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Efficient, one-pot synthesis of xanthene derivatives using boron sulphonic acid as a solid heterogeneous catalyst under solvent-free conditions

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Abstract An efficient, convenient and green method has been introduced for the synthesis of biologically active xanthene derivatives through a one-pot condensation of aldehydes, 2-naphthol, and dimedone/naphthols in the presence of boron sulphonic acid ($B(HSO_4)_3$) as an efficient heterogeneous reusable solid acid catalyst under solvent-free conditions with good to excellent yields. Different types of aromatic and aliphatic aldehydes were used in the reaction and in all cases the products synthesized successfully. In addition, short reaction times, straightforward procedure, non-toxicity, and reusability of the catalyst are other noteworthy advantages of the present method.

Keywords Xanthenes · Boron sulphonic acid · Solid-acid catalyst · One-pot reaction · Solvent-free

Introduction

The synthesis of xanthenes and benzoxanthenes has gained considerable attention in organic synthesis because of their wide range of therapeutic and biological properties, such as agricultural bactericide activity [1], anti-inflammatory [2], and antiviral activity [3]. They have also been used for photodynamic therapy [4], used as dyes [5], and as fluorescent material for visualization of biomolecules [6] and in laser technologies [7]. Thus, a broad utility range has made xanthenes prime synthetic candidates, thereby accentuating the need to develop newer synthetic routes for scaffold manipulation of xanthene derivatives.

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Various methods are available for the construction of xanthenes and benzoxanthenes involving trapping of benzynes by phenols [8], cyclo-condensation reaction between 2-hydroxyaromatic aldehydes and 2-tetralone [9], intramolecular phenyl carbonyl coupling reactions of benzaldehydes and acetophenones [10], and the reaction of 2-naphthol with 2-naphthol-1-methanol [11] and carbon monoxide [12]. However, these methods have many disadvantages such as low yields, the need for a prolonged reaction time, the use of toxic organic solvents, excess reagents, and harsh reaction conditions. Because of these drawbacks, the reaction has been improved by mixing aldehydes with 2-naphthol or dimedone in the presence of a catalyst, such as *p*-TSA [13, 14], Na^+-montmorillonite sulfonic acid [15], molecular iodine [16], silica sulfuric acid [17], amberlyst-15 [18], wet cyanuric choloride [19], $K_{12}[As_2W_{18}Cu_3O_{68}]\cdot 30H_2O$ and $K_{12}[As_2W_{18}U_3O_{74}]\cdot 21H_2O$ [20], boric acid [21], PVPP-BF_3 [22], ceric ammonium nitrate (CAN) [23], and $InCl_3$ [24].

The increasing demand for clean and efficient chemical reactions has resulted in solvent-free reaction conditions, which are of great current interest. Also, economic and environmental concerns encourage the application of heterogeneous catalysts to carry out various organic transformations. These catalysts can conveniently be handled and removed from the reaction mixture, making the experimental procedure simple and ecofriendly. Recently, $B(HSO_4)_3$ was found to be an effective solid acid catalyst in the synthesis of thiocyanohydrins [25] and anthraquinone derivatives [26]. In continuation of our studies on the development of practical, safe, and environmentally friendly procedures for some important transformations [14, 27, 28], we had an opportunity to explore the catalytic activity of $B(HSO_4)_3$ towards the synthesis of xanthenes under solvent-free reaction conditions.

Experimental

General

All products were characterized by comparison of their spectral (FT-IR, ^{1}H NMR, ^{13}C NMR) data or by comparison of their physical and spectroscopic data with those reported in the literature. Melting points were measured by using capillary tubes on an electrothermal digital apparatus and are uncorrected. The progress of the reactions was monitored by thin-layer chromatography (TLC) using *n*-hexane/EtOAc as an eluent. The FT-IR spectrum of the samples were recorded on a Unicom Galaxy Series FT-IR 5000 spectrophotometer in the region 4,000–400 cm^{-1} using pressed KBr discs. The ^{1}H and ^{13}C NMR spectra were recorded on a Brucker Avance spectrometer operating at 300 and 75 MHz for proton and carbon-13, respectively, in DMSO-d_6 or $CDCl_3$ with TMS as an internal standard. Elemental analysis was performed on a Vario EL III elemental analyzer.

Preparation of catalyst (boron sulfonic acid) [25]

Boric acid (0.62 g, 10 mmol) was charged in a 50-mL flask and chlorosulfonic acid (3.50 g, 30 mmol) was added dropwise over a period of 1 h at room temperature.

HCl evolved immediately. After completion of the addition, the mixture was shaken for 1 h, while the residual HCl was eliminated. The mixture was then washed with diethyl ether to remove the unreacted chlorosulfonic acid. Finally, the obtained solid catalyst was dried under vacuum.

General procedure for the preparation of 14-aryl-14*H*-dibenzo[*a,j*]xanthenes **(5a–5f)**

A mixture of aldehyde (1 mmol), 2-naphthol (2 mmol) and boron sulphonic acid (0.10 mmol) was stirred at 120 °C for the appropriate time indicated in Table 2. The progress of the reactions was monitored by TLC (eluent: EtOAc/*n*-hexane, 1:5). After completion of the reaction, the reaction mixture was cooled to room temperature, the crude product was heated in ethanol (30 mL), and the catalyst was removed by filtration. The pure product was obtained by cooling of the filtrate.

General procedure for the preparation of tetrahydrobenzo[*a*]xanthen-11-one derivatives **(6a–6h)**

A mixture of aldehyde (1 mmol), 2-naphthols (1 mmol), dimedone (1.1 mmol) and boron sulphonic acid (0.10 mmol) was stirred at 120 °C for the appropriate time indicated in Table 2. The progress of reactions was monitored by TLC (eluent: EtOAc/*n*-hexane, 1:5). After completion of the reaction, the reaction mixture was cooled to room temperature, the crude product was heated in ethanol (30 mL), and the catalyst was removed by filtration. The pure product was obtained by cooling of the filtrate.

Spectral data of new compounds

*Compound **5a** (Table 2, Entry 1)*

IR (KBr): ν_{max} = 3,069, 2,992, 2,936, 2,826, 1,624, 1,589, 1,506, 1,458, 1,418, 1,321, 1,240, 1,181, 1,125, 1,080, 1,005, 957, 818, 748, 671, 540 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ = 8.42 (d, J = 8.52 Hz, 2H), 7.87 (d, J = 8.1 Hz, 2H), 7.82 (d, J = 8.9 Hz, 2H), 7.61 (t, J = 7.1 Hz, 2H), 7.50 (d, J = 8.9 Hz, 2H), 7.45 (t, J = 7.9 Hz, 2H), 6.72 (s, 2H), 6.46 (s, 1H), 3.73 (s, 6H), 3.67 (s, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 153.2, 148.9, 140.6, 136.9, 131.6, 131.2, 128.9, 128.8, 126.7, 124.3, 122.8, 117.9, 117.4, 106.0, 60.7, 56.2, 38.1 ppm; Anal. calcd. for $C_{30}H_{24}O_4$:C, 80.34; H, 5.39. Found: C, 80.26; H, 5.43.

*Compound **5f** (Table 2, Entry 6)*

IR (KBr): ν_{max} = 3,063, 2,985, 2,934, 2,845, 1,663, 1,595, 1,516, 1,462, 1,391, 1,287, 1,244, 1,080, 804, 746 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ = 8.07 (d, J = 8.4 Hz, 2H), 7.89 (d, J = 8.1 Hz, 2H), 7.80 (d, J = 8.9 Hz, 2H), 7.67 (t, J = 7.1 Hz, 2H), 7.50 (t, J = 7.8 Hz, 2H), 7.35 (d, J = 8.9 Hz, 2H), 4.64 (s, 2H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 147.7, 132.3, 130.4, 128.9, 128.7, 127.4,

124.9, 123.5, 117.8, 111.7, 22.3 ppm; Anal. calcd. for $C_{21}H_{14}O$:C, 89.34; H, 5.00. Found: C, 89.39; H, 5.05.

*Compound **6a** (Table 2, Entry 7)*

IR (KBr): ν_{max} = 3,183, 3,055, 3,003, 2,955, 2,841, 1,626, 1,514, 1,451, 1,379, 1,233, 1,138, 1,028, 829, 756 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6): δ = 9.47 (s, 1H), 7.64 (d, J = 8.8 Hz,1H), 7.61 (d, J = 8.8 Hz, 1H),7.29 (s, 1H), 7.09 (d, J = 8.8 Hz, 1H), 6.97 (s, 1H), 6.95 (d, J = 8.6 Hz, 1H), 6.72 (d, J = 8.2 Hz, 1H), 6.65 (d, J = 8.2 Hz, 1H), 5.38 (s, 1H), 3.78 (s, 3H), 3.71 (s,3H), 2.57 and 2.56 (AB system, J = 4.4 Hz, 2H), 2.18 and 2.29 (AB system, J = 16.3 Hz, 2H), 1.12 (s, 3H), 0.96 (s, 3H) ppm; ^{13}C NMR (75 MHz, DMSO-d_6): δ = 196.4, 164.1, 156.9, 148.7, 148.1, 147.7, 137.8, 133.1, 130.6, 129.0, 125.9, 120.6, 117.6, 116.2, 113.9, 112.9, 112.1, 105.8, 56.0, 55.8, 50.6, 34.1, 32.2, 29.3, 26.6 ppm; Anal. calcd. for $C_{27}H_{26}O_5$:C, 75.33; H, 6.09. Found: C, 75.25; H, 6.12.

*Compound **6b** (Table 2, Entry 8)*

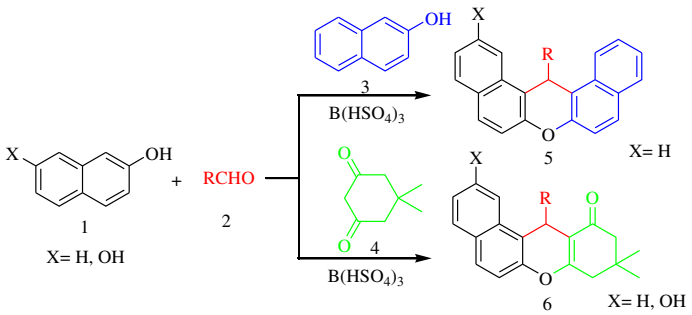
IR (KBr): ν_{max} = 3,227, 3,081, 2,961, 2,891, 1,630, 1,518, 1,451, 1,381, 1,238, 1,182, 1,030, 841, 698, 617, 530, 442 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6): δ = 9.86 (s, 1H), 8.08 (d, J = 7.2 Hz, 2H), 7.80 (d, J = 8.8 Hz, 1H), 7.75 (d, J = 8.9 Hz, 1H), 7.45 (d, J = 7.3 Hz, 2H), 7.21 (d, J = 8.8 Hz, 1H), 7.15 (s, 1H), 6.97 (d, J = 8.8 Hz, 1H), 5.5 (s, 1H), 2.58 and 2.69 (AB system, J = 17.6 Hz, 2H), 2.13 and 2.34 (AB system, J = 16.3 Hz, 2H), 1.06 (s, 3H), 0.86 (s, 3H) ppm; ^{13}C NMR (75 MHz, DMSO-d_6): δ = 196.4, 164.9, 157.1, 152.5, 148.1, 146.3, 132.8, 130.8, 129.9, 129.8, 125.9, 123.8, 117.8, 114.5, 114.0, 112.6, 105.5, 50.4, 34.9, 32.2, 29.2, 26.6 ppm; Anal. calcd. for $C_{25}H_{21}NO_5$:C, 72.28; H, 5.10; N, 3.37. Found: C, 72.34; H, 5.13; N, 3.44.

*Compound **6e** (Table 2, Entry 11)*

IR (KBr): ν_{max} = 3,187, 2,959, 2,932, 2,870, 1,624, 1,595, 1,451, 1,397, 1,235, 1,175, 1,150, 1,028, 833 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ = 9.12 (s, 1H), 7.83 (s, 1H), 7.73 (d, J = 8.8 Hz, 1H), 7.63 (d, J = 8.8 Hz, 1H), 7.14 (d, J = 8.8 Hz, 1H), 7.02 (d, J = 8.8 Hz, 1H), 4.81 (s, 1H), 2.60 (s, 2H), 2.51 (s, 2H), 1.91 (q, J = 5.7 Hz, 2H), 1.24–1.20 (s, 6H), 1.07 (m, 4H), 0.90 (m, 2H), 0.69 (t, J = 6.5 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ = 200.1, 168.2, 156.3, 148.7, 132.9, 130.3, 127.9, 126.4, 117.3, 116.4, 113.5, 112.8, 105.6, 50.6, 41.5, 34.3, 32.3, 31.9, 29.5, 28.3, 27.2, 24.6, 22.5, 13.9 ppm; Anal. calcd. for $C_{24}H_{28}O_3$:C, 79.09; H, 7.74. Found: C, 79.03; H, 7.80.

Results and discussion

In this paper, the condensation of 2-naphthols, various aldehydes, and 2-naphthol/dimedone in the presence of a heterogeneous solid acid catalyst of $B(HSO_4)_3$ for the preparation of xanthenes derivatives has been studied (Scheme 1).



Scheme 1 Synthesis of xanthene derivatives

Table 1 Optimization of reaction conditions

Entry	Solvent	Conditions	$B(HSO_4)_3$ (mol%)	Time (min)	Yield (%)[a]
1	$CHCl_3$	Reflux	10	180	65
2	C_2H_5OH	Reflux	10	180	43
3	CH_3CN	Reflux	10	180	31
4	THF	Reflux	10	180	72
5	Solvent-free	120 °C	10	10	91
6	Solvent-free	120 °C	–	60	<5
7	Solvent-free	120 °C	20	10	91

[a] Isolated yields

Initially, in order to optimize the reaction parameters, we investigated the catalytic activity of $B(HSO_4)_3$ in the synthesis of xanthene derivatives under different reaction conditions, using the condensation reaction of benzaldehyde and 2-naphthol as a model reaction. As shown in Table 1, among the tested solvents, such as ethanol, CH_3CN, THF, $CHCl_3$, and a solvent-free system, the best result was obtained after 10 min under solvent-free conditions in excellent yield (91 %). When the same reaction was performed in the absence of the catalyst, the corresponding product was obtained in only <5 % yield, whereas, $B(HSO_4)_3$ in excess of 0.2 equival. did not improve the yield to a greater extent (Table 1, entry 7).

After optimizing the conditions, we next examined the scope and generality of this method with respect to various aromatic aldehydes with electron-withdrawing and electron-donating substituents as well as aliphatic aldehydes to prepare a series of dibenzoxanthene derivatives (Table 2). α,β-Unsaturated aldehyde such as cinnamaldehyde was also examined but its yield was not satisfactory even after 1 h. The present methodology afforded good to high yields of the products within short times (10–40 min).

To further show the applicability of this method, the preparation of tetrahydrobenzo[*a*]-xanthene-11-one derivative was also investigated. A variety of aromatic aldehydes bearing electron-withdrawing groups (such as nitro and halide), electron-donating groups (such as methyl and methoxy), and dimedone were treated

Table 2 Synthesis of xanthene derivatives in presence of boron sulphonic acid under solvent-free conditions

Entry	R (aldehyde)	X	Pro.	Time (min)	Yeild (%)[a]	MP (°C)	
						Found	Reported [lit.]
1	3,4,5-$(MeO)_3C_6H_2$	H	**5a**	25	85	192–193	–
2	C_6H_5	H	**5b**	10	91	186–187	184–185 [29]
3	2-ClC_6H_4	H	**5c**	15	85	213–214	214–216 [29]
4	CH_3CH_2	H	**5d**	35	76	154–156	150–152 [30]
5	$(CH_3)_2CHCH_2$	H	**5e**	40	73	114–115	112–113 [31]
6	2-OH-1-naphthyl	H	**5f**	30	39	205–207	–
7	3,4-$(MeO)_2C_6H_3$	OH	**6a**	25	89	228–230	–
8	4-$NO_2C_6H_4$	OH	**6b**	15	93	266–267	–
9	4-MeC_6H_4	OH	**6c**	20	91	302–303	303–305 [32]
10	2-ClC_6H_4	OH	**6d**	15	89	280–281	282–283 [32]
11	$CH_3(CH_2)_3CH_2$	OH	**6e**	60	78	167–169	–
12	4-$MeOC_6H_4$	H	**6f**	30	86	195–197	198–202 [21]
13	C_6H_5	H	**6g**	10	87	156–157	151–154 [21]
14	4-MeC_6H_5	H	**6h**	25	83	178–180	173–175 [21]

[a] Isolated yields

Table 3 The comparison efficiency of $B(HSO_4)_3$ with other reported catalysts for synthesis of **4b** and **4c**

Entry	Catalyst	Conditions	Time (min)/yield (%)[a]		Reference
			5b	**5c**	
1	$B(HSO_4)_3$	Solvent-free/120 °C	10/91	15/93	This method
2	Dowex-50 W	Solvent-free/100 °C	90/78	90/91	[33]
3	Amberlyst-15	Solvent-free/125 °C	120/94	20/91	[18]
4	Cellulose sulfuric acid	Solvent-free/110 °C	90/95	120/93	[34]
5	P(4-VPH)HSO_4	Solvent-free/100 °C	55/94	57/92	[29]
6	$HClO_4$–SiO_2	Solvent-free/125 °C	10/95	10/88	[19]

[a] Isolated yields

with 2-naphthol or 2,7-naphthalenediol under the same experimental conditions, and the corresponding products were obtained in good to high yields without any difficulties (Table 2). Interestingly, aliphatic aldehydes such as hexanal also gave the expected xanthene derivatives in good yield.

In order to assess the efficiency and generality of this methodology, the obtained result from the reaction of benzaldehyde and 2-chlorobenzaldehyde with 2-naphthol and the reaction of benzaldehyde and 4-methoxybenzaldehyde with 2-naphthol and dimedone by this method have been compared with those of the previously reported methods using inorganic or organic catalysts (Tables 3, 4). It was found that the

Table 4 The comparison efficiency of $B(HSO_4)_3$ with other reported catalysts for synthesis of **5f** and **5g**

Entry	Catalyst	Conditions	Time (min)/yield (%)[a]		Reference
			6f	**6g**	
1	$B(HSO_4)_3$	Solvent-free/120 °C	30/86	10/87	This method
2	$InCl_3$	Solvent-free/120 °C	45/76	30/84	[24]
3	P_2O_5	Solvent-free/120 °C	55/71	40/76	[24]
4	TCT	Solvent-free/80 °C	70/86	50/90	[35]
5	$H_4SiW_{12}O_{40}$	Solvent-free/100 °C	15/83	15/89	[36]
6	CCl_3COOH	Solvent-free/120 °C	20/75	30/73	[21]

[a] Isolated yields

Table 5 Reuse of the catalyst for the synthesis of **5b** (Table 2, entry 2)

Run	Time (min)	Yield (%)[a]
1	10	91
2	10	90
3	10	88
4	10	82
5	10	75

All reactions carried out at 120 °C

[a] Isolated yields

$B(HSO_4)_3$ is a fairly good reagent for this reaction with respect to reaction time, yield of product, and amount of the catalyst.

For investigation of the reusability and recycling of $B(HSO_4)_3$ as a catalyst, we stirred a mixture of benzaldehyde (1 mmol) and 2-naphthol (2 mmol) in the presence of $B(HSO_4)_3$ (10 mol%) at 120 °C for 10 min. At the end of the reaction, the $B(HSO_4)_3$ was easily separated by simple filtration and washed with hot ethanol and the recovered catalyst was reused for at least five runs without significant degradation in catalytic activity and performance (Table 5).

Conclusion

In conclusion, this paper describes a convenient and efficient process for the synthesis of tetrahydrobenzo[*a*]xanthen-11-one and dibenzo[*a,j*]xanthene derivatives in the presence of boron sulphonic acid [$B(HSO_4)_3$] under solvent-free conditions at 120 °C. Some advantages of this environmentally safe and benign protocol include a simple reaction set-up, high products yields, short reaction times, and elimination of toxic solvents. The catalyst is readily prepared and inexpensive, and can conveniently be handled and removed from the reaction mixture.

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