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β-Cyclodextrin as an efficient catalyst for the one-pot synthesis of tetrahydrobenzo[b]pyran derivatives in water

Jun Lu[1,2,3] · Xue-wen Fu[1] · Ge Zhang[2,3] · Chun Wang[1]

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Abstract Various 4*H*-benzo[b]pyran derivatives were synthesized via a one-pot three-component condensation of arylaldehyde, malononitrile and dimedone in water using β-cyclodextrin (β-CD) as an efficient catalyst. This method provides several advantages, including mild reaction conditions, operational simplicity, high yields and minimal pollution of the environment.

Keywords 4*H*-benzo[b]pyran · Aqueous media · β-Cyclodextrin · One-pot reaction

Introduction

Synthesis of 4*H*-benzo[b]pyran and its derivatives has attracted great interest from organic and medicinal chemists, because of these products' useful biological and pharmacological properties, such as spasmolytic, diuretic, anti-coagulant, anti-cancer, and anti-anaphylactic activities [1–5]. 4*H*-pyrans is an important structural unit of a series of natural products [6, 7]. Additionally, some benzo[b]pyran derivatives have photochemical activities [8]. Numerous synthetic methods have been reported for the synthesis of 4*H*-pyrans, involving use of a variety of catalysts such as TEBA [9], $RE(PFO)_3$ [10], TBAF [11], TMAH [12], $CeCl_3$ [13], Na_2SeO_4

✉ Chun Wang
wangchun@cib.ac.cn

[1] Chengdu Institute of Biology, Chinese Academy of Sciences, Chengdu 610041, People's Republic of China

[2] Institute for Advancing Translational Medicine in Bone and Joint Diseases, School of Chinese Medicine, Hong Kong Baptist University, Hong Kong, People's Republic of China

[3] Institute of Integrated Bioinformedicine and Translational Science, HKBU Shenzhen Research Institute and Continuing Education, Shenzhen, People's Republic of China

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[14], Phenylboronic acid [15], DABCO [16], ZnO-beta zeolite [17], or the Ru(II) complex [18]. However, these methods suffer from a variety of drawbacks, such as the use of hazardous and toxic solvents, high temperature, and difficult workup. Water is one of the most abundant, cheapest, and environmentally friendly solvents. However, most organic reactants are not soluble in aqueous media. Thus, an efficient phase transfer reagent is necessary to ensure the reaction is conducted in aqueous media. Recently, Azath et al. [19] reported a new method to prepare 2-amino-4*H*-benzo[b]pyran, using per-6-amino-β-cyclodextrin under a solvent-free condition. Despite the difficulty in preparing the catalyst, organic solvent(s) were still needed in the product isolation step.

In this paper, we describe a convenient, inexpensive and environmentally friendly method for the synthesis of 4*H*-benzo[b]pyran, using β-CD in water.

Experimental design

All starting materials were purchased from commercial sources and used without further purification. Melting points were measured by the X-6 (Beijing Fukai Co., Ltd., Beijing, China) melting point apparatus and are uncorrected. Infrared (IR) spectra were recorded on a Perkin Elmer Spectrum One FTIR spectrometer in KBr. ^{1}H and ^{13}C nuclear magnetic resonance (NMR) (600 and 150 MHz, respectively) spectra were determined on Bruker AVANCE-600 spectrometers. Electrospray ionization mass spectrometry (ESI-MS) spectra were recorded on a BioTOF Q mass spectrometer. All the spectrometers were provided by Chengdu Institute of Biology, Chinese Academy of Sciences, P. R. China.

General procedure for synthesis target compounds

Substrates (**1a**–**1p**, 1.0 mmol), malononitrile **2** (1.1 mmol), dimedone **3** (1.1 mmol) and β-CD (0.02 mmol) were added to a round-bottom flask containing 5 mL water. The mixture was stirred at room temperature for a certain time to complete the reaction (monitored by TLC, Table 2). After completion, the generated solid was filtered off and further purified by recrystallization with 95 % ethanol to give a pure product.

*2-Amino-4-(4-methoxy-phenyl)-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (**4b**)* Yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$): $\delta = 1.03$ (s, 3 H), 1.10 (s, 3 H), 2.20 (d, $J = 16.2$ Hz, 1 H), 2.23 (d, $J = 16.2$ Hz, 1 H), 2.41 (d, $J = 17.7$ Hz, 1 H), 2.45 (d, $J = 17.7$ Hz, 1 H), 3.76 (s, 3 H), 4.36 (s, 1 H), 4.52 (s, 2 H), 6.81 (d, $J = 8.6$ Hz, 2 H), 7.14 (d, $J = 8.7$ Hz, 2 H). IR (KBr): $\nu_{max} = 3381$, 3188, 2929, 2192, 1682, 1655, 1606, 1509, 1369 cm^{-1}. ESI–MS: $m/z = 347.1$ $[M+Na]^+$.

*2-Amino-4-(4-hydroxy-phenyl)-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (**4d**)* Yellow solid; ^{1}H NMR (600 MHz, DMSO-d_6): $\delta = 0.93$ (s, 3 H), 1.01 (s, 3 H), 2.05 (d, $J = 4.1$ Hz, 1 H), 2.07 (d, $J = 4.1$ Hz, 1 H), 2.21 (d, $J = 16.1$ Hz, 1 H), 2.44 (d, $J = 17.6$ Hz, 1 H), 4.04 (s, 1 H), 6.63 (d, $J = 8.7$ Hz, 2 H), 6.87 (s, 2 H, NH_2), 6.91 (d, $J = 8.5$ Hz, 2 H), 9.21 (s, 1 H). IR (KBr): $\nu_{max} = 3392$, 3322, 2961, 2195, 1682, 1651, 1512, 1369 cm^{-1}. ESI–MS: $m/z = 333.1$ $[M+Na]^+$.

*2-Amino-4-(4-fluoro-phenyl)-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (**4f**)* White solid; ^{1}H NMR (600 MHz, $CDCl_3$): $\delta = 1.03$ (s, 3 H), 1.11 (s, 3 H), 2.19 (d, $J = 16.4$ Hz, 1 H), 2.19 (d, $J = 16.4$ Hz, 1 H), 2.44 (s, 2 H), 4.40 (s, 1 H), 4.57 (s, 2 H), 6.97 (t, $J = 8.6$ Hz, 2 H), 7.20 (dd, $J = 5.2$, 5.3 Hz, 1 H). IR (KBr): $v_{max} = 3368$, 3188, 2963, 2189, 1684, 1651, 1604, 1506, 1370 cm^{-1}. ESI–MS: $m/z = 335.1$ $[M+Na]^+$.

*2-Amino-4-(4-chloro-phenyl)-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (**4g**)* White solid; ^{1}H NMR (600 MHz, $CDCl_3$): $\delta = 1.03$ (s, 3 H), 1.11 (s, 3 H), 2.19 (d, $J = 16.4$ Hz, 1 H), 2.24 (d, $J = 16.4$ Hz, 1 H), 2.45 (s, 2 H), 4.39 (s, 1 H), 4.56 (s, 2 H), 7.17 (d, $J = 8.4$ Hz, 2 H), 7.26 (d, $J = 8.4$ Hz, 2 H). IR (KBr): $v_{max} = 3396$, 3212, 2961, 2199, 1681, 1660, 1604, 1371 cm^{-1}. ESI–MS: $m/z = 351.1$ $[M+Na]^+$.

*2-Amino-7,7-dimethyl-4-(4-nitro-phenyl)-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (**4h**)* Yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$): $\delta = 1.03$ (s, 3 H), 1.13 (s, 3 H), 2.19 (d, $J = 16.5$ Hz, 1 H), 2.26 (d, $J = 16.5$ Hz, 1 H), 2.48 (s, 2 H), 4.52 (s, 1 H), 4.65 (s, 2 H), 7.42 (d, $J = 8.8$ Hz, 2 H), 8.16 (d, $J = 8.7$ Hz, 1 H). IR (KBr): $v_{max} = 3396$, 3212, 2925, 2192, 1735, 1682, 1651, 1604, 1467 cm^{-1}. ESI–MS: $m/z = 362.1$ $[M+Na]^+$.

*2-Amino-7,7-dimethyl-4-(3-nitro-phenyl)-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (**4i**)* Yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$): $\delta = 1.04$ (s, 3 H), 1.13 (s, 3 H), 2.19 (d, $J = 16.3$ Hz, 1 H), 2.26 (d, $J = 16.3$ Hz, 1 H), 2.46 (d, $J = 17.7$ Hz, 1 H), 2.51 (d, $J = 17.7$ Hz, 1 H), 4.53 (s, 1 H), 4.72 (s, 2 H), 7.48 (t, $J = 7.9$ Hz, 1 H), 7.67 (d, $J = 7.7$ Hz, 1 H), 8.04 (t, $J = 1.7$ Hz, 1 H), 8.08 (d, $J = 7.9$ Hz, 1 H). IR (KBr): $v_{max} = 3391$, 3181, 2959, 2184, 1682, 1651, 1601, 1425, 1372 cm^{-1}. ESI–MS: $m/z = 362.1$ $[M+Na]^+$.

*2-Amino-7,7-dimethyl-4-(2-nitro-phenyl)-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (**4j**)* Yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$): $\delta = 0.99$ (s, 3 H), 1.05 (s, 3 H), 2.12 (d, $J = 16.0$ Hz, 1 H), 2.19 (d, $J = 16.0$ Hz, 1 H), 2.45 (s, 2 H), 4.71 (s, 2 H), 5.19 (s, 1 H), 7.32 (m, 2 H), 7.51 (t, $J = 7.4$, 8.0 Hz, 1 H), 7.79 (d, $J = 8.0$ Hz, 1 H). IR (KBr): $v_{max} = 3470$, 3372, 2924, 2193, 1687, 1663, 1525, 1469, 1364 cm^{-1}. ESI–MS: $m/z = 362.1$ $[M+Na]^+$.

*2-Amino-4-(3,4-dimethoxy-phenyl)-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (**4l**)* Yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$): $\delta = 1.04$ (s, 3 H), 1.10 (s, 3 H), 2.19 (d, $J = 16.4$ Hz, 1 H), 2.24 (d, $J = 16.4$ Hz, 1 H), 2.44 (s, 2 H), 3.82 (s, 3 H), 1.86 (s, 3 H), 4.34 (s, 1 H), 4.79 (s, 2 H), 6.75 (dd, $J = 1.9$, 1.9 Hz, 1 H), 6.79 (d, $J = 8.0$ Hz, 2 H). IR (KBr): $v_{max} = 3393$, 3328, 2932, 2193, 1680, 1657, 1606, 1513, 1365 cm^{-1}. ESI–MS: $m/z = 377.1$ $[M+Na]^+$.

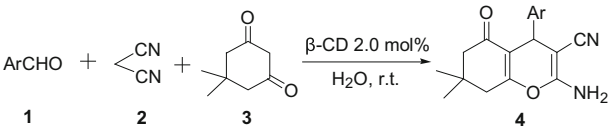
*2-Amino-4-pyridin-7,7-dimethyl-5-oxo-3-yl-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (**4m**)* Yellow solid; ^{1}H NMR (600 MHz, $CDCl_3$): $\delta = 1.02$ (s, 3 H), 1.11 (s, 3 H), 2.19 (d, $J = 16.4$ Hz, 1 H), 2.24 (d, $J = 16.4$ Hz, 1 H), 2.44 (d, $J = 17.8$ Hz, 1 H), 2.48 (d, $J = 17.8$ Hz, 1 H), 4.43 (s, 1 H), 4.96 (br, 2 H), 7.24 (dd, $J = 4.9$, 7.8 Hz, 1 H), 7.62 (td, $J = 1.8$, 7.8 Hz, 1 H), 8.45 (dd, $J = 1.4$, 4.7 Hz, 1 H), 8.49 (d, $J = 2.2$ Hz, 1 H). ^{13}C NMR (150 MHz, $CDCl_3$): $\delta = 27.71$, 28.75, 32.22, 33.64, 40.66, 50.55, 61.73, 113.16, 118.38, 123.59, 135.95, 139.00,

147.89, 148.79, 158.07, 162.13, 195.79. IR (KBr): v_{max} = 3431, 3336, 2956, 2186, 1679, 1599, 1530, 1349 cm^{-1}. ESI–MS: m/z = 318.1 $[M+Na]^+$.

*2-Amino-4-isopropyl-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (**4o**)* White solid; ^1H NMR (600 MHz, $CDCl_3$): δ = 0.74 (d, J = 6.7 Hz, 3 H), 1.04 (d, J = 7.1 Hz, 3 H) 1.10 (d, J = 9.6 Hz, 6 H), 1.90 (m, 1 H), 2.26 (d, J = 16.4 Hz, 1 H), 2.30 (d, J = 16.4 Hz, 1 H), 2.38 (s, 2 H), 3.34 (d, J = 2.3 Hz, 1 H), 4.75 (s, 2 H). IR (KBr): v_{max} = 3387, 3207, 2924, 2194, 1657, 1380 cm^{-1}. ESI–MS: m/z = 283.1 $[M+Na]^+$.

Results and discussion

β-CD is a water-soluble cyclic oligosaccharide possessing hydrophobic cavities; it binds substrates selectively and catalyzes chemical reactions by involving the reversible formation of host–guest complexes with noncovalent bonding, as seen in enzymes [20, 21]. Therefore, it is widely used as a catalyst for organic reactions in



Scheme 1 Synthesis of 4*H*-benzo[b]pyran derivatives

Table 1 One-pot synthesis of 4*H*-benzo[b]pyran **4g** in the presence of different conditions[a]

Entry	Additive (mmol %)	Time (h)	Temp (°C)	Yield[b] (%)
1	–	5.0	r.t.	23[c]
2	TBAC (10)[d]	5.0	r.t.	81
3	HTAB (10)[d]	5.0	r.t.	70
4	TEAI (10)[d]	5.0	r.t.	53
5	β-CD (10)	5.0	r.t.	94
6	β-CD (4.0)	5.0	r.t	93
7	β-CD (1.0)	5.0	r.t.	82
8	β-CD (2.0)	5.0	r.t.	93
9	β-CD (2.0)	3.5	40	93
10	β-CD (2.0)	2.5	60	92
11	β-CD (2.0)	1.0	80	94

[a] Condition: the reaction was performed by using 4-chlorobenz-aldehyde (1.0 mmol), malononitrile (1.1 mmol), dimedone (1.1 mmol), in 5 ml water

[b] Yields refer to isolated products

[c] Yield of the product isolated after column chromatography

[d] *TBAC* tetrabutyl ammonium chloride, *HTAB* hexadecyl trimethyl ammonium bromide bromide, *TEAI* tetraethyl ammonium iodide

Table 2 Synthesis of 4*H*-benzo[b]pyrans catalyzed by β-CD[a]

Entry	Ar	Product[b]	Yield[c] (%)	Time (h)	Mp (°C)	
					Found	Reported
1	C_6H_5	**4a**	92	5.0	224–226	226–228 [13]
2	4-MeO–C_6H_4	**4b**	88	5.0	199–200	198–200 [12]
3	4-Me–C_6H_4	**4c**	91	5.0	212–214	214–216 [12]
4	4-OH–C_6H_4	**4d**	91	5.0	208–210	204–205 [10]
5	3-OH–C_6H_4	**4e**	90	5.0	230–232	236–238 [10]
6	4-F–C_6H_4	**4f**	94	5.0	210–212	210–211 [13]
7	4-Cl–C_6H_4	**4g**	93	5.0	210–212	207–209 [13]
8	4-NO_2–C_6H_4	**4h**	94	5.0	177–179	177–178 [12]
9	3-NO_2–C_6H_4	**4i**	93	5.0	210–212	208–211 [12]
10	2-NO_2–C_6H_4	**4j**	86	5.0	223–225	224–226 [12]
11	3,4-Cl_2–C_6H_3	**4k**	89	5.0	253–255	255–257 [10]
12	3,4-$(CH_3O)_2$–C_6H_3	**4l**	83	5.0	175–177	173–174 [13]
13	3-Pyridine	**4m**	90	5.5	207–209	–[d]
14	2-Furan	**4n**	91	5.0	225–227	226–228 [13]
15	$(CH_3)_2CH$	**4o**	72	9.0	179–181	–[e]
16	$C_6H_5CH{=}CH$	**4p**	83	7.5	182–183	182–184 [11]

[a] Condition: the reaction was performed by using aldehyde 1 (1.0 mmol), malononitrile 2 (1.1 mmol), and dimedone 3 (1.1 mmol) in the present of 2.0 mol % of β-CD in 5 mL H_2O at the room temperature

[b] All products were characterized by ^{1}H NMR, IR spectroscopy and mass spectroscopy

[c] Yield refer to isolated products

[d] Novel compound

[e] Melting point not reported

water [22]. As part of our efforts to develop a green chemistry through the one-pot synthesis of target molecules in water, we report here the Knoevenagel-Cyclocondensation reaction of arylaldehyde **1**, malononitrile **2**, and dimedone **3**, for the preparation of 4*H*-benzo[b]pyran derivatives, using β-CD as a catalyst in aqueous medium (Scheme 1).

In a typical experimental procedure, we examined the effects of the amount of catalysts, reaction time and temperature on the reaction to optimize the conditions of the model reaction of 4-chlorobenzaldehyde **1g**, malononititrile **2** with dimedone **3** (Scheme 1). Although Azath et al. stated that under β-CD and water condition, no product was observed [19], our results (Table 1) suggested that β-CD certainly catalyzed the three-component reaction (Table 1, entries 1 and 5). The three-component reaction of 4-chlorobenzaldehyde **1g**, malononititrile **2** with dimedone **3**, in the absence of any catalyst, in water for 5 h gave a corresponding **4g** with only 23 % yield (Table 1, entry 1). In addition, β-CD showed the most catalytic activity among the screened catalysts (Table 1, entries 2–5). The amount of β-CD was also critical to the reaction. We found that 2.0 mol % of catalyst was preferred, that decreasing the amount of catalyst resulted in poor yield, and that increasing the

amount of catalyst showed no improvement of yields (Table 1, entries 5–8). Further study on temperature effects revealed that from 25 to 80 °C, the temperature has little effect on the yields, but accelerated the reaction from 5 to 1 h (Table 1, entries 8–11).

Having established the reaction conditions, various arylaldehydes were treated with malononitrile and dimedone to investigate the reaction scope. As summarized in Table 2, the electron-withdrawing group (such as –F, –Cl, $–NO_2$) or the electron-donating group (such as $–OCH_3$, $–CH_3$, –OH) on the aromatic ring of aldehyde has no distinct impact on the reaction (entries 2–12, Table 2). However, the steric properties of the arylaldehydes have great impact on the efficiency of the reaction. Arylaldehydes without substituents or with mono-substitutes showed generally higher efficiency than those with disubstituent aldehydes (entry 1–9 and 11–12, Table 2). Bulky groups at ortho- positions of the aldehydes showed a relatively lower yield (entries 10, Table 2). Alkylaldehyde was less reactive compared with arylaldehydes (entry 15, Table 2). Our results indicate that the catalysis process involves the formation of a size-selective inclusion complex between arylaldehyde and β-CD.

Complexation of β-CD and arylaldehydes has been reported by Rao et al. [23]. Hydrophobic interactions between the hydrophobic cavity of β-CD and the aromatic ring were the driving force in the inclusion process, but hydrogen bonding between primary or second hydroxyl groups of β-CD and polar substituents of the arylaldehydes as well as the directions of the substituents may also be important factors in the formation of the complexes. The catalyst is also found to be reusable. Up to three times, there is little change in its catalytic efficiency.

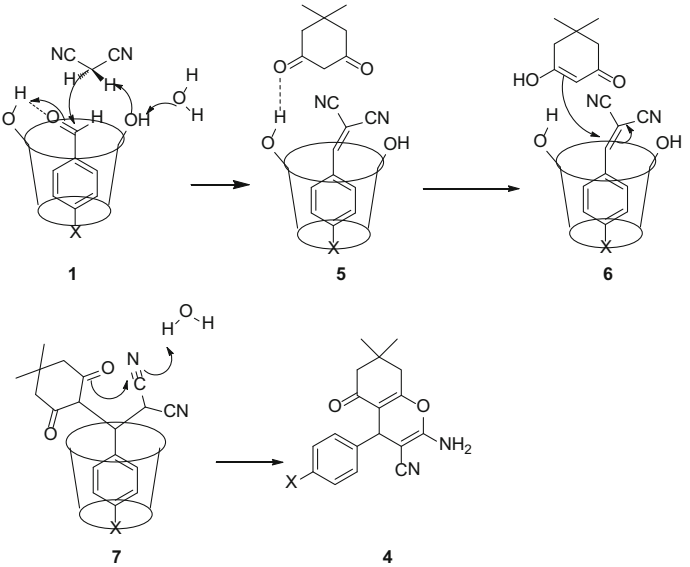


Fig. 1 Proposed reaction mechanism of β-Cyclodextrin-catalyzed synthesis of 4*H*-benzo[b]pyran derivatives

A proposed mechanism for the reaction is outlined in Fig. 1. The reaction was promoted by the formation of an inclusion complex between β-CD and arylaldehyde **1**. The intermediate cyano olefin **5** was readily formed in situ from Knoevenagel condensation between β-CD solubilized aryladelhyde **1** and active methylene compound **2** in water. This can be evidenced by the steric effects of the arylaldehydes on the reaction efficiency (Table 2). β-CD also catalyzed the formation of the enolic form of dimedone **3** through hydrogen bonding stabilization, which could easily react with cyano olefin **5** and give intermediate **6**, followed by cyclization and tautomerization of **7** to give the final products **4**. It is worth pointing out that this transformation may not need a base catalysis as suggested by Moustafa et al. [24], as evidenced by our experimental results and other non-base catalyzed synthesis of 4*H*-benzo[b]pyran [9, 13, 15].

Conclusion

We have demonstrated that β-CD is an efficient catalyst for the synthesis of 4*H*-benzo[b]pyran derivatives in aqueous media. Compared with previously reported procedures, our protocol provides advantages, such as operational simplicity, excellent yields, an environmentally benign catalyst and solvent, and mild reaction conditions, that make it a useful and attractive method for the preparation of these compounds.

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