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Camphor-10-sulfonic acid catalyzed condensation of 2-naphthol with aromatic/aliphatic aldehydes to 14-aryl/alkyl-14*H*-dibenzo[*a*,*j*]xanthenes

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Abstract: The (±)-camphor-10-sulfonic acid (CSA) catalyzed condensation of 2-naphthol with both aliphatic/aromatic aldehydes at 80 °C yielded 14-alkyl/ /aryldibenzoxanthenes as the sole products in high yields. However, the same condensation with benzaldehyde at 25 °C afforded a mixture of the intermediate 1,1-bis-(2-hydroxynaphthyl)phenylmethane and 14-phenyldibenzoxanthene, while the condensation with aliphatic aldehydes at 25 °C furnished the corresponding 14-alkyldibenzoxanthenes as the sole products. Moreover, the condensation of 2-naphthol with aromatic/aliphatic aldehydes with low catalyst loading (2 mol %) was greatly accelerated under microwave irradiation to afford the corresponding 14-aryl/alkyldibenzoxanthenes as the sole products in high yields.

Keywords: dibenzoxanthenes; *β*-naphthol; (±)-camphor-10-sulfonic acid; microwave irradiation.

INTRODUCTION

Xanthenes and benzoxanthenes are important biologically active heterocycles as they possess several important bioactivities including antiviral,[1] anti-inflammatory,[2] and antibacterial activities.[3] Furthermore, these heterocycles are used as sensitizers in photodynamic therapy,[4] leuco dyes in laser technology,[5] antagonists of the paralyzing action of zoxazolamine (2-amino-5-chlorobenzoxazole)[6] and pH-sensitive fluorescent materials for the visualization of biomolecules.[7] Therefore, the synthesis of various xanthene derivatives is of great synthetic importance. Several methods have been reported for the synthesis of xanthenes and benzoxanthenes, which include trapping of benzynes with phenols,[8] cyclodehydration,[9] cyclocondensation of 2-hydroxyaromatic aldehydes with 2-tetralone[10] and intramolecular phenyl carbonyl coupling reactions of benzaldehydes

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and acetophenones.[11] In addition, 14-aryl-14*H*-dibenzo[*a*,*j*]xanthenes and related compounds have been synthesized from the reaction of 2-naphthol with formamide,[12] 1-(hydroxymethyl)naphthalen-2-ol[13] and carbon monoxide.[14] Moreover, various catalysts, including $TaCl_5$,[15] $Sr(OTf)_2$,[16] $Yb(OTf)_3$,[17] sulfamic acid,[18] iodine,[19] Amberlyst-15,[20] cyanuric chloride,[21] ceric ammonium nitrate (CAN),[22] $BF_3:SiO_2$,[23] P_2O_5 or $InCl_3$,[24] $Sc[N(SO_2C_8F_{17})_2]_3$,[25] $RuCl_3 \cdot nH_2O$,[26] poly(ethylene glycol)–SO_3H (PEG)–SO_3H,[27] an ionic liquid,[28] dodecylphosphonic acid (DPA) or dodecylsulfamic acid (DSA),[29] nano-silica phosphoric acid (nano-SPA),[30] nano-$SnCl_4 \cdot SiO_2$,[31] 4-dodecylbenzenesulfonic acid (DBSA),[32] tungstophosphoric acid[33] and a functionalized mesoporous material[34] were found to catalyze condensation reactions of 2-naphthol with different aldehydes to afford 14-substituted-14*H*-dibenzo[*a*,*j*]xanthenes. However, many of the reported methodologies suffered from one or more disadvantages, such as long reaction time, use of toxic and expensive catalysts, harsh reaction conditions, *etc*. Bearing this in mind, as well as the increasing importance of benzoxanthenes in the pharmaceutical industry, there is still a necessity for the development of an efficient, environmentally benign and inexpensive catalyst for their synthesis. Herein, a convenient microwave-assisted synthesis of 14-aryl/alkyl-14*H*-dibenzo[*a*,*j*]xanthenes from 2-naphthol and aromatic/aliphatic aldehydes using (±)-camphor-10-sulfonic acid as a catalyst is reported.

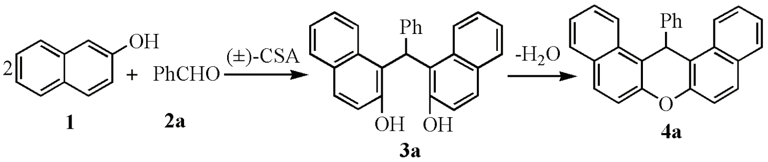
RESULTS AND DISCUSSION

Camphor-10-sulfonic acid (CSA) is an inexpensive and easy to handle organo-catalyst that has been used in various organic transformations. Recently, it was observed that CSA is a highly efficient catalyst for the activation of imines in the Mannich type reaction of enolizable ketones.[35] As an extension to this work, it was considered of interest to explore its catalytic potential for the activation of aldehydes in the condensation of 2-naphthol with aldehydes to afford dibenzoxanthenes. In a preliminary study, the reaction of 2-naphthol (**1**) with benzaldehyde (**2a**, 0.55 equiv.) was performed at ambient temperature (25 °C) with (±)-CSA (5 mol. %) under solvent-free conditions. The reaction was found to be very sluggish as both the intermediate bis-(2-hydroxy-1-naphthyl)phenylmethane (**3a**) as well as 14-phenyl-14*H*-dibenzo[*a*,*j*]xanthene (**4a**) were isolated in 55 and 10 % yields, respectively, along with unreacted **1** (≈18 %) and **2a** (≈10 %) after stirring for 24 h (Table I, entry 1). Compounds **3a** and **4a** were characterized by physical and spectroscopic (IR, ^{1}H-NMR, ^{13}C-NMR and MS) data. Increasing the catalyst loading (10 mol %) (25 °C, 24 h) led to marginal improvements in the yields of both **3a** and **4a**, although small amounts of the respective starting materials were still recovered (Table I, entry 2).

However, with 20 mol % catalyst, both starting compounds were consumed while **3a** and **4a** were isolated in 75 and 21 % yields, respectively (Table I, entry

3). In order to improve the yield and selectivity of the desired product **4a**, the above reaction was performed at an elevated temperature (80 °C) with 10 mol % CSA as the catalyst. Gratifyingly, both the starting materials **1** and **2a** were consumed and **4a** was isolated as a sole product in 81 % yield, while the intermediate **3a** was found to be absent (Table I, entry 4). Further increase in catalyst loading (15 mol %) led to completion of the reaction within 2 h at 80 °C to yield **4a** as the sole product in 89 % yield (Table I, entry 5).

TABLE I. Optimization of the reaction conditions for the (±)-CSA-catalyzed condensation of 2-naphthol (**1**) with benzaldehyde (**2a**)

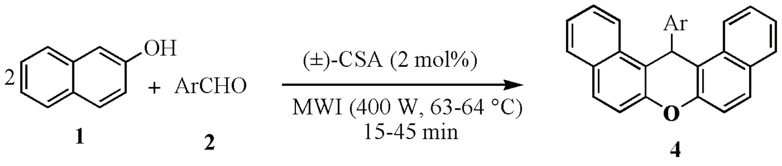


Entry	Reaction conditions[a]	(±)-CSA, mol. %	Time, h	Yield, %[b]	
				3a	**4a**
1	25 °C	5	24.0	55	10
2	25 °C	10	24.0	63	14
3	25 °C	20	16.0	75	21
4	80 °C	10	8.0	–	81
5	80 °C	15	2.0	–	89
6	MWI, 63–64 °C	10	0.25	–	89
7	MWI, 63–64 °C	5	0.25	–	88
8	MWI, 63–64 °C	2	0.25	–	88
9	MWI, 63–64 °C	0	0.50	–	0

[a] 2-Naphthol was reacted with benzaldehyde (0.55 equiv.) under solvent-free conditions; [b] isolated yields

Microwave irradiation (MWI) is known to be an important tool in organic synthesis to improve the selectivity, rate enhancement and reduction of thermal degradative byproducts.[36] To reduce the catalyst loading as well as the reaction time required for the condensation at high temperature, reaction of **1** with **2a** (0.55 equiv.) was performed without solvent using a varied amount of (±)-CSA (2–10 mol %) under MWI (400 W, 63–64 °C) when **4a** was obtained as the sole product in 88–89 % yields (Table I, entries 6–8). The reaction without using CSA as catalyst did not yield any product **4a** and both the starting compounds were recovered. (Table I, entry 9). As evidenced from Table I, optimal catalyst loading for the microwave-assisted condensation was found to be 2 mol %, affording **4a** in 88 % yield and therefore the same conditions were used for all subsequent reactions.

To see the scope and generality of this microwave-assisted protocol, CSA catalyzed condensation of 2-naphthol with a variety of aromatic aldehydes (**2b**–**k**) was investigated under MWI. In all the cases, the desired dibenzoxanthenes were obtained (Scheme 1) in good to high yields and the results are summarized in Table II. In the case of solid aromatic aldehydes, such as 4-cyanobenzaldehyde (**2h**), acetonitrile (0.25 ml $mmol^{-1}$) was used as solvent to make a uniform slurry, when the corresponding cyano dibenzoxanthene **4h** was isolated in 86 % yield. Solvent optimization of this reaction with other microwave-active solvents, *viz.* H_2O and DMF without changing catalyst loading was also investigated. To this end, the use of H_2O led to the formation of **4h**, albeit in poor yield (32 %), while no product was detected using DMF as solvent, when both starting compounds were recovered.



(54-98%)

a: Ar = Ph
b: Ar = 4-BrC_6H_4
c: Ar = 3-$OCH_3C_6H_4$
d: Ar = 2-$OCH_3C_6H_4$
e: Ar = 4-$OCH_3C_6H_4$
f: Ar = 4-OHC_6H_4
g: Ar = 4-$NHCOCH_3C_6H_4$
h: Ar = 4-CNC_6H_4
i: Ar = 4-$CO_2HC_6H_4$
j: Ar = 4-$NO_2C_6H_4$
k: Ar = 3-FC_6H_4

Scheme 1. Reaction scheme with structures of reactants **2b–k** and products **4b–k**.

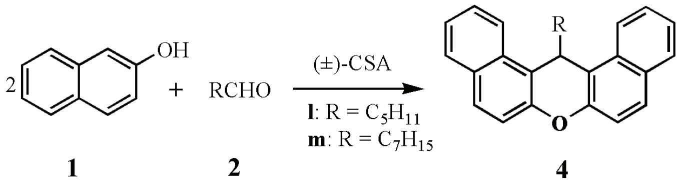
TABLE II. Microwave assisted, (±)-CSA catalyzed condensation of 2-naphthol (**1**) with aromatic aldehydes (ArCHO) to 14-aryl-14*H*-dibenzo[*a*,*j*]xanthene (**4b–k**); reaction conditions: 2-naphthol, aldehydes (0.55 equiv.) and (±)–CSA (0.02 equiv.) were irradiated in a microwave reactor at 400 W, 63–64 °C for 15–45 min

Entry	Aldehydes	Product	Time, min	Yield, %[a]	Melting point, °C	
					Found	Reported
1	**2b**	**4b**	30	82	293–294	295–296
2	**2c**	**4c**	15	98	177–178	179–180
3	**2d**	**4d**	45	54	258–259	258–260
4	**2e**	**4e**	15	88	207–208	205–206
5[b]	**2f**	**4f**	45	61	138–139	138–140
6[b]	**2g**	**4g**	45	86	153–154	–
7[b]	**2h**	**4h**	30	86	294–295	291–292
8[b]	**2i**	**4i**	30	85	>300	–
9[b]	**2j**	**4j**	30	84	>300	310–311
10	**2k**	**4k**	30	95	256–257	259

[a] Isolated yields; [b] acetonitrile (0.25mL $mmol^{-1}$) was used to make a uniform reaction mixture

The electronic effect of the substituents in the aromatic aldehydes was found to have a minimal effect, as evidenced by the high yield of the corresponding dibenzoxanthenes in almost all the cases (**4b–k**) (Table II, entries 1–10). However, the moderate yield of **4d** derived from 2-methoxybenzaldehyde (**2d**) could be explained by the large steric strain (resulting from the bulky methoxy group) involved during the cyclodehydration step to dibenzoxanthene (Table II, entry 3).

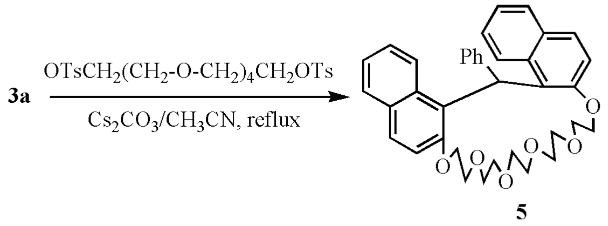
To explore the generality of the catalyst, CSA catalyzed condensation of 2-naphthol (**1**) with aliphatic aldehydes was also investigated. In contrast to benzaldehyde, the CSA catalyzed (10 mol %) condensation of **1** with hexanal (**2l**) at 25 °C without solvent (24 h) led to complete consumption of starting materials and 14-pentyldibenzoxanthene (**4l**) was obtained as the sole product in 73 % yield; no trace of 1,1-bis(2-hydroxynaphthyl)hexane was detected (Scheme 2). Similarly, the reaction with octanal (**2m**) led to the formation of 14-heptyldibenzoxanthene (**4m**) as the only product. However, at a higher temperature (80 °C), both the reactions (with hexanal and octanal) were completed within 20 min using a lower catalyst loading (5 mol %) to afford the corresponding dibenzoxanthenes in 93 and 86 % yields, respectively (Scheme 2). Moreover, the above reactions under MWI were completed in 15 min with a lower catalyst loading (2 mol %), as compared to the high temperature reactions, affording the corresponding dibenzoxanthenes (**4l** and **4m**) in 92 and 95 % yields, respectively (Scheme 2). Most catalysts[24–26,29] reported so far for the condensation of **1** with aliphatic aldehydes furnished the corresponding dibenzoxanthenes in longer reaction times and in moderate yields. Therefore, CSA displayed its catalytic efficiency in the faster condensation of **1** with both aliphatic and aromatic aldehydes to afford corresponding alkyl/aryl substituted dibenzoxanthenes in high yields. Incidentally, the catalytic potential of CSA in terms of reaction time and yield was found to be superior/similar to most of the hitherto reported catalysts.



4l:	**4m:**
73% (CSA (10 mol%), 25 °C, 24 h)	70% (CSA (10 mol%), 25 °C, 24 h)
93% (CSA (5 mol%), 80 °C, 20 min)	86% (CSA (5 mol%), 80 °C, 20 min)
92% (CSA (2 mol%), MWI, 15 min)	95% (CSA (2 mol%), MWI, 15 min)

Scheme 2. (±)-CSA-catalyzed condensation of 2-naphthol (**1**) with aliphatic aldehydes (RCHO) to 14-alkyl-14*H*-dibenzo[*a*,*j*]xanthene (**4l**,**m**).

Compound **3a**, obtained from the condensation of **1** and **2a** at ambient temperature, belongs to the important class of tethered bis(2-naphthol)s, which in principle could be exploited further for the synthesis of a variety of macrocycles, including crown ethers. To this end, reaction of **3a** with pentaethyleneglycol ditosylate and K_2CO_3 in acetonitrile yielded the corresponding crown ether **5** in good yield (Scheme 3).



Scheme 3. Synthesis of *bis*-napthalenocrown-6.

EXPERIMENTAL

Microwave irradiation (MWI) was performed with an Anton Paar microwave reactor (model SYNTHOS 3000). The melting points were determined using a Fisher–Johns melting point apparatus and are uncorrected. The IR spectra were scanned with a Jasco FT IR 4100 spectrophotometer. The ^{1}H- and ^{13}C-NMR spectra were recorded with a Bruker AC 200/300 MHz spectrometer. Spectra were referenced to the residual chloroform (δ 7.25 ppm, ^{1}H; 77.0 ppm, ^{13}C). The low-resolution mass spectra were recorded on a Varian 500 mass spectrometer (ESI/APCI) and a Shimadzu GC-MS 2010 mass spectrometer (EI 70 eV). The high-resolution mass spectra were recorded on a Q-TOF (YA-105) micromass spectrometer (ESI, Ar) and a Bruker maXis impact mass spectrometer (ESI). The microanalyses were realized using a Vario Micro elemental analyzer. (±)-Camphor-10-sulfonic acid (CSA) was purchased from Aldrich, USA, and was used as received. The spectral data of the known compounds were in accordance with those reported in the literature, while the unknown compounds were characterized by IR, NMR, HRMS and microanalytical data, which are given as Supplementary Material to this paper.

*Typical procedure for the solvent-free condensation of 2-naphthol (**1**) with benzaldehyde (**2a**) to the dibenzoxanthene **4a***

A mixture of 2-naphthol (1.44 g, 10.0 mmol), benzaldehyde (0.584 g, 5.50 mmol) and (±)-CSA (0.192 g, 15.0 mol. %) was heated at 80 °C without any solvent. After completion of the reaction (monitored by TLC), the formed pinkish solid was quenched with water, filtered, washed with water and air-dried. The crude solid was recrystallized with hexane–ethyl acetate, 9:1, to afford pure **4a** (1.60 g, 89 %).

*Typical procedure for the solvent-free condensation of 2-naphthol (**1**) with benzaldehyde (**2a**) to the dibenzoxanthene **4a** under microwave irradiation*

A mixture of 2-naphthol (1.44 g, 10.0 mmol), benzaldehyde (0.584 g, 5.50 mmol) and (±)-CSA (0.026 g, 2.0 mol. %) was placed in a microwave vessel and irradiated (400 W, 63––64 °C). After 15 min, TLC showed complete consumption of both the starting materials and the formation of **4a** as the only product. The thus formed solid was quenched with water, filtered, washed with water and air-dried. The crude solid was recrystallized with hexane––ethyl acetate, 9:1, to afford pure **4a** (1.59 g, 88 %).

*Typical procedure for the synthesis of bis-napthocrown-6 (**5**) from **3a***

A mixture of bis-(2-naphthol) **3a** (0.790 g, 2.10 mmol), pentaethyleneglycol ditosylate (1.26 g, 2.30 mmol, 1.1 equiv.) and Cs$_2$CO$_3$ (1.73 g, 5.30 mmol, 2.5 equiv.) in dry acetonitrile (30 mL) was refluxed for 8 h when TLC showed the absence of both **3a** and the ditosylate. The solvent was removed under *vacuo*, cooled, quenched with 1 M HCl and extracted with ethyl acetate. The organic layer was washed with water and brine, and dried (Na$_2$SO$_4$). Removal of solvent afforded a thick mass which was purified by silica gel column chromatography (using $CHCl_3$ as the eluant) to afford pure **5** (0.948 g, 78 %).

CONCLUSIONS

In conclusion, a simple, efficient and microwave-assisted (15–45 min) protocol for the CSA-catalyzed condensation of 2-naphthol with aromatic/aliphatic aldehydes to 14-aryl/alkyl-14*H*-dibenzo[*a*,*j*]xanthenes was developed. In contrast to the poor yields of 14-alkyl-dibenzo[*a*,*j*]xanthenes from aliphatic aldehydes with most of the reported catalysts, CSA was found to be a superior catalyst to yield the corresponding dibenzoxanthenes in high yields. Shorter reaction time, simple work-up procedure and easy isolation of the products in high yields are some of the salient features of this new protocol. The intermediate bis(2-naphthol) derived from the condensation of 2-naphthol and benzaldehyde at ambient temperature was used in the synthesis of a novel class of crown ethers.

SUPPLEMENTARY MATERIAL

The physical, analytic and spectral data for the prepared compounds as well as their ^{1}H- and ^{13}C-NMR spectra are available electronically from http://www.shd.org.rs/JSCS/, or from the corresponding author on request.

ИЗВОД

КОНДЕНЗАЦИЈА 2-НАФТОЛА СА АРОМАТИЧНИМ/АЛИФАТИЧНИМ АЛДЕХИДИМА У СИНТЕЗИ 14-АРИЛ/АЛКИЛ-14*H*-ДИБЕНЗО[*a*,*j*]КСАНТЕНА КАТАЛИЗОВАНА КАМФОР-10-СУЛФОНСКОМ КИСЕЛИНОМ

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(±)-Камфор-10-сулфонска киселина (CSA) катализује кондензацију 2-нафтола са ароматичним/алифатичним алдехидима, на 80 °C у синтези 14-арил/алкил-14*H*-дибензо[*a*,*j*]ксантена као јединих производа, у високом приносу. У реакција кондензације са бензалдехидом на 25 °C добија се смеша интермедијера бис(2-хидроксинафтил)-фенилметана и производа 14-фенил-дибензоксантена, док кондензацијом са алифатичним алдехидима на 25 °C настају 14-алкил-дибензоксантени као једини производи. Даље, кондензацијом 2-нафтола са ароматичним/алифатичним алдехидима у присуству малих количина катализатора (2 mol %), под условима озрачивања микроталасима, реакција се брже одвија и настају одговарајући 14-арил/алкилдибензоксантени као једини производи.

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