# Silica Gel-Supported Polyphosphoric Acid (PPA/SiO<sub>2</sub>): An Efficient and Reusable Heterogeneous Catalyst for Facile Synthesis of 14-Aryl-14*H*-dibenzo[*a*,*j*]xanthenes under Solvent-free Conditions

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A simple, efficient and green method for the synthesis of 14-aryl-14*H*-dibenzo[ $a_j$ ]xanthenes by a one-pot condensation reaction of  $\beta$ -naphthol and aryl aldehydes using silica gel-supported polyphosphoric acid (PPA/SiO<sub>2</sub>), an effective and reusable catalyst, under solvent-free conditions is described. The present methodology offers several advantages, such as a simple procedure with an easy work-up, short reaction times, high yields, and the absence of any volatile and hazardous organic solvents.

**Keywords** 14-Aryl-14*H*-dibenzo[a,j]xanthenes, PPA/SiO<sub>2</sub>, heterogeneous catalysis, green chemistry, solvent-free conditions

#### Introduction

The principles of green chemistry have been introduced to eliminate or at least to reduce the use of hazardous materials, such as  $H_2SO_4$  or  $H_3PO_4$  in chemical processes. Cleaner technologies could become possible by making use of the environmental friendly materials, such as the use of solid acids. These catalysts have many advantages over liquid acid catalysts.<sup>1-3</sup> They are not corrosive but environmentally benign, presenting fewer disposal problems. Thus the development and use of solid green catalysts are very important in organic syntheses.

Recently, solid-supported reagents, such as the silica gel-supported acids, have gained considerable interests in organic synthesis because of their unique properties, such as high efficiency due to more surface area, more stability and reusability, low toxicity, greater selectivity and ease of handling.<sup>4-6</sup> Although the catalytic applications of silica supported reagents for organic synthesis have been established, to the best of our knowledge, there is no published report on the use of PPA-SiO<sub>2</sub> in the synthesis of 14-aryl-14*H*-dibenzo[*a,j*]xanthenes.

Xanthenes and dibenzoxanthenes' derivatives are important classes of organic compounds with a large number of naturally occurring, as well as synthetic derivatives, and occupy a prominent position in medicinal chemistry.<sup>7</sup> A number of these compounds have been considered as dyes and fluorescent visualization materials for biomolecules and laser technologies.<sup>8,9</sup> Furthermore, these compounds have been investigated for their agricultural bactericide activity,<sup>10-12</sup> anti-inflammatory effect,<sup>13</sup> and antiviral activity.<sup>14</sup> There are several methods reported for the synthesis of xanthenes and dibenzoxanthenes, such as palladium catalyzed cyclization of polycyclic aryltriflate esters,15 and intramolecular trapping of benzynes by phenols.<sup>16</sup> Furthermore, 14-aryl-14H-dibenzo[a,j]xanthenes and their analogues were prepared by reaction of  $\beta$ -naphthol with 2-naphthol-1-methanol,<sup>17</sup> formamide,<sup>18</sup> carbon monoxide<sup>19</sup> as well as by condensation of  $\beta$ -naphtol with aldehydes using various acid catalysts.<sup>20-24</sup> Many of these methodologies, however, suffer from disadvantages, such as unsatisfactory yields, expensive catalysts, long reaction times, toxic organic solvents, and harsh reaction conditions. Therefore, the development of simple, efficient, high-yielding, and environmentaly friendly methods using new catalysts for the synthesis of these compounds is still necessary.

As part of our current studies on the development of new routes for the synthesis of organic compounds using reusable catalysts,<sup>25-33</sup> herein we wish to report a green and rapid method for the synthesis of 14-aryl-14*H*-dibenzo[a,j]xanthenes by condensation of  $\beta$ -naphthol with aryl aldehydes using PPA/SiO<sub>2</sub>, as a reusable catalyst, under solvent-free conditions (Scheme 1).

#### **Experimental**

All chemicals were available commercially and used without additional purification. The catalyst was synthe-

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sized according to the literature.<sup>34</sup> The amount of  $H^+$  in the PPA/SiO<sub>2</sub> was determined by acid-base titration. Melting points were recorded on an electrothermal type 9100 melting point apparatus. The IR spectra were obtained using a 4300 Shimadzu spectrophotometer as KBr disks. The <sup>1</sup>H NMR (500 MHz) spectra were recorded with a Bruker DRX500 spectrometer.

# General procedure for the synthesis of 14-aryl-14*H*-dibenzo[a,j]xanthenes 3a — 3j using PPA/SiO<sub>2</sub> as catalyst

A mixture of  $\beta$ -naphthol 1 (2 mmol), aromatic aldehyde **2a**—**2j** (1 mmol), and PPA/SiO<sub>2</sub> (0.030 g, 0.015 mmol of H<sup>+</sup>) was heated in the oil bath at 120 °C for 30—40 min. During the procedure, the reaction was monitored by TLC. Upon completion, the reaction mixture was cooled to room temperature and hot ethanol was added. The catalyst was dissolved in hot ethanol and filtered off. The product was then collected from the filtrate after cooling to room temperature and recrystallized from ethanol to give compounds **3a**—**3j** in high yields.

#### Recycling and reusing of the catalyst

Due to the fact that the catalyst was dissolved in hot ethanol, it could therefore be recycled by a simple filtration. The separated catalyst was washed with cold ethanol, dried at 100  $^{\circ}$ C under vacuum for 2 h and was reused in another reaction. The catalyst could be reused at least three times without significant loss of activity.

#### <sup>1</sup>H NMR data for compounds 3a—3j

**3a** (Ar=Ph): <sup>1</sup>H NMR (CDCl<sub>3</sub>)  $\delta$ : 6.53 (s, 1H, CH), 7.04 (t, *J*=7.4 Hz, 1H, arom-H), 7.19 (t, *J*=7.6 Hz, 2H, arom-H), 7.45 (t, *J*=7.0 Hz, 2H, arom-H), 7.54 (d, *J*= 8.9 Hz, 2H, arom-H), 7.58 (d, *J*=7.3 Hz, 2H, arom-H), 7.62 (td, *J*=7.0, 1.2 Hz, 2H, arom-H), 7.83 (d, *J*=8.9 Hz, 2H, arom-H), 7.87 (d, *J*=8.0 Hz, 2H, arom-H), 8.44 (d, *J*=8.5 Hz, 2H, arom-H).

**3b** (Ar=4-BrC<sub>6</sub>H<sub>4</sub>): <sup>1</sup>H NMR (CDCl<sub>3</sub>)  $\delta$ : 6.39 (s, 1H, CH), 7.19 (d, J=8.0 Hz, 2H, arom-H), 7.32—7.40 (m, 4H, arom-H), 7.42 (d, J=8.8 Hz, 2H, arom-H), 7.53 (t, J=7.5 Hz, 2H, arom-H), 7.74 (d, J=8.8 Hz, 2H, arom-H), 7.78 (d, J=7.9 Hz, 2H, arom-H), 8.26 (d, J= 8.3 Hz, 2H, arom-H).

**3c** (Ar=4-ClC<sub>6</sub>H<sub>4</sub>): <sup>1</sup>H NMR (CDCl<sub>3</sub>)  $\delta$ : 6.51 (s, 1H, CH), 7.14 (d, J=8.3 Hz, 2H, arom-H), 7.22—7.55 (m, 6H, arom-H), 7.62 (t, J=7.6 Hz, 2H, arom-H), 7.84 (d, J=8.9 Hz, 2H, arom-H), 7.87 (d, J=8.0 Hz, 2H, arom-H), 8.35 (d, J=8.4 Hz, 2H, arom-H).

**3d** (Ar=4-FC<sub>6</sub>H<sub>4</sub>): <sup>1</sup>H NMR (CDCl<sub>3</sub>)  $\delta$ : 6.53 (s, 1H,

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CH), 6.85 (t, J=8.7 Hz, 2H, arom-H), 7.46 (t, J=7.8 Hz, 2H, arom-H), 7.48—7.55 (m, 4H, arom-H), 7.62 (td, J=7.7, 1.2 Hz, 2H, arom-H), 7.84 (d, J=8.9 Hz, 2H, arom-H), 7.87 (d, J=8.1 Hz, 2H, arom-H), 8.37 (d, J=8.5 Hz, 2H, arom-H).

**3e** (Ar=3-HOC<sub>6</sub>H<sub>4</sub>): <sup>1</sup>H NMR (CDCl<sub>3</sub>)  $\delta$ : 4.55 (s, 1H), 6.42—6.60 (m, 2H), 6.84 (s, 1H), 7.06 (t, *J*=8.2 Hz, 1H), 7.21 (d, *J*=7.8 Hz, 1H), 7.34—7.45 (m, 4H), 7.52—7.60 (m, 2H), 7.73—7.81 (m, 4H), 8.36 (d, *J*= 8.5 Hz, 2H).

**3f** (Ar=4-HOC<sub>6</sub>H<sub>4</sub>): <sup>1</sup>H NMR (CDCl<sub>3</sub>)  $\delta$ : 5.21 (sbr. 1H), 6.44 (s, 1H), 6.56 (d, *J*=8.2 Hz, 2H), 7.37 (t, *J*= 9.8 Hz, 2H), 7.43 (d, *J*=7.6 Hz, 2H), 7.50 (d, *J*=8.8 Hz, 2H), 7.61 (t, *J*=7.7 Hz, 2H), 7.75 (d, *J*=6.4 Hz, 2H), 7.89 (d, *J*=8.5 Hz, 2H), 8.33 (d, *J*=8.2 Hz, 2H).

**3g** (Ar=4-MeOC<sub>6</sub>H<sub>4</sub>): <sup>1</sup>H NMR (CDCl<sub>3</sub>)  $\delta$ : 3.65 (s, 3H, OCH<sub>3</sub>), 6.49 (s, 1H, CH), 6.70 (d, *J*=8.8 Hz, 2H, arom-H), 7.40—7.47 (m, 4H, arom-H), 7.51 (d, *J*=8.9 Hz, 2H, arom-H), 7.61 (td, *J*=8.3, 1.1 Hz, 2H, arom-H), 7.81 (d, *J*=8.9 Hz, 2H, arom-H), 7.86 (d, *J*=8.0 Hz, 2H, arom-H), 8.42 (d, *J*=8.5 Hz, 2H, arom-H).

**3h** (Ar=4-MeC<sub>6</sub>H<sub>4</sub>): <sup>1</sup>H NMR (DMSO- $d_6$ )  $\delta$ : 2.04 (s, 3H, CH<sub>3</sub>), 6.66 (s, 1H, CH), 6.92 (d, J=8.0 Hz, 2H, arom-H), 7.45 (t, J=7.4 Hz, 2H, arom-H), 7.48 (d, J=8.1 Hz, 2H, arom-H), 7.54 (d, J=8.8 Hz, 2H, arom-H), 7.61 (t, J=7.2 Hz, 2H, arom-H), 7.91 (d, J=8.9 Hz, 2H, arom-H), 7.92 (d, J=7.7 Hz, 2H, arom-H), 8.65 (d, J=8.5 Hz, 2H, arom-H).

**3i** (Ar=3-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>): <sup>1</sup>H NMR (CDCl<sub>3</sub>)  $\delta$ : 6.63 (s, 1H, CH), 7.32 (t, J=8.0 Hz, 1H, arom-H), 7.47 (t, J= 7.4 Hz, 2H, arom-H), 7.55 (d, J=8.9 Hz, 2H, arom-H), 7.65 (t, J=7.4 Hz, 2H, arom-H), 7.80—7.92 (m, 6H, arom-H), 7.34 (d, J=8.5 Hz, 2H, arom-H), 8.45 (s, 1H, arom-H).

**3j** (År=4-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>): <sup>1</sup>H NMR (CDCl<sub>3</sub>)  $\delta$ : 6.63 (s, 1H, CH), 7.47 (t, *J*=7.5 Hz, 2H, arom-H), 7.54 (d, *J*= 8.9 Hz, 2H, arom-H), 7.63 (t, *J*=7.6 Hz, 2H, arom-H), 7.70 (d, *J*=8.6 Hz, 2H, arom-H), 7.84—7.91 (m, 4H, arom-H), 8.02 (d, *J*=8.6 Hz, 2H, arom-H), 8.31 (d, *J*= 8.4 Hz, 2H, arom-H).

#### **Results and discussion**

PPA-SiO<sub>2</sub> was prepared according to the literature procedure.<sup>34</sup> Initially, the synthesis of compound **3a** was selected as a model reaction to optimize the reaction conditions. The reaction was carried out by heating a mixture of  $\beta$ -naphthol (2 mmol) and benzaldehyde (1 mmol) under various conditions.

The efficiency of the reaction is affected mainly by the amount of PPA/SiO<sub>2</sub> (Table 1). No product was obtained in the absence of the catalyst (Entry 1) indicating that the catalyst is necessary for the reaction. Increasing the amount of the catalyst increased the yields of the product **3a**. The optimal amount of PPA/SiO<sub>2</sub> was 0.03 g (Entry 4); increasing the amount of the catalyst beyond this value did not increase the yield noticeably (Entries 5, 6). 
 Table 1
 Effect of PPA/SiO2 amount on the model reaction<sup>a</sup>



<sup>*a*</sup> 2 mmol  $\beta$ -naphthol and 1 mmol benzaldehyde at 120 °C; <sup>*b*</sup> the yields were calculated based on benzaldehyde and refer to the pure isolated product.

Furthermore, the reaction was carried out in different solvents and under solvent-free conditions. As shown in Table 2, the yields of the reaction under solvent-free conditions were greater and the reaction times were generally shorter than the conventional methods.

The same model reaction in presence of 0.03 g of the catalyst was carried out at different temperatures in solvent-free conditions to assess the effect of temperatures on the reaction yield. It was observed that yield is a function of temperature since the yield was increased as the reaction temperature was raised (Table 3). At 120  $^{\circ}$ C, the product **3a** was obtained in excellent yield. Therefore, all subsequent reactions were carried out at 120  $^{\circ}$ C in presence of 0.03 g of PPA/SiO<sub>2</sub> under solvent-free conditions.

In order to evaluate the generality of this model reaction, we prepared a range of 14-aryl-14*H*-dibenzo-[a,j]xanthenes under optimized reaction conditions. In all cases, aromatic aldehydes with substituents carrying either electron-donating or electron-withdrawing groups

**Table 2** Synthesis of 14-phenyl-14*H*-dibenzo[a,j]xanthene **3a** in the presence of PPA/SiO<sub>2</sub> (0.03 g) in different solvents

Entry	Solvent	Temperature/°C	Time/h	Yield <sup>a</sup> /%
1	MeOH	64	5	Trace
2	EtOH	78	5	65
3	CH <sub>3</sub> CN	81	5	82
4	$H_2O$	100	5	None
6	Solvent-free	120	0.5	90

<sup>*a*</sup> The yields were calculated based on benzaldehyde and refer to the pure isolated product.

**Table 3** Synthesis of 14-phenyl-14*H*-dibenzo[a,j]xanthene **3a** in the presence of PPA/SiO<sub>2</sub> (0.03 g) at different temperatures in solvent-free conditions

Entry	Temperature/°C	Time/min	Yield <sup>a</sup> /%
1	80	30	72
2	100	30	81
3	110	30	86
4	120	30	90
5	140	30	90

<sup>*a*</sup> The yields were calculated based on benzaldehyde and refer to the pure isolated product.

reacted successfully and gave the expected products in excellent yields and short reaction times. The type of aldehyde had no significant effect on the reaction. The results are shown in Table 4.

Reusability of the catalyst was also investigated. For this purpose, the same model reaction was again studied under optimized conditions. After the completion of the reaction, the reaction mixture was cooled to room temperature, and hot ethanol was added. The catalyst was separated by simple filtration, dried at 100  $^{\circ}$ C under vacuum for 2 h, and reused for a similar reaction. As shown in Figure 1, the catalyst could be reused at least three times without significant loss of activity.

Entry	Ar	Product <sup>b</sup>	Time/min	Yield <sup>c</sup> /%	m.p. (Lit. m.p.)/°C
1		3a	30	90	184—186 (182—183) <sup>21</sup>
2	Br	Br Green Strategy Br Br Br Br Br Br Br Br Br Br Br Br Br	35	86	296—298 (295—296) <sup>21</sup>

	Table 4	Preparation of 14-a	ryl-14H-dibenzo[a	a,j]xanthenes using	PPA/SiO <sub>2</sub> (0.03	g) as catalyst <sup><math>a</math></sup>
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					Continued
Entry	Ar	Product <sup>b</sup>	Time/min	Yield <sup>c</sup> /%	m.p. (Lit. m.p.)/°C
3	CI-	CI CI CI CI CI CI CI CI CI CI CI CI CI C	40	89	290—291 (287—288) <sup>21</sup>
4	F	F G G G G G G G G G G G G G G G G G G G	35	90	238—240 (239—240) <sup>21</sup>
5	HO	OH O Je	30	88	243—245 (242—243) <sup>21</sup>
6	но-	OH U OH 3f	35	90	138—140 (139—140) <sup>21</sup>
7	MeO-	OMe J J J J J J J	40	89	202—205 (202—203) <sup>21</sup>
8	Me-	Me	40	88	228—230 (227—228) <sup>21</sup>
9	O <sub>2</sub> N	Sin NO <sub>2</sub>	40	86	210—212 (210—211) <sup>21</sup>

<sup>&</sup>lt;sup>*a*</sup> 2 mmol  $\beta$ -naphthol and 1 mmol aryl aldehyde at 120 °C under solvent-free conditions. <sup>*b*</sup> All the products were characterized by <sup>1</sup>H NMR and IR spectral data and comparison of their melting points with those of authentic samples. <sup>*c*</sup> The yields were calculated based on aryl aldehyde and refer to the pure isolated products.



Figure 1 Reusability of PPA/SiO<sub>2</sub> for model reaction

Although we did not investigate the reaction mechanism, to show the catalyst's role, a plausible mechanism for the present reaction was proposed as depicted in Scheme 2. The addition of nucleophiles to the aldehydes is promoted by the protonation of the carbonyl group using a Brønsted acid, enhancing the electrophilicity of this moiety. Therefore, it is proposed that PPA/SiO<sub>2</sub> = HA facilitates the formation of intermediate I such that after dehydration, intermediate II is produced. Nucleophilic attack of another molecule of  $\beta$ -naphthol at the

Scheme 2

 $\beta$ -carbon of the  $\alpha$ , $\beta$ -unsaturated carbonyl group in activated intermediate II yields the intermediate III, which finally converts to the product **3** by a cyclocondensation reaction. Under these conditions, attempts to isolate the intermediates I, II and III failed after careful monitoring of reactions.

#### Conclusion

In conclusion, we have reported a simple new catalytic method for the synthesis of 14-aryl-14*H*-dibenzo-[a,j]xanthenes by one-pot condensation reaction of  $\beta$ -naphthol and aryl aldehydes using PPA/SiO<sub>2</sub> as an efficient, reusable, and green heterogeneous catalyst under solvent-free conditions. The catalyst can be recycled after a simple work-up, and reused at least three times without substantial reduction in its catalytic activity. High yields, short reaction times, easy work-up and absence of any volatile and hazardous organic solvents are just a few of the advantages in this procedure.

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