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Potassium phthalimide-catalysed one-pot multi-component reaction for efficient synthesis of amino-benzochromenes in aqueous media

Hamzeh Kiyani*, Fatemeh Ghorbani

School of Chemistry, Damghan University, Damghan 36715-364, Iran

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2-Amino-4-aryl-4*H*-benzo[*h*]chromenes and 3-amino-1-aryl-1*H*-benzo[*f*]chromenes were prepared by treating cyano-methylene compounds (malononitrile or ethyl cyanoacetate), substituted aromatic aldehydes, and naphtholic compounds in the presence of potassium phthalimide as a green, mild, efficient, and commercially available organocatalyst in aqueous media. The procedure was readily conducted and affords remarkable advantages such as safety, short reaction times, environmentally benign milder reaction conditions, no organic solvent required, and high yields.

Keywords: three-component process, amino-benzochromene, potassium phthalimide, nitrile compounds

Introduction

Since the first multi-component reaction (MCR) was discovered by Strecker (1850), multi-component reactions (MCRs) have proved to be valuable tools for the synthesis of a wide variety of useful chemical materials, including natural and pharmaceutically active compounds (Ramn & Yus, 2005; Ulaczyk-Lesanko & Hall, 2005; Murthy et al., 2009; Singh & Chowdhury, 2012; Karami et al., 2013). Recently, multi-component approaches have attracted great interest due to their high efficiency, mild conditions, simple eco-friendly procedures, and minimal time-demands (Langer, 2002; Simon et al., 2004; Brahmachari & Das, 2012; Jiang et al., 2011). Other characteristics of MCRs are as follows: (*i*) simple procedures for the formation of final products in a one-pot process from at least three starting materials, (*ii*) green process of bond-forming, (*iii*) atom economy and convergent character, (*iv*) minimal waste generation, (*v*) facile construction of complex organic molecules, (*vi*) no need for complicated purification processes (Dömling et al., 2012; de Graaff et al., 2012; Wan & Liu, 2012; Ugi, 2001). MCRs using water as the reaction medium will be one of the methods, which will form a significant component of green chemistry (Chanda & Fokin, 2009; Kumaravel & Vasuki, 2009; Syamala, 2009; Candeias et al., 2010; Gu, 2012).

In addition, the heterocyclic framework containing the amino-benzochromene moiety represents a significant class of organic compounds, being the central components of numerous biologically valuable pharmacological compounds. Amino-benzochromene derivatives have a variety of properties, including anti-anaphylactic (Poupaert et al., 2005), antimicrobial (Bedair et al., 2001; Khafagy et al., 2002; Kathrotiya & Patel, 2012), antifungal (Abrunhosa et al., 2007), antibacterial (Kidwai et al., 2005), antitumour (El-Agrody et al., 2013a, 2013b, 2014), and anti-leishmanial (Narender et al., 2004). For example, LY290181 (see *IVc*, Table 1), is an inhibitor of diabetes-induced vascular dysfunction (Birch et al., 1996).

The synthesis of amino-benzochromenes via condensation reactions has been performed by using many homogeneous or heterogeneous catalysts, for example I_2/K_2CO_3 (Ren & Cai, 2008), cetyltrimethylammonium bromide (CTABr) (Jin et al., 2004), $NaHCO_3$ (Zhou et al., 2008), Na_2CO_3 (Naimi-Jamal et al., 2010), basic alumina (Maggi et al., 2004), nano-

*Corresponding author, e-mail: hkiyani@du.ac.ir

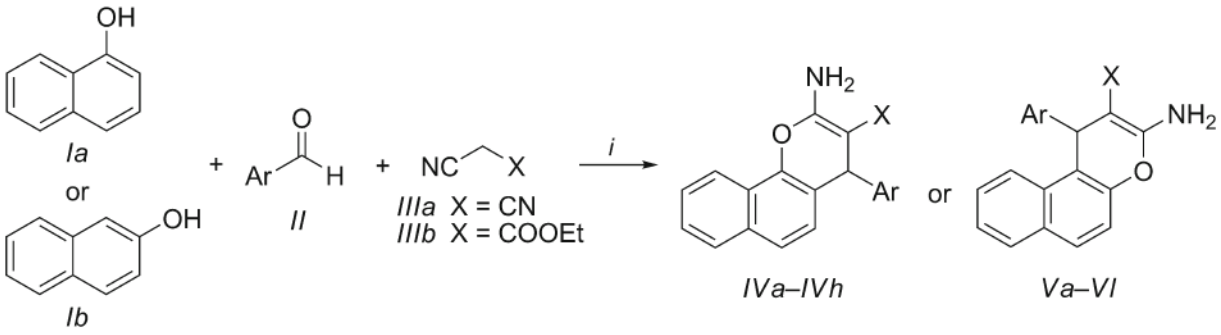


Fig. 1. Preparation of 2-amino-4-aryl-4*H*-benzo[*h*]chromenes (*IVa–IVh*) and 3-amino-1-aryl-1*H*-benzo[*f*]chromenes (*Va–VI*) via one-pot three-component reaction catalysed by PPI. Reaction conditions: *i*) PPI (15 mole %), H_2O, reflux.

sized MgO (Kumar et al., 2007), nano-structured diphosphate $Na_2CaP_2O_7$ (Solhy et al., 2010), triazine-functionalised ordered mesoporous organosilica (Mondal et al., 2012), potassium phosphate tribasic trihydrate (Zhou et al., 2012), piperidine under microwave (Mekheimer & Sadek, 2009a, 2009b), Mg/Al hydrotalcite (Surpur et al., 2009), DBU (Khurana et al., 2010), heteropolyacid (Heravi et al., 2007), Amberlyst A21 (Bihani et al., 2013), DABCO (Shinde et al., 2013), CeO_2/CaO nanocomposite oxide (Samantaray et al., 2012), poly(4-vinylpyridine) (Albadi et al., 2013), Triton B (Sabitha et al., 2011), tetrabutylammonium chloride (TBAC) (Mehrabi & Kamali, 2012), nano-eggshell powder (Mosaddegh, 2013), piperidine (El-Agrody et al., 2013a, 2013b, 2014), and potassium phthalimide-*N*-oxyl (POPINO) (Dekamin et al., 2013).

Although the above procedures have their own merits, most of them suffer from one or more drawbacks, including complex steps, prolonged reaction times, low yields, tedious preparation procedure, use of expensive catalysts and organic solvents. Accordingly, it is essential to develop more effective, fast, simple, and green procedures to prepare similar skeleton compounds. Water has a unique place in chemistry and is one of the best solvents, owing to its features such as being environmentally friendly, safe, nontoxic, non-flammable, clean, green, low-cost and readily available in organic transformations. Also, the use of water not only diminishes the risk entailed in the use of organic solvents but also improves the rate of many chemical reactions (Lindström, 2002; Pirrung, 2006; Li & Chan, 2007; Ogawa & Kobayashi, 2007; Candeias et al., 2010; Chitra et al., 2011; Xu & Qu, 2013).

Organocatalysts, on the other hand, typically are small organic molecules that are able to facilitate chemical transformations with a substoichiometric amount of an organic compound. Organocatalytic strategies have attracted attention for their application in organic synthesis due to their efficiency and selectivity. Compared with metallic catalysts, the preparation and handling of most organocatalysts is easier, cheaper, relatively stable, and environmentally friendly (Dalko & Moisan, 2004; Dalko, 2007; Zhong & Shi, 2010; Dondoni & Massi, 2008). Organocatalysts also possess other advantages such as low cost, ready availability, metal-free environment, relatively low toxicity, simple functionality, non-sensitivity to air and moisture, promotion of a variety of chemical transformations via various activation modes, mildness of the reaction conditions required, huge potential for the development of large-scale production (Verma et al., 2010; Khazaei et al., 2012; Cozzi & Hayashi, 2012).

In recent years, the challenge in organic synthesis has been the development of solid basic catalytic systems as well as organocatalysts utilising inexpensive, clean, environmentally benign, and commercially available catalysts (Tanabe & Hölderich, 1999; Debecker et al., 2009; Niknam & Jamali, 2012). Potassium phthalimide (PPI) is a mild, green, inexpensive, commercially available, efficient basic recyclable organocatalyst, and stable reagent. It has been utilised as a reagent in the synthesis of primary amines by the Gabriel method (Salzberg & Supniewski, 1941; Smith & March, 2001), phthalimide derivatives (Stewart et al., 2007; Motoshima et al., 2009; Pascale et al., 2010; Chan et al., 2009; Singh et al., 2011; Manley-King et al., 2011; Zwanenburg & Mwakaboko, 2011) and as organocatalyst for the preparation of cyanohydrin trimethylsilyl ethers (Dekamin & Karimi, 2009; Dekamin et al., 2009). We have recently reported that PPI is an efficient and green organocatalyst for the synthesis of 3,4-disubstituted isoxazol-5(4*H*)-ones (Kiyani & Ghorbani, 2014) and 4-aryl-7-(arylmethylene)-3,4,6,7-tetrahydro-1*H*-cyclopenta[*d*]pyrimidin-2(5*H*)-ones/thiones (Kiyani & Ghiasi, 2014). To the best of our knowledge, there has been no report on the use of PPI as a catalyst in the synthesis of chromene derivatives. The present study reports on the applicability of PPI as a readily available, efficient and solid basic organocatalyst for the synthesis of a wide variety of functionalised chromene derivatives using the one-pot three-component process (Fig. 1).

Table 1. Synthesis of 2-amino-4-aryl-4*H*-benzo[*h*]chromenes (*IVa–IVh*) and 3-amino-1-aryl-1*H*-benzo[*f*]chromenes (*Va–Vl*) via three-component reaction of 1-naphthol (*Ia*) or 2-naphthol (*Ib*), aromatic aldehyde (*II*), and nitrile compound (*IIIa* or *IIIb*)[a]

Entry	Aldehyde	Nitrile	Product	Time/min	Yield[b]/%	M.p./°C	
						Found	Reported
1	C_6H_5CHO	*IIIa*	*IVa*	35	91	207–208	210–211[c]
2	4-$O_2NC_6H_4CHO$	*IIIa*	*IVb*	30	90	240–242	242–243[c]
3	3-$O_2NC_6H_4CHO$	*IIIa*	*IVc*	30	89	210–212	210–212[d]
4	4-ClC_6H_4CHO	*IIIa*	*IVd*	30	96	231–233	232–234[e]
5	4-MeC_6H_4CHO	*IIIa*	*IVe*	40	93	198–200	202–204[f]
6	4-OHC_6H_4CHO	*IIIa*	*IVf*	36	96	246–247	247–249[g]
7	4-$MeOC_6H_4CHO$	*IIIa*	*IVg*	35	93	187–189	188–189[c]
8	3-$O_2NC_6H_4CHO$	*IIIb*	*IVh*	60	90	199–201	198–200[h]
9	C_6H_5CHO	*IIIa*	*Va*	50	93	272–274	273–275[d]
10	4-$O_2NC_6H_4CHO$	*IIIa*	*Vb*	40	90	181–183	182–183[c]
11	3-$O_2NC_6H_4CHO$	*IIIa*	*Vc*	40	89	189–191	189–190[f]
12	4-ClC_6H_4CHO	*IIIa*	*Vd*	40	94	200–201	203–205[d]
13	4-BrC_6H_4CHO	*IIIa*	*Ve*	42	92	242–243	242–244[i]
14	4-MeC_6H_4CHO	*IIIa*	*Vf*	40	89	256–257	270–272[i]
15	4-OHC_6H_4CHO	*IIIa*	*Vg*	60	98	245–247	246–248[i]
16	4-$MeOC_6H_4CHO$	*IIIa*	*Vh*	60	92	185–186	185–187[j]
17	4-$(Me)_2NC_6H_4CHO$	*IIIa*	*Vi*	60	95	243–245	244–245[f]
18	C_6H_5CHO	*IIIb*	*Vj*	65	89	167–168	166–168[h]
19	3-$O_2NC_6H_4CHO$	*IIIb*	*Vk*	55	90	189–191	188–190[h]
20	3-ClC_6H_4CHO	*IIIb*	*Vl*	55	90	202–205	202–204[h]

a) Reaction conditions: 1 mmol of each reactant, water (5 mL), PPI (15 mole %), reflux; *b*) yield of pure isolated product; *c*) Zhou et al. (2008); *d*) Naimi-Jamal et al. (2010); *e*) Mekheimer and Sadek (2009a); *f*) Mehrabi and Kamali (2012); *g*) Ren and Cai (2008); *h*) Sabitha et al. (2011); *i*) Albadi et al. (2013); *j*) Dekamin et al. (2013).

Experimental

All chemicals, unless otherwise specified, were purchased from commercial sources and were used without further purification, with the exception of benzaldehyde, which was distilled prior to use. The products were characterised by comparing their physical data with those of known compounds or by their spectral data. Melting points were measured on a Büchi 510 melting point apparatus and are uncorrected. ^{1}H and ^{13}C NMR spectra were recorded at ambient temperature on a Bruker AVANCE DRX-500 using $CDCl_3$ or DMSO-d_6 as solvent. FTIR spectra were recorded on a Perkin–Elmer RXI spectrometer. The progress of reactions was monitored by thin layer chromatography (TLC) on Merck pre-coated silica gel 60 F_{254} aluminium sheets, visualised by UV light.

General procedure for synthesis of chromene derivatives IV and V

A mixture of naphthol (*I*) (1 mmol), aldehyde (*II*) (1 mmol), malononitrile or ethyl cyanoacetate (*III*) (1 mmol), and potassium phthalimide (15 mole %) in distilled water (5 mL) was heated under reflux for the time indicated (Table 1). The progress of the reaction was monitored by TLC. On completion of the reaction, the reaction mixture was cooled to ambient temperature and the precipitated solid was filtered, washed with cold distilled water (4 mL) and air-dried to obtain the pure products. If necessary, the solid products were recrystallised from hot ethanol. After evaporation of the water from the filtrate, the catalyst was recovered and used in the subsequent reaction. Selected spectral data for the representative compounds are summarised in Table 2.

Results and discussion

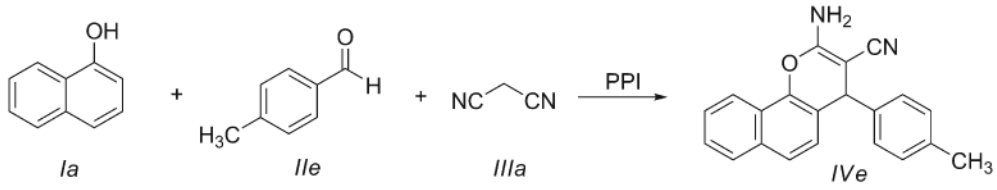
First, the reaction conditions were optimised using equimolar quantities (1 mmol) of 1-naphthol (*Ia*), 4-methylbenzaldehyde (*IIf*), and malononitrile (*IIIa*) as the model. A range of reaction conditions was explored, including solvents, amounts of catalyst and temperatures. The results are summarised in Table 3.

Table 1 shows that, in the absence of the catalyst, the reaction did not proceed at ambient temperature. Only a trace amount of the 2-amino-4-(*p*-tolyl)-4*H*-benzo[*h*]chromene-3-carbonitrile (*IVe*) was obtained (entry 1). It was also revealed that the reaction was rather slow and afforded a poor yield (20 %) in the absence of the catalyst when the reaction was carried out in refluxing water for 50 min (entry 2), indicating that the catalyst is necessary for this transformation. Further, increasing the reaction time was not significant for changing the product yield. The yield increased slightly when PPI was added to the reaction mixture at ambient temperature (entry 3). The effect of catalyst-loading on completion of the reaction at ambient temperature was also studied. When

Table 2. Spectral data of representative compounds

Compound	Spectral data
IVd	IR, $\tilde{\nu}$/cm^{-1}: 879, 1100, 1377, 1415, 1574, 1600, 1668, 2202, 3334, 3452 ^{1}H NMR (500 MHz, DMSO-d_6), δ: 4.55 (s, 1H, CH), 6.67 (s, 2H, NH_2), 7.01 (d, J = 8.7 Hz, 1H, ArH), 7.20–7.33 (m, 4H, ArH), 7.42–7.47 (m, 3H, ArH), 7.79 (d, J = 7.1 Hz, 1H, ArH), 8.16 (d, J = 8.2 Hz, 1H, ArH) ^{13}C NMR (125 MHz, DMSO-d_6), δ: 42.5, 55.7, 119.8, 120.5, 122.1, 122.5, 123.6, 125.5, 125.8, 126.5, 127.6, 128.8, 130.1, 132.7, 133.9, 142.3, 146.9, 160.7
IVe	IR, $\tilde{\nu}$/cm^{-1}: 1127, 1383, 1436, 1592, 1654, 2210, 3268, 3394 ^{1}H NMR (300 MHz, DMSO-d_6), δ: 2.26 (s, 3H, CH_3), 4.55 (s, 1H, CH), 6.90 (s, 2H, NH_2), 7.07 (d, J = 8.0 Hz, 1H, ArH), 7.15–7.26 (m, 4H, ArH), 7.34–7.41 (m, 3H, ArH), 7.66 (d, J = 8.0 Hz, 1H, ArH), 8.04 (d, J = 8.0 Hz, 1H, ArH) ^{13}C NMR (75 MHz, DMSO-d_6), δ: 31.4, 43.7, 55.9, 119.7, 120.3, 122.0, 122.7, 123.8, 124.0, 125.6, 126.2, 127.4, 128.7, 130.5, 132.7, 133.7, 141.6, 145.2, 160.6
Ve	IR, $\tilde{\nu}$/cm^{-1}: 736, 819, 1012, 1080, 1234, 1410, 1485, 1591, 1641, 2193, 3051, 3194, 3323, 3416 ^{1}H NMR (400 MHz, $CDCl_3$), δ: 4.64 (s, 2H, NH_2), 5.24 (s, 1H, CH), 7.07 (d, J = 8.4 Hz, 2H, ArH), 7.28 (d, J = 6.3 Hz, 2H, ArH), 7.39–7.44 (m, 4H, ArH), 7.61–7.64 (m, 1H, ArH), 7.84 (d, J = 8.5 Hz, 2H, ArH) ^{13}C NMR (100 MHz, DMSO-d_6), δ: 37.9, 57.7, 115.5, 117.3, 120.2, 120.9, 124.0, 125.5, 127.7, 128.9, 129.7, 130.2, 130.5, 131.3, 132.1, 145.6, 147.3, 160.2
Vf	IR, $\tilde{\nu}$/cm^{-1}: 810, 1000–1300, 1400, 1580, 1640, 2190, 3050, 3310, 3410 ^{1}H NMR (300 MHz, $CDCl_3$), δ: 2.31 (s, 3H, CH_3), 4.59 (s, 1H, NH_2), 5.23 (s, 2H, CH), 7.08–7.25 (m, 4H, ArH), 7.33 (d, J = 8 Hz, 1H, ArH), 7.39–7.42 (m, 2H, ArH), 7.69–7.73 (m, 1H, ArH), 7.83 (d, J = 6.2 Hz, 2H, ArH) ^{13}C NMR (75 MHz, DMSO-d_6), δ: 21.0, 38.2, 58.5, 116.3, 117.3, 121.1, 124.2, 125.4, 127.4, 127.5, 128.9, 129.7, 129.9, 130.7, 131.3, 136.2, 143.3, 147.3, 160.1

Table 3. Optimisation of reaction conditions for one-pot three-component synthesis of 2-amino-4-(*p*-tolyl)-4*H*-benzo[*h*]chromene-3-carbonitrile (*IVe*)[a]



Ia *IIe* *IIIa* *IVe*

Entry	Solvent	Amount of catalyst/mole %	Time/min	Temperature/°C	Yield[b]/%
1	H_2O	–	50	ambient	trace
2	H_2O	–	50	reflux	20
3	H_2O	5	50	ambient	22
4	H_2O	10	50	ambient	30
5	H_2O	15	50	ambient	32
6	H_2O	20	50	ambient	35
7	H_2O	5	45	reflux	42
8	H_2O	10	40	reflux	82
9	H_2O	15	40	reflux	93
10	H_2O	20	40	reflux	94
11	EtOH	15	40	reflux	50
12	MeCN	15	40	reflux	trace
13	1,4-dioxane	15	40	reflux	trace
14	CH_2Cl_2	15	40	reflux	trace
15	EtOAc	15	40	reflux	trace
16	$CHCl_3$	15	40	reflux	trace
17	H_2O/EtOH[c]	15	40	reflux	65

a) Reaction conditions: 1-naphthol (1 mmol), 4-methylbenzaldehyde (1 mmol), malononitrile (1 mmol), solvent (5 mL); *b*) isolated yields; *c*) φ_r = 1 : 1.

the amount of catalyst was increased to 20 mole %, a slight improvement was achieved in the yield (entries 4–6). Alternatively, the reaction was carried out with varying amounts of catalyst in water under reflux conditions. It was found that, with increasing the amount of the catalyst (5–15 mole %) under reflux conditions, the yield of *IVe* was improved (entries 7–9). A higher amount of the catalyst (20 mole %) neither increased

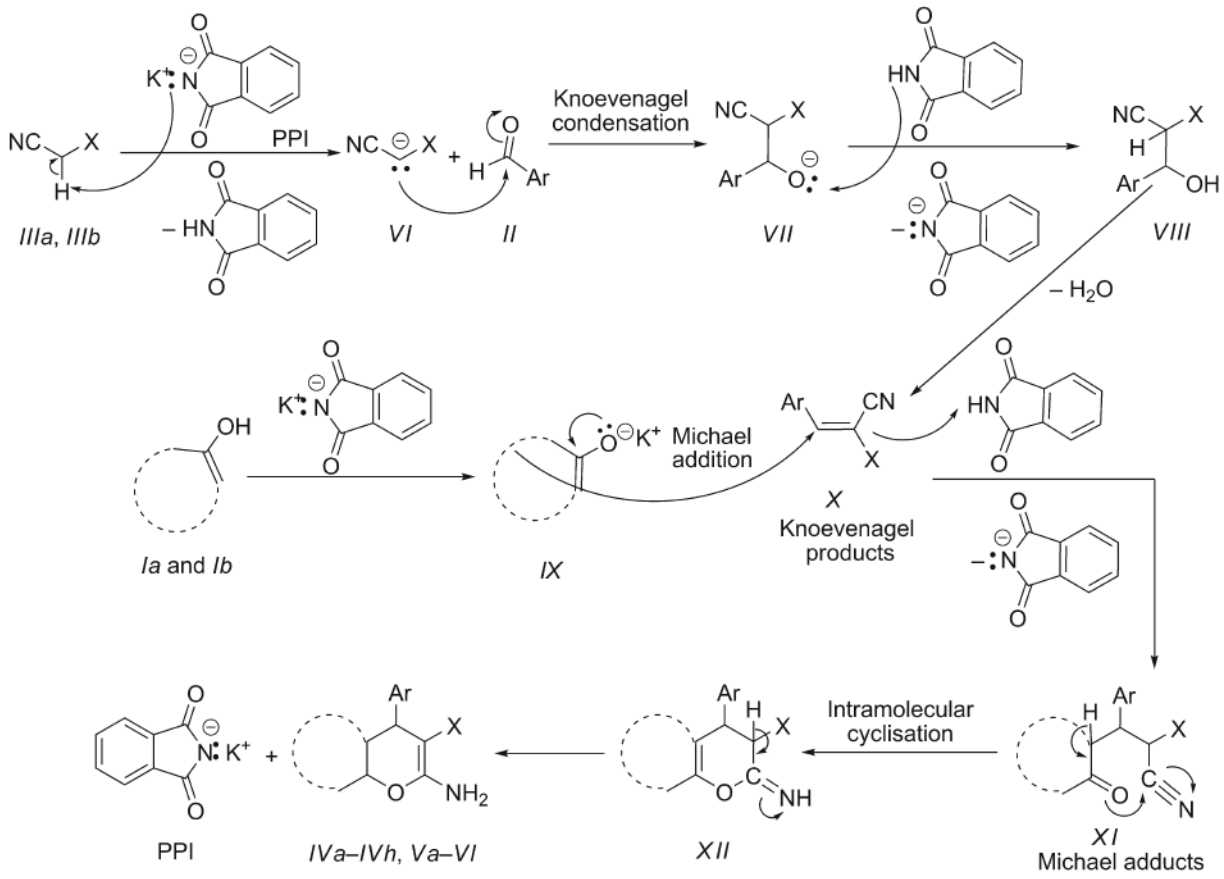


Fig. 2. Proposed mechanism for condensation reaction of aromatic aldehyde, cyano-compound and 1-naphthol or 2-naphthol in the presence of PPI.

the yield nor shortened the conversion time (entry 10). On the other hand, the use of EtOH and a mixture of solvents (EtOH/H_2O, $\varphi_r = 1:1$) afforded a lower yield of *IVe* under similar conditions (entries 11 and 17). Also, carrying out the reaction in other organic solvents such as MeCN, 1,4-dioxane, CH_2Cl_2, EtOAc, or $CHCl_3$ led to the formation of a trace amount of product *IVe* (entries 12–16). It is probable that the high yields obtained in water relative to the other organic solvents investigated are due to the high solubility of PPI in water. Hence, the 15 mole % catalyst-loading and refluxing in water was deemed to be the optimal conditions for this reaction.

Under the optimised reaction conditions (water, 15 mole % of PPI, reflux), a series of substituted aromatic aldehydes were studied in order to establish the scope of this simple reaction procedure. The reaction time and yield of the products are presented in Table 1.

Under the above optimised conditions, chromene derivatives *IVa–IVh* were obtained with good to high yields (entries 1–8). When 1-naphthol was replaced with 2-naphthol under similar reaction conditions, chromene derivatives *Va–IVl* were isolated with high yields (entries 9–20). The reaction also proceeded smoothly with various aryl aldehydes substituted affording the corresponding products with good to excellent yields. Hence, the electronic nature of the substituent did not influence reaction yields. However, aromatic aldehydes with electron-withdrawing groups reacted faster than those with electron-releasing groups. Using less reactive ethyl cyanoacetate (entries 8, 18–20) instead of malononitrile afforded the corresponding products with lower yields and the reaction required a longer reaction time, probably due to the greater involvement of the cyanide group in stabilising the reaction intermediates than the ester group (Dekamin et al., 2013; Zhu et al., 2007; Yu et al., 2011).

The experimental procedure with this catalyst is simple and the catalyst can be easily regenerated by evaporation of the filtrate. The solid products formed after cooling the reaction mixture to ambient temperature can be easily separated by filtering. The desired products are sufficiently pure; however, if further purification is required, they can be recrystallised from ethanol.

The probable mechanism for the formation of final products *IV* and *V* is outlined in Fig. 2. PPI, as a weak base, effectively catalyses the in-situ formation of α-cyanocinnamonitrile intermediate (Knoevenagel product) *X* via the Knoevenagel condensation reaction from aromatic aldehyde *II* and intermediate

Table 4. Reusability of PPI in synthesis of 2-amino-4-(*p*-tolyl)-4*H*-benzo[*h*]chromene-3-carbonitrile (*IVe*)

No. of cycles	Fresh	Run 1	Run 2	Run 3	Run 4
Time/min	40	40	40	45	45
Yield[a]/%	93	92	90	88	85

a) Isolated yield.

nitrile anion *VI*. Next, the Michael-type addition of the naphtholic compound to the Knoevenagel product *X* results in the in-situ formation of the Michael addition product *XI*, which subsequently undergoes intramolecular nucleophilic cyclisation to afford the desired compound.

The reusability of the PPI in the one-pot three-component condensation reaction was investigated using 1-naphthol, 4-methylbenzaldehyde and malononitrile as the model reaction. A comparison of the melting point and FTIR spectrum of the solid material recovered (washed with small amounts of distilled water) with those of the authentic sample indicated that the recovered material is PPI. The recovered catalyst was dried at ambient temperature for 8 h and heated in the oven at 90 °C for 1 h. This catalyst was employed for a further five additional successive reaction cycles. In all cases, consistent catalytic activity of PPI was observed (Table 4). The decreasing yield is probably related to a slight reduction in the catalytic activity of the catalyst or could be attributed to the loss of catalyst recovery in the course of the reaction.

A comparison of the results obtained using the PPI with the results for selected previously known catalysts reported in the literature (Table 5) showed that PPI is superior in terms of reaction time and yield, as well as being an effective catalyst for the synthesis of amino-benzochromenes.

Conclusions

In summary, an efficient PPI-catalysed one-pot three-component methodology for the synthesis of a variety of 2-amino-4*H*-benzo[*h*]chromene and 3-amino-1*H*-benzo[*f*]chromene derivatives affording high to excellent yields was developed. This approach is very simple from the experimental perspective and would provide easy access to large families of amino-benzochromenes. Other attractive features of this method include the clean reaction profiles, no requirement for hazardous organic solvents, minimal amounts of waste entailed in each organic transformation, efficiency, reasonable reaction times, easy purification, aqueous conditions, green, mild and low cost organocatalyst.

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Table 5. Comparison of results obtained using PPI with those obtained using different catalytic system for condensation of 4-methoxybenzaldehyde with malononitrile and 1-naphthol

Entry	Catalysts/conditions	Time/min	Yield[a]/%	Reference
1	Basic alumina/H_2O, 100 °C	180	83	Maggi et al. (2004)
2	Mg/Al hydrotalcite/140 °C, microwave, solvent-free	16	76	Surpur et al. (2009)
3	$I_2/K_2CO_3/H_2O$, 100 °C	60	89	Ren and Cai (2008)
4	$H_{14}[NaP_5W_{30}O_{110}]/H_2O$, 100 °C	195	91	Heravi et al. (2007)
5	$NaHCO_3$/grinding	90	79	Zhou et al. (2008)
6	TBAC/H_2O, 100 °C	150	96	Mehrabi and Kamali (2012)
7	TBAC/100 °C, solvent-free	35	90	Mehrabi and Kamali (2012)
8	CeO_2/CaO nanocomposite oxide/H_2O, 80 °C	75	81	Samantaray et al. (2012)
9	Amberlyst A21/EtOH, r.t.	360	71	Bihani et al. (2013)
10	Nano-structured $Na_2CaP_2O_7/H_2O$, reflux	300	76	Solhy et al. (2010)
11	CTABr/H_2O, ultrasound	150	88	Jin et al. (2004)
12	Nano-MgO/(PEG/water)[b]	90	82	Kumar et al. (2007)
13	Na_2CO_3/125 °C, solvent-free	40	91	Naimi-Jamal et al. (2010)
14	PPI/H_2O, reflux	35	93	this work

a) Isolated yield; *b*) PEG/water, $\varphi_r = 1:1$.

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