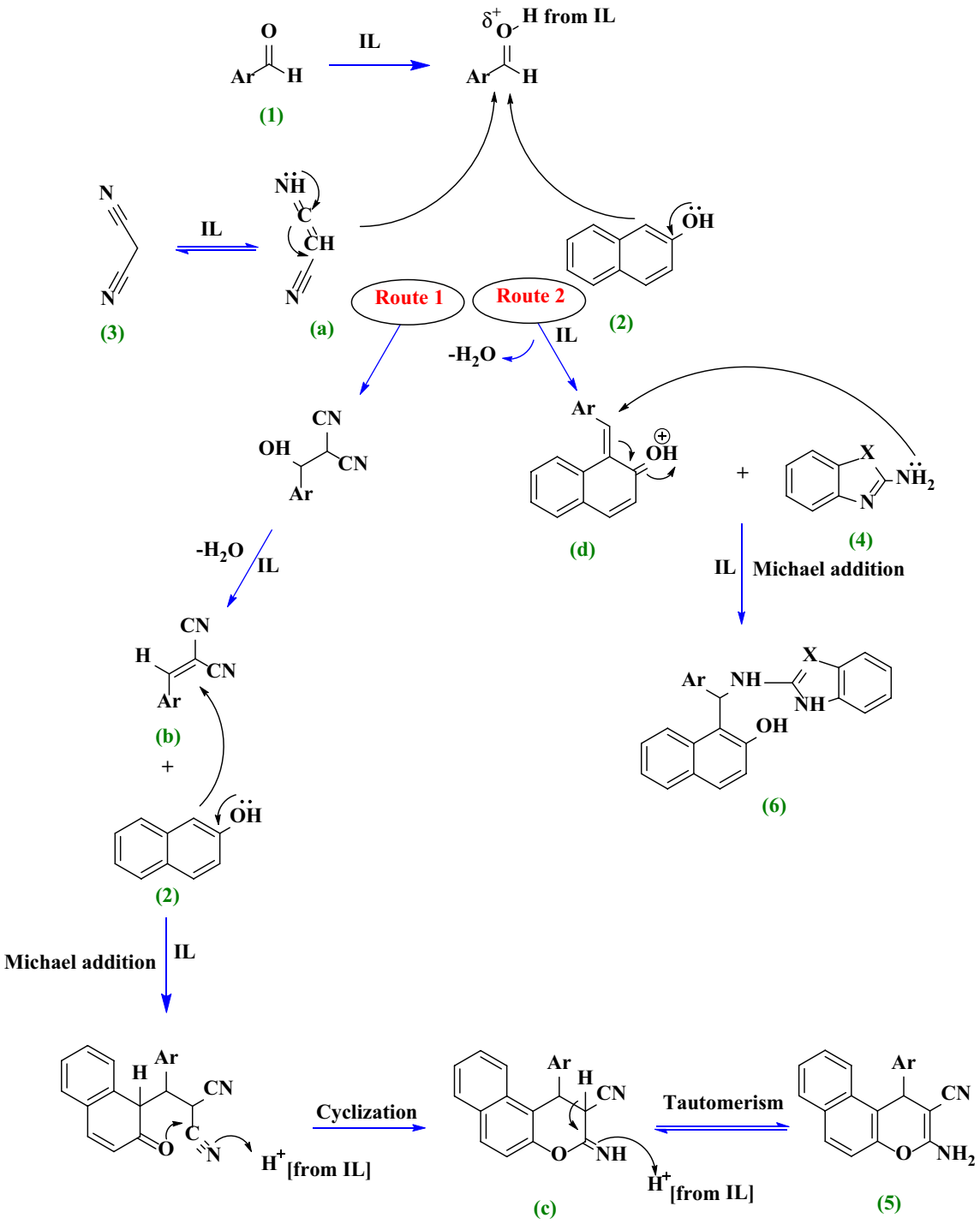
The proposed mechanism for these reactions is shown in Scheme 3. According to this mechanism, at first, carbonyl group of the aldehyde (1) is activated in the presence of $[H_2\text{-DABCO}][HSO_4]_2$. After that, the mechanism is divided into two distinct routes. In route 1, this activated carbonyl group is attacked by nucleophilic compound (a) to give the cyanoolefin (b). In continue, compound (b) is attacked by 2-naphthol (2) to produce Michael-addition product. Then, after cyclization and tautomerization steps, the corresponding product (5a–5l) is produced.

In route 2, the activated carbonyl group can be attacked by 2-naphthol (2), to give compound (d). Then, compound (d) is attacked by 2-aminobenzimidazole/2-aminobenzothiazole (4) to produce compounds 6a–6m.

To investigate the recoverability of the catalyst, the reactions of 3-amino-1-(4-chlorophenyl)-1*H*-benzo[*f*]chromene-2-carbonitrile (5b) and 1-benzothiazolylamino-(4-chlorophenylmethyl)-2-naphthol (6b) were studied again. After completion of the reactions, the products were washed with water to remove the catalyst, and then, the filtrate was evaporated under vacuum up to 70 °C to obtain the recovered catalyst. Ultimately, the obtained catalyst was washed with diethyl ether, and dried and reused for the same reactions. This procedure was repeated four times for each reaction and the products were obtained by the recovered catalyst with the least change in the reaction times and yields. The results are demonstrated in Figs. 1 and 2.

Conclusion

In this study, $[H_2\text{-DABCO}][HSO_4]_2$ is used as a novel acidic ionic liquid catalyst in the promotion of multicomponent synthesis of 3-amino-1-aryl-1*H*-benzo[*f*]chromene-2-carbonitriles, 1-(benzothiazolylamino)phenylmethyl-2-naphthols and 1-(benzoimidazolylamino)phenylmethyl-2-naphthols. The most appealing feature of this catalyst is using accessible and inexpensive materials to synthesize the catalyst. Besides, the presented protocols represent some advantages such as avoiding the use of toxic solvents, ease of preparation of the catalyst, excellent yields, simple work-up, and recyclability of the catalyst. Moreover, the results clearly show that this method can tolerate a wide range of various substituted aldehydes including electron-withdrawing or electron-donating groups.



Scheme 3 Proposed mechanism for the one-pot synthesis of 3-amino-1-aryl-1*H*-benzo[*f*] chromene-2-carbonitrile, 1-(benzothiazolylamino)phenylmethyl-2-naphthol, and 1-(benzoimidazolylamino)phenylmethyl-2-naphthol derivatives using $[H_2\text{-DABCO}][HSO_4]_2$ as the catalyst

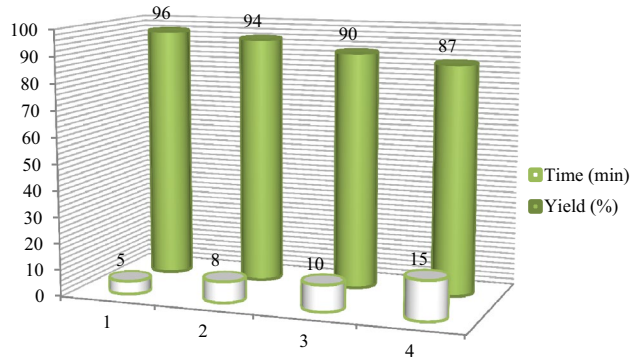


Fig. 1 Reusability of $[H_2\text{-DABCO}][HSO_4]_2$ in the synthesis of 3-amino-1-(4-chlorophenyl)-1*H*-benzo[*f*] chromene-2-carbonitrile (5b)

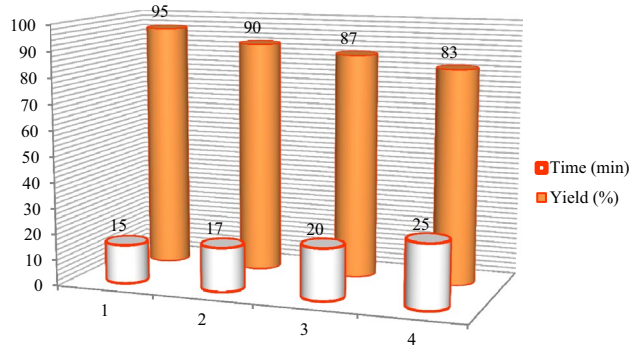


Fig. 2 Reusability of $[H_2\text{-DABCO}][HSO_4]_2$ in the synthesis of 1-benzothiazolylamino-(4-chlorophenylmethyl)-2-naphthol (6b)

Acknowledgements We are thankful to the University of Guilan Research Council for the partial support of this work.

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