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ORIGINAL PAPER

$Co(NO_3)_2{\cdot}6H_2O$ as a powerful and reusable catalyst for the synthesis of phenylbenzo[*g*]chromenes

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Abstract An efficient and easy protocol for the one-pot three-component synthesis of phenylbenzo[*g*]chromenes was developed. The synthesis was achieved by the reaction of aromatic aldehydes, Meldrum's acid, and 2-hydroxynaphthalene-1,4-dione in the presence of catalytic amount of $Co(NO_3)_2{\cdot}6H_2O$ at room temperature. The important advantages of this procedure are short reaction times, high yields, easy work-up, reusable catalyst, and no need to column chromatography.

Keywords Phenylbenzo[*g*]chromenes · Aromatic aldehydes · Meldrum's acid · 2-hydroxynaphthalene-1 · 4-dione

Introduction

Heterocycles, an important class of organic compounds, consist of more than 70% of promising bioactive and drug molecules presently available in literature. Because of their wide broad biological and natural affluence, Oxygen heterocycles have a special interest and pharmaceutical significance to chemists [1]. *O*-heterocycles, "*chromene*" heterocyclic scaffolds represent a "distinguished" structural motif well-distributed in natural products with a broad spectrum of biological activities including anti-Alzheimer"s [2], anti-proliferative [3], anti-microbial [4], anti-cancer [5], anti-tumor [6], TNF-α inhibitor [7] anti-viral [8, 9], anti-Parkinson disease, anti-inflammatory [10], anticonvulsant [11], antimalarial [12, 13], anti-helminthic [14, 15], sex-hormonal activity [16], anti-Huntington"s disease [17], estrogenic [18] and others [19] (Fig. 1). The possibility of wide range of biological properties make chromens as an interesting synthetic target. Therefore, developing straightforward and flexible synthetic methods toward functionalized chromens has attracted attention from organic researchers [20–23]. Some methods such as microwave irradiation [24], use of L-proline and nickel nanoparticles as catalyst [25, 26] have been reported to facilitate the synthesis of phenylbenzo[*g*]chromenes. In continuation of our research on chromene synthesis and considering the important biological and pharmaceutical properties of these compounds [27–29], we investigate to report an efficient approach for the synthesis of phenylbenzo[*g*]chromenes via the one-pot three-component condensation between aromatic aldehydes, Meldrum's acid, and 2-hydroxynaphthalene-1,4-dione in the presence of catalytic amount of $Co(NO_3)_2{\cdot}6H_2O$ at ambient condition (Scheme 1).

Experimental

General

Melting points and IR spectra were measured on an Electrothermal 9100 apparatus and a JASCO FT/IR-460 plus spectrometer. The ^1H and ^{13}C NMR spectra were recorded on a Bruker DRX-300 Advance instrument with $CDCl_3$ and DMSO as solvent at 300 and 75 MHz, respectively. The

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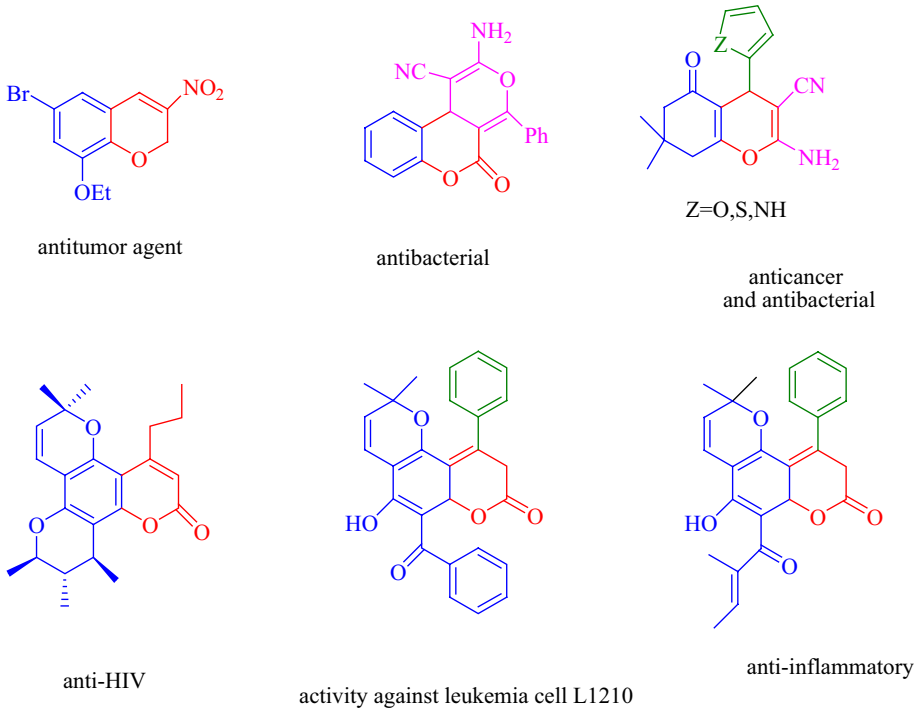


Fig. 1 Some bioactive chromenes heterocycles

mass spectra were recorded on an Agilent Technology (HP) mass spectrometer, operating at an ionization potential of 70 eV. Aromatic aldehydes, Meldrum's acid, and 2-hydroxynaphtalene-1, 4-dion were obtained from Merck (Darmstadt, Germany), Aldrich and Fluka (Buchs, Switzerland), and used without further purification.

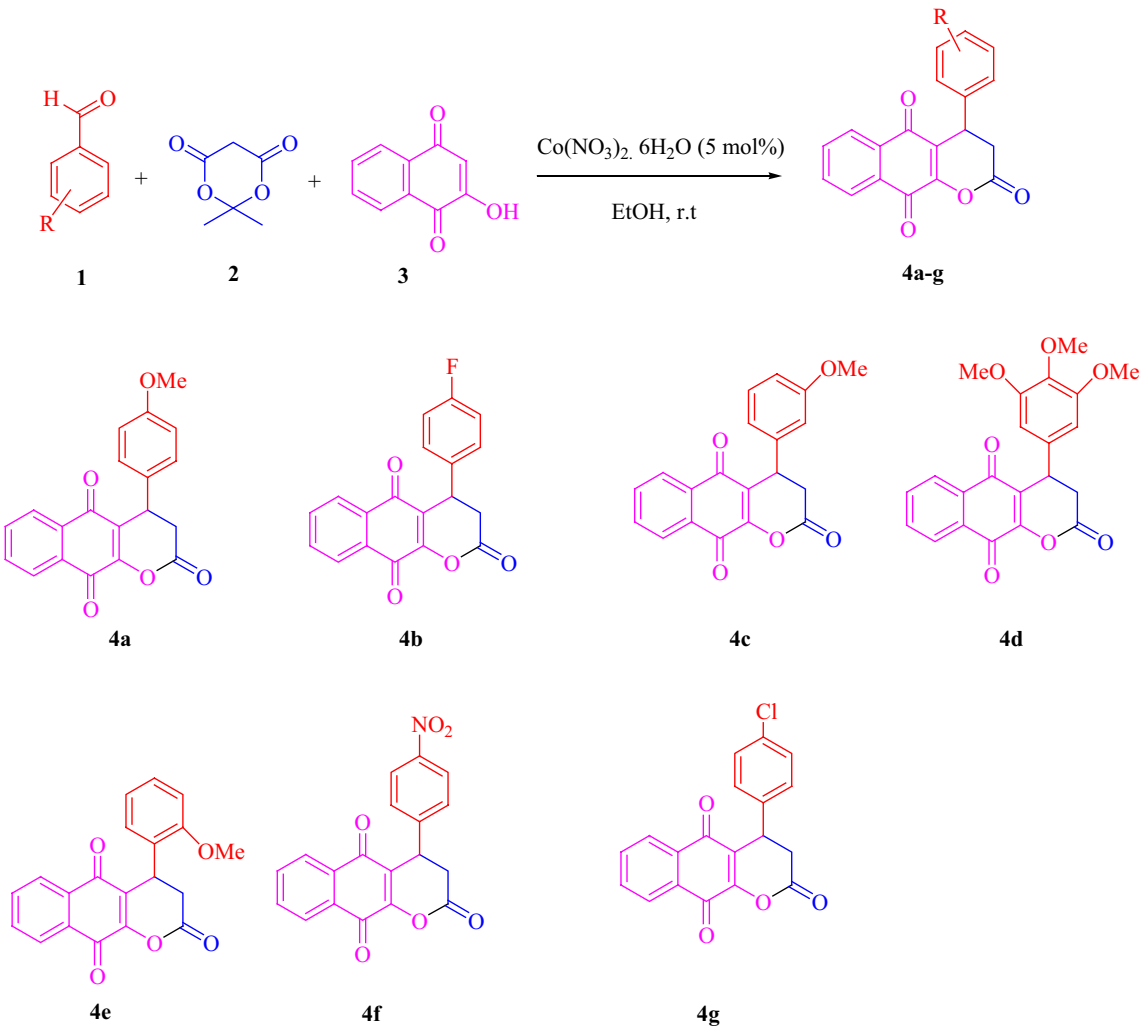
*General procedure for the synthesis of (**4a–g**)*

2-Hydroxynaphtalen-1, 4-dion (1.0 mmol) was added to the stirred solution of aromatic aldehydes (1.0 mmol) and Meldrum's acid (1.0 mmol) in EtOH (3 mL). Then, $Co(NO_3)_2{\cdot}6H_2O$ (5 mol%) was added to the above mixture. The reaction mixture was stirred for the time indicated in Table 2. The progress of the reaction was monitored by thin-layer chromatography (TLC). After the completion of the reaction, the thick precipitation was filtered off and washed with ethanol (3×2 cm^3) to remove the catalyst. The solid was purified by recrystallization from ethanol to give product **4a–g**.

3,4-Dihydro-4-(4-methoxyphenyl)benzo[*g*]chromene-2,5,10-trione (4a) IR (KBr, cm^{-1}): 2966, 2842, 1782, and 1742. ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 3.10 (d, $J = 4.2$ Hz, 2H), 3.78 (s, 3H, OCH_3), 4.63 (s, 1H), 6.86 (d, $J = 8.4$ Hz, 2H, Ar), 7.20 (d, $J = 8.1$ Hz, 2H, Ar), 7.79–7.80 (m, 2H, Ar), 8.19–8.22 (m, 2H, Ar). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 34.4, 35.4, 55.3, 114.7, 125.9, 126.7, 126.8, 127.9, 130.7, 130.8, 131.3, 134.1, 134.5, 151.1, 159.3, 164.0 (C=O), 177.3 (C=O), 182.7 (C=O). MS (EI, 70 eV) m/z (%): 334 (M^+, 59), 306 (100), 277 (94), 249 (15), 228 (24), 189 (16), 165 (47), 134 (34), 104 (73), 76 (80), 43 (23).

3,4-Dihydro-4-(4-fluoro phenyl)benzo[*g*]chromene-2,5,10-trione (4b) IR (KBr, cm^{-1}): 2921, 2843, 1782, and 1686. ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 3.07–3.13 (m, 2H), 4.68 (dd, $J = 6.0$, 2.7 Hz, 1H), 7.04 (t, $J = 8.4$ Hz, 2H,Ar), 7.28–7.29 (m, 2H, Ar), 7.79–7.783 (m, 2H, Ar), 8.09–8.23 (m, 2H, Ar). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 34.5, 35.3, 116.4 (d, $J = 21.75$ Hz), 125.4, 126.7, 126.9, 128.5 (d, $J = 7.8$ Hz), 130.8, 131.2, 134.2, 134.5, 134.5, 134.6, 151.3, 160.7, 163.7 (d, $J = 228.0$ Hz), 164.0 (C=O), 177.2 (C=O), 182.6 (C=O). MS (EI, 70 eV) m/z (%): 322 (M^+, 81), 294 (4), 272 (3), 251 (70), 224 (16), 196 (27), 170 (13), 133 (30), 104 (96), 76 (100), 50 (30).

3, 4-Dihydro-4-(2-methoxyphenyl)benzo[*g*]chromene-2,5,10-trione (4c) IR (KBr, cm^{-1}): 2998, 2823, 1784, and 1680. ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 3.12 (d, $J = 4.8$ Hz, 2H), 3.79 (s, 3H, OCH_3), 4.64 (t, $J = 4.2$ Hz, 1H), 6.80–6.84 (m, 3H, Ar), 7.22–7.29 (m, 1H, Ar), 7.55–7.81 (m, 2H, Ar), 8.07–8.20 (m, 2H, Ar). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 35.1, 35.2, 55.2, 113.0, 113.2, 118.7, 125.5, 126.7, 126.8, 130.5, 130.8, 131.3, 134.1, 134.5, 140.2, 151.4, 160.2, 163. 9 (C=O), 177.3 (C=O), 182.6 (C=O). MS (EI, 70 eV) m/z (%): 334 (M^+, 30), 306 (11), 277 (16), 251 (25), 228 (3), 207 (11), 178 (12), 149 (36), 127 (10), 104 (59), 76 (18), 43 (100).



Scheme 1 Synthesis of phenylbenzo[*g*]chromene derivatives

3, 4-Dihydro-4-(3, 4, 5-trimethoxyphenyl) benzo[*g*] chromene-2, 5, 10-trione (4d) IR (KBr, cm^{-1}): 2941, 2844, 1708, and 1669. ^{1}H NMR (300 MHz, CDC_{13}) δ (ppm): 3.11–3.13 (m, 2H), 3.81 (s, 3H, OCH_3), 3.84 (s, 6H, $2OCH_3$), 4.62 (dd, J = 6.0, 3 Hz, 1H), 6.49 (s, 2H, Ar), 8.11–8.14 (m, 2H, Ar), 8.20–8.23 (m, 2H, Ar). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 35.1, 35.3, 56.2 ($2OCH_3$), 60.8 (OCH_3), 103.9, 125.6, 126.7, 126.9, 130.8, 131.3, 134.2, 134.4, 134.6, 137.8, 151.1, 153.8, 164.2 (C=O), 177.3 (C=O), 182.7 (C=O). MS (EI, 70 eV) m/z (%): 349 (M^+, 91), 366 (25), 336 (4), 307 (21), 277 (10), 251 (19), 223 (15), 176 (20), 146 (7), 104 (86), 76 (100), 43 (80).

3,4-Dihydro-4-(2-methoxyphenyl)benzo[*g*] chromene-2,5,10-trione (4e) IR (KBr, cm^{-1}): 2931, 2883, 1783, and 1680. ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 2.96–3.13 (m, 2H), 3.77 (s, 3H, OCH_3), 4.67 (d, J = 8.4 Hz, 1H), 6.90 (d, J = 8.1 Hz, 1H, Ar), 6.95 (t, J = 7.5 Hz, 1H, Ar), 7.35–7.31 (m, 1H, Ar), 7.38 (d, J = 7.5 Hz, 1H, Ar), 7.72–7.76 (m, 2H, Ar), 8.02–8.20 (m, 2H, Ar). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 33.5, 35.3, 54.3, 110.8, 120.9, 122.8, 126.5, 126.7, 127.0, 129.4, 130.5, 130.8, 131.5, 133.8, 134.3, 151.1, 157.2, 163.7 (C=O), 177.7 (C=O), 183.1 (C=O). MS (EI, 70 eV) m/z (%): 334 (M^+, 93), 306 (66), 278 (15), 247 (95), 218 (9), 189 (26), 165 (34), 134 (50), 104 (78), 76 (100), 43 (32).

3,4-Dihydro-4-(4-nitrophenyl)benzo[*g*] chromene-2,5,10-trione (4f) IR (KBr, cm^{-1}): 2937, 2862, 1783, and 1680. ^{1}H NMR (300 MHz, $CDCl_3$, DMSO) δ (ppm): 2.96 (d, J = 16.2 Hz, 1H), 3.41 (d, 1H), 4.74 (d, J = 8.4 Hz, 1H), 7.50–8.14 (m, 8H, Ar). ^{13}C NMR (75 MHz, $CDCl_3$, DMSO) δ (ppm): 34.9, 35.2, 123.3, 124.2, 124.4, 125.9, 126.5, 126.5, 128.5, 129.1, 131.0, 131.3, 133.1, 134.4, 134.7, 152.3, 164.4 (C=O), 177.2 (C=O), 182.5 (C=O). MS (EI, 70 eV) m/z (%): 349 (M^+, 30), 3 246 (21),

Scheme 2 Model reaction for optimization amount of catalyst for the synthesis of phenylbenzo[*g*]chromenes

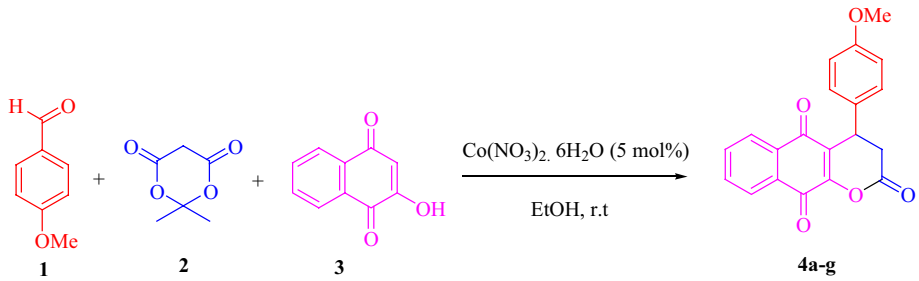


Table 1 Optimization conditions for the synthesis of phenylbenzo[g] chromenes

Entry	Catalyst	Solvent	Time	Isolated yield (%)
1	–	EtOH	48	–
2	Fe $(NO_3)_3{\cdot}9H_2O$	EtOH	48	Knovenogel product
3	*p*-toluene sulfonic acid	EtOH	12 h	40
4	Lactic acid (5 mol%)	EtOH	24 h	40
5	$ZrCl_4$	EtOH	18 h	50
6	$HClO_4$–SiO_2	EtOH	9 h	60
7	$KHSO_4$	EtOH	7 h	50
8	$Co(NO_3)_2{\cdot}6H_2O$ (3 mol%)	EtOH	50 min	30
9	$Co(NO_3)_2{\cdot}6H_2O$ (5 mol%)	EtOH	10 min	90
10	$Co(NO_3)_2{\cdot}6H_2O$ (10 mol%)	EtOH	10 min	90

221 (16), 293 (31), 218 (7), 189 (34), 149 (38), 127 (10), 104 (73), 76 (100), 43 (66).

3,4-Dihydro-4-(4-chlorophenyl)benzo[g] chromene-2,5,10-trione (4g) IR (KBr, cm^{-1}): 3024, 2959, 1741, and 1680. ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 2098–3.06 (m, 2H), 4.57 (dd, J = 6.0, 2.7 Hz, 1H), 7.12 (d, J = 8.4 Hz, 2H, Ar), 7.23 (d, J = 8.4 Hz, 2H, Ar), 7.68–7.72 (m, 2H, Ar), 7.99–8.13 (m, 2H, Ar). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 34.6, 35.1, 125.1, 126.7, 126.1, 126.7, 126.9, 127.1, 128.2, 128.8, 129.6, 130.8, 131.2, 134.2, 134.2, 134.6, 151.4, 163.9 (C=O), 177.1 (C=O), 182.5 (C=O). MS (EI, 70 eV) m/z (%): 340 (M^{+2}, 32), 338 (M^+, 82), 310 (40), 282 (58), 254 (11), 233 (40), 205 (12), 176 (38), 138 (60), 104 (82), 76 (100), 43 (41).

Result and discussion

The reaction conditions were optimized using 4-methoxybenzaldehyde, Meldrum's acid, and 2-hydroxynaphthalene-1,4-dion in equimolar amounts as a model system (Scheme 2). Initially, the control experiment confirmed that the reaction did not proceed without the catalyst (Table 1, Entries 1). The model reaction was then performed in the presence of different catalysts such as $Fe(NO_3)_3{\cdot}9H_2O$, *p*-toluene sulfonic acid and lactic acid at room temperature. As it can be seen in Table 1, the best reaction condition was obtained at room temperature in the presence of 5 mol% of $Co(NO_3)_2{\cdot}6H_2O$ (Table 1, Entry 7). The increasing amount of catalyst did not have any effect on the yield and time of reaction. With respect to green chemistry concept, we did not try other organic solvents for this synthesis.

Using these optimized experimental conditions, a wide verity of products **4a–g** were synthesized via the reaction of aromatic aldehydes, Meldrum's acid and 2-hydroxynaphthalene-1, 4-dion (Table 2).

Table 2 Interestingly, different aryl aldehydes including electron withdrawing or releasing substituents (ortho-, meta-, and para-substituted) participated well in this reaction and gave the phenylbenzo[*g*]chromene derivatives in good-to-excellent yield. The structures of the products **4a–g** were detected from IR, ^{1}H and ^{13}C NMR, and mass spectra. The mass spectrum of these compounds displayed molecular ion peaks with appropriate *m/z* values. Any initial fragmentation

Table 2 Synthesis of phenylbenzo[*g*]chromene derivatives

Entry	Aldehyde	Product	Time (min)	Yield (%)	M.p °C	Lit. M.p °C
1	4-OMe–C_6H_4	**4a**	10	90	200–202	191–192 [24]
2	4-F–C_6H_4	**4b**	15	95	210–212	210–212 [25]
3	3-OMe–C_6H_4	**4c**	15	85	201–203	191–192 [24]
4	3,4,5-(OMe)2–C_6H_2	**4d**	20	90	202–204	203–206 [25]
5	2-OMe–C_6H_4	**4e**	20	85	204–205	–
6	4-NO_2–C_6H_4	**4f**	10	90	219–221	219–221 [25]
7	4-Cl–C_6H_4	**4g**	10	85	222–224	224–227 [25]

involves the loss of the C=O moieties. The assignments of IR, ^{1}H, ^{13}C NMR are given in the experimental section.

According to the literature [24, 25], a mechanistic explanation for the formation of phenylbenzo[*g*]chromenes is shown in Scheme 3. First, formation of arylidene Meldrum's acid **A** by the Knoevenagel condensation between an aldehyde **1** and Meldrum's acid. Then, in addition to arylidene Meldrum's acid, 2-hydroxynaphtalene-1,4-dione undergoes a Michael-type **A** to give intermediate **B** which undergoes cyclization with concomitant loss of acetone and carbon dioxide to afford the desired product.

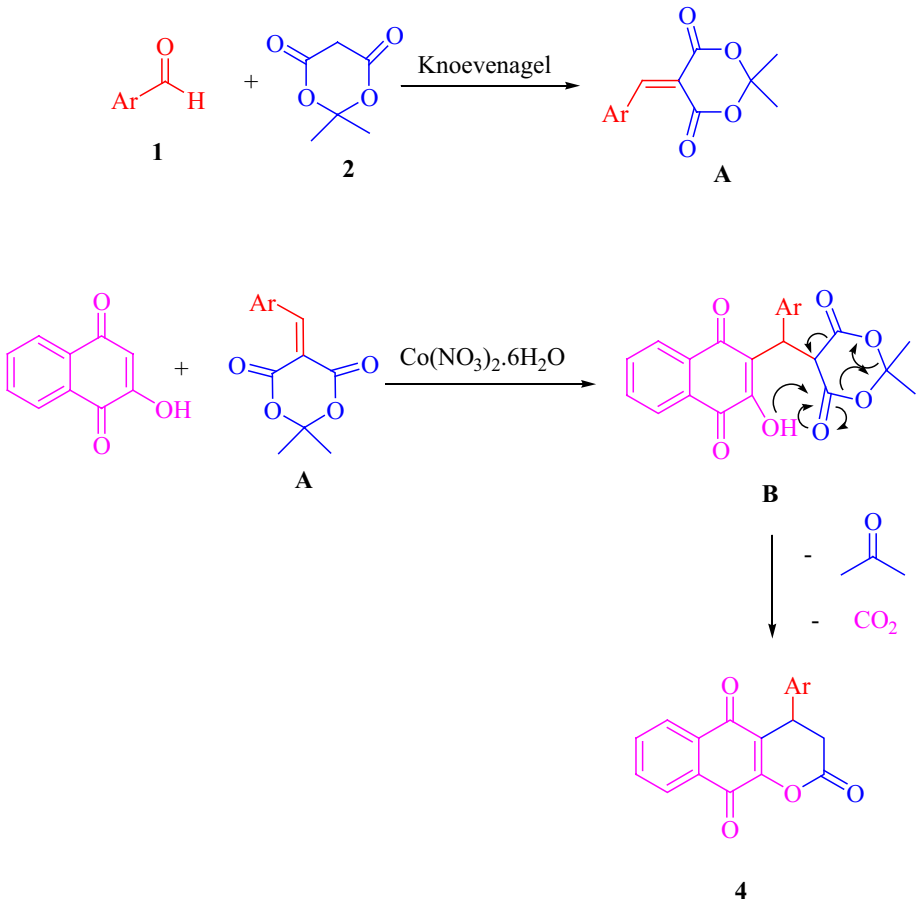
In order to assess the efficiency and generality of this methodology, the result from the reaction of 4-chlorobenzaldehyde and Meldrum's acid with 2-hydroxynaphtalene-1,4-dione by this method has been compared with those of the previously reported methods [24–26] (Table 3). According to the green chemistry, if possible, synthetic methods should be conducted at room temperature and pressure [30]. This methodology was performed at room temperature for economic and environmental impacts.

Recovery of the catalysts is important in green organic synthesis. Thus, we also for recyclability of the catalysts, investigated the recycling of $Co(NO_3)_2{\cdot}6H_2O$ under reaction conditions using a selected model reaction of 4-methylbenzaldehyde, Meldrum's acid, and 2-hydroxynaphthalene-1,4-dione. After completion of the reaction, the thick precipitation was filtered off and washed with ethanol (3×2 cm^3) to remove the catalyst. The $Co(NO_3)_2{\cdot}6H_2O$ was dissolved in EtOH and filtered for separation of the crude product. The separated product was washed twice with EtOH (2×5 mL). The resulting product subsequently recrystallized from hot ethanol to give the pure solid. In order to recover the catalyst, since $Co(NO_3)_2{\cdot}6H_2O$ is soluble in EtOH, the filtrate was extracted with diethyl ether. The aqueous layer was separated, and its solvent was evaporated under reduced pressure and $Co(NO_3)_2{\cdot}6H_2O$ was recovered and reused. The catalytic system worked well up to five catalytic runs. The

Table 3 Comparison of the efficiency of $Co(NO3)_2{\cdot}6H_2O$ with other reported catalysts in literature

Entry	Catalyst/condition	Time	Yield (%)	References
1	HOAc/MWI	4 min	93	[24]
2	L-prolin/H_2O, reflux	4 h	73	[25]
3	PEG-Ni nps/EG, r.t	10 min	79	[26]
4	$Co(NO_3)_2{\cdot}6H_2O$/EtOH, r.t	10 min	90	[Present wok]

Scheme 3 The proposed mechanism for the synthesis of phenylbenzo[*g*]chromenes



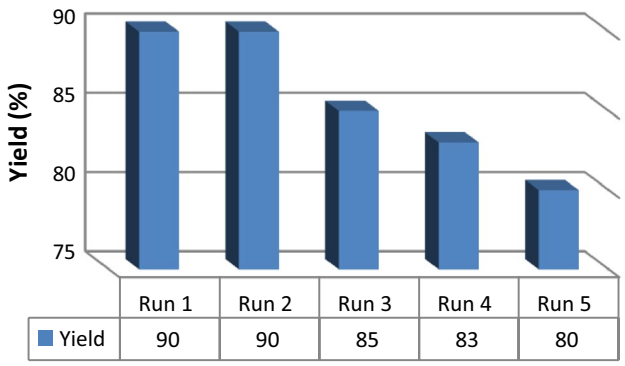


Fig. 2 Results of recycling over five consecutive recycling experiments

recovered catalyst was reused five times without any loss of its activities (Fig. 2).

Conclusion

In conclusion, $Co(NO_3)_2{\cdot}6H_2O$ is a simple and efficient catalyst for the three-component synthesis of medicinally relevant phenylbenzo[g]chromenes. The reaction is performed under mild conditions. The procedure utilizes the simple equipment and easily carried out. It is also valuable from the viewpoint of diversity-oriented large-scale processes. The catalyst shows an environmental-friendly feature which is inexpensive, clean, safe, nontoxic, reusable and easily obtained. Moreover, the procedure offers several advantages including high yields, operational simplicity, clean reaction conditions, and the minimum pollution of the environment, which make it a useful and attractive process for the synthesis of these compounds.

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