

Silica Supported Perchloric Acid: An Efficient Catalyst for the Synthesis of 14-Aryl-14*H*-dibenzo[*a,i*]xanthene-8,13-diones

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Este trabalho descreve a obtenção de 14-aryl-14*H*-dibenzo[*a,i*]xanteno-8,13-dionas, com excelentes rendimentos e em um curto tempo de reação, a partir da reação de condensação do β -naftol com aldeídos aromáticos e 2-hidroxinaftaleno-1,4-dionas na presença de sílica suportada em ácido perclórico em um meio livre de solvente. A reação é muito simples e o catalisador pode ser separado facilmente da mistura reacional e, ainda, ser reutilizado por muitas vezes em reações subsequentes.

The condensation of β -naphthol with aromatic aldehydes and 2-hydroxynaphthalene-1,4-dione in presence of silica supported perchloric acid under solvent-free media to afford the corresponding 14-aryl-14*H*-dibenzo[*a,i*]xanthene-8,13-diones in excellent yields and short reaction times is described. The reaction work-up is very simple and the catalyst can be easily separated from the reaction mixture and reused several times in subsequent reactions.

Keywords: dibenzo[*a,i*]xanthenes, β -naphthol, 2-hydroxynaphthalene-1,4-dione, silica supported perchloric acid, solvent-free

Introduction

Xanthenes and benzoxanthenes have been reported to possess diverse biological and therapeutic properties such as antibacterial,¹ antiviral,² and anti-inflammatory activities,³ as well as photodynamic therapy⁴ and for antagonism of the paralyzing action of zoxazolamine.⁵ The other useful applications of this heterocycles are as dyes,⁶ fluorescent materials for visualization of biomolecules,⁷ and in laser technologies.⁸ Many procedures have been developed for the synthesis of xanthenes and benzoxanthenes, which include trapping of benzynes by phenols,⁹ cyclocondensation between 2-hydroxyaromatic aldehydes and 2-tetralone,¹⁰ cyclodehydrations,¹¹ and intramolecular phenyl carbonyl reaction of aldehydes with 5,5-dimethylcyclohexane-1,3-dione¹² or β -naphthol.¹³ Furthermore, the synthesis of benzoxanthenes and their related products include the reaction of β -naphthol with formamide,¹⁴ carbon monoxide,¹⁵ 2-naphthol-1-methanol,¹⁶ aldehydes and cyclic 1,3-dicarbonyl compounds.¹⁷

In recent years, the use of heterogeneous catalysts has received considerable interest in various disciplines including organic synthesis. They are advantageous over

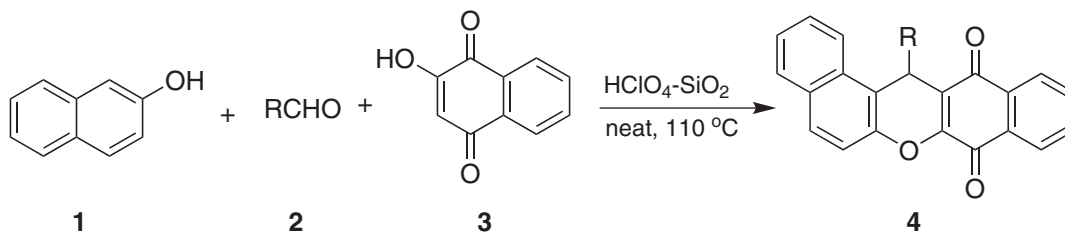
their homogeneous counterparts due to the prime advantage that in most of the cases the catalyst can be recovered easily and reused. Silica supported perchloric acid ($\text{HClO}_4\text{-SiO}_2$) has been used as an efficient heterogeneous catalyst for many organic transformations because of its low cost, ease of preparation, catalyst recycling, and ease of handling.¹⁸ We now report a simple and efficient route to synthesis of 14-aryl-14*H*-dibenzo[*a,i*]xanthene-8,13-diones using $\text{HClO}_4\text{-SiO}_2$ as an efficient catalyst under solvent-free conditions (Scheme 1).

Results and Discussion

Initially, to optimize the amount of catalyst and the reaction temperature, the reaction of β -naphthol, benzaldehyde and 2-hydroxy-1,4-naphthoquinone was studied under solvent-free conditions in the presence of $\text{HClO}_4\text{-SiO}_2$ at different temperatures. The results were summarized in Table 1, and showed that the reaction using 5 mol % $\text{HClO}_4\text{-SiO}_2$ at 110 °C proceeded in highest yield.

Based on the optimized reaction conditions, several syntheses of 14-aryl-14*H*-dibenzo[*a,i*]xanthene-8,13-diones from the condensation of β -naphthol, 2-hydroxynaphthalene-1,4-dione, and a wide range of aromatic aldehydes utilizing $\text{HClO}_4\text{-SiO}_2$ under solvent-free

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conditions at 110 °C were examined. All reactions were complete within 1-2 h, as indicated in Table 2; in all cases the reactions afforded the desired products in excellent yields. When this reaction was carried out with aliphatic aldehyde such as butanal or pentanal, TLC and ¹H NMR spectra of the reaction mixture showed a combination of starting materials and numerous products, the yield of the expected product was very poor.

Table 1. Amounts of catalyst and temperature optimization for the synthesis of 14-phenyl-14*H*-dibenzo[*a,i*]xantheno-8,13-dione^a

Entry	HClO ₄ -SiO ₂ / mol %	Temp. / °C	time / h	Yield / % ^b
1	5	50	3	49
2	5	80	2	64
3	5	90	1.5	72
4	5	100	1	82
5	0	110	5	0
6	1	110	2	52
7	2	110	2	61
8	3	110	1.5	74
9	4	110	1	82
10	5	110	1	92
11	6	110	1	92
12	7	110	1	91
13	5	120	1	90
14	5	130	0.5	91

^aReaction conditions: β-naphthol (1 mmol); benzaldehyde (1 mmol); 2-hydroxynaphthalene-1,4-dione (1 mmol); neat. ^bIsolated yield.

The reusability of the catalyst was checked by separating HClO₄-SiO₂ from the reaction mixture by simple filtration, washing with CHCl₃, and drying in a vacuum oven at 60 °C for 10 h prior to reuse in subsequent reactions. The recovered catalyst can be reused at least three additional times in subsequent reactions without significant loss in product yield (Table 3).

Although the detailed mechanism of the above reaction remains to be fully clarified, the formation of 14-aryl-14*H*-dibenzo[*a,i*]xantheno-8,13-diones could be explained by a reaction sequence presented in Scheme 2. We proposed

Table 2. Preparation of 14-aryl-14*H*-dibenzo[*a,i*]xantheno-8,13-diones catalyzed by HClO₄-SiO₂^a

Entry	R	time / h	Product	Yield / % ^b
1	C ₆ H ₅	1	4a	92
2	4-Cl-C ₆ H ₄	1	4b	94
3	4-MeO-C ₆ H ₄	1	4c	89
4	4-Me-C ₆ H ₄	1	4d	90
5	4-NO ₂ -C ₆ H ₄	1.5	4e	89
6	3-NO ₂ -C ₆ H ₄	2	4f	88
7	2,4-Cl ₂ -C ₆ H ₃	1.5	4g	87
8	3,4-Cl ₂ -C ₆ H ₃	1.5	4h	92
9	2-Cl-C ₆ H ₄	2	4i	88

^aReaction conditions: β-naphthol (1 mmol); aldehyde (1 mmol); 2-hydroxynaphthalene-1,4-dione (1 mmol); HClO₄-SiO₂ (0.05 mmol); 110 °C; neat. ^bIsolated yield.

Table 3. Effect of reusability of HClO₄-SiO₂ catalyst on 14-phenyl-14*H*-dibenzo[*a,i*]xantheno-8,13-dione synthesis^a

Run	Cycle	Yield / % ^b
1	0	92
2	1	91
3	2	89
4	3	88

^aReaction conditions: β-naphthol (1 mmol); benzaldehyde (1 mmol); 2-hydroxynaphthalene-1,4-dione (1 mmol); HClO₄-SiO₂ (0.05 mmol); 110 °C; neat. ^bIsolated yield.

that the reaction proceeded *via* a reaction sequence of condensation, addition, cyclization and dehydration. First, the condensation of aldehyde (2) and β-naphthol (1) gave the intermediate *ortho*-quinone methide (5). The addition of 2-hydroxynaphthalene-1,4-dione (3) to (5) leading to the formation of (6), which on intermolecular cyclization gave rise to (7). In the last step, the intermediate product (7) undergoes dehydration to afford the desired product (4). In β-naphthol the electron density at the benzylic C-1 position (which is in conjugation with the aromatic ring) is higher than that at the C-3 position. Thus the regioselective formation of the *ortho*-quinone methide from this compound involving the C-1 and C-2 positions is favored. In simple phenolic compounds and α-naphthol (which are weaker nucleophiles

compared to β -naphthol) the electron density at the *ortho* position of the hydroxyl group is not sufficient for the reaction of these compounds with the aldehydes leading to the formation of the corresponding *ortho*-quinone methides.

Conclusions

In conclusion, we have developed a simple and efficient protocol for the synthesis of 14-aryl-14*H*-dibenzo[*a,i*]xanthene-8,13-diones using $\text{HClO}_4\text{-SiO}_2$ under solvent-free conditions. Short reaction times, simple work-up in isolation of the products in high yields with high purity, mild reaction conditions and recyclability of supported catalyst are features of this procedure.

Experimental

NMR spectra were determined on Bruker AV-400 spectrometer at room temperature using TMS as internal standard. Chemical shifts (δ) are given in ppm and coupling constants (*J*) in Hz. Mass spectra were recorded on a Finnigan LCQ Advantage mass spectrometer. Elemental analysis were performed by a Vario-III elemental analyzer. Melting points were determined on a XT-4 binocular microscope and were uncorrected. Commercially available reagents were used throughout without further purification unless otherwise stated.

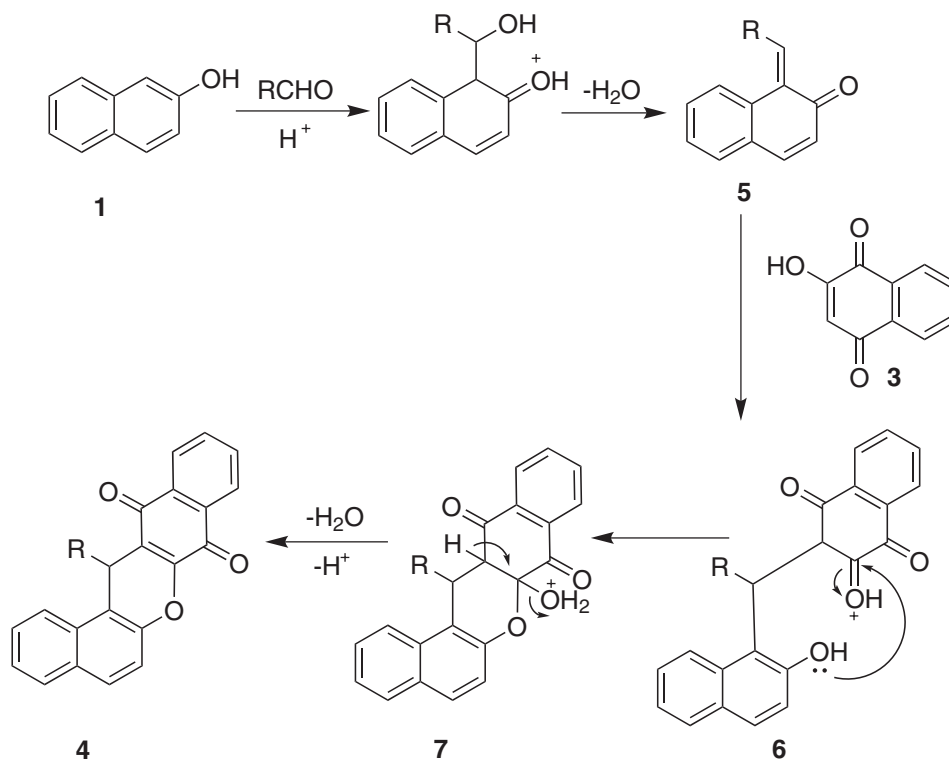
Preparation of $\text{HClO}_4\text{-SiO}_2$ catalyst

HClO_4 (1.8 g, 12.5 mmol, as 70% aqueous solution) was added to a suspension of SiO_2 (230-400 mesh, 23.7 g) in Et_2O (70.0 mL). The mixture was concentrated and the residue was heated at 100 °C for 72 h under vacuum to furnish $\text{HClO}_4\text{-SiO}_2$ (0.5 mmol g^{-1}) as a free flowing powder (50 mg = 0.025 mmol of HClO_4).

General procedure for the preparation of 14-aryl-14*H*-dibenzo[*a,i*]xanthene-8,13-diones

A mixture of β -naphthol (1 mmol), aldehyde (1 mmol), 2-hydroxynaphthalene-1,4-dione (1 mmol) and $\text{HClO}_4\text{-SiO}_2$ (100 mg, 0.05 mmol) was heated at 110 °C for an appropriate time and monitored by TLC until the final conversion. After cooling, the reaction mixture was washed with CHCl_3 and filtered to recover the catalyst. Solvent was evaporated and the crude product purified by silica gel column chromatography using CHCl_3 as eluent to afford the pure product.

14-Phenyl-14*H*-dibenzo[*a,i*]xanthene-8,13-dione (4a): Yellow powder, mp 319-320 °C; $^1\text{H NMR}$ (CDCl_3 , 400 MHz) δ 8.17 (d, 1H, *J* 7.6 Hz), 8.12 (d, 1H, *J* 7.6 Hz), 7.99 (d, 1H, *J* 8.4 Hz), 7.91-7.77 (m, 3H), 7.61-7.41 (m, 6H), 7.20 (t, 2H, *J* 8.0 Hz), 7.12-7.09 (m, 1H), 5.95 (s, 1H);



Scheme 2.

^{13}C NMR (CDCl_3 , 100 MHz) δ 178.3, 157.2, 147.3, 143.1, 135.1, 131.9, 131.2, 131.0, 130.9, 130.0, 129.5, 129.4, 128.6, 127.5, 126.8, 125.5, 124.5, 123.8, 116.9, 116.8, 116.6, 35.2; MS (ESI): m/z 389 $[\text{M}+\text{H}]^+$; Anal. Calc. for $\text{C}_{27}\text{H}_{16}\text{O}_3$: C, 83.49; H, 4.15. Found: C, 83.25; H, 4.12%.

14-(4-Chlorophenyl)-14H-dibenzo[a,i]xanthene-8,13-dione (4b): Yellow powder, mp 305-306 °C; ^1H NMR (CDCl_3 , 400 MHz) δ 8.16 (d, 1H, J 7.6 Hz), 8.13 (d, 1H, J 7.6 Hz), 7.92-7.77 (m, 4H), 7.62-7.44 (m, 4H), 7.34 (d, 2H, J 8.4 Hz), 7.15 (d, 2H, J 8.4 Hz), 5.90 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 178.2, 157.3, 147.2, 141.6, 135.2, 132.7, 131.9, 131.4, 130.8, 130.7, 130.0, 120.0, 129.8, 129.5, 128.7, 127.6, 125.7, 124.6, 123.6, 116.8, 116.3, 116.0, 34.6; MS (ESI): m/z 423 $[\text{M}+\text{H}]^+$; Anal. Calc. for $\text{C}_{27}\text{H}_{15}\text{ClO}_3$: C, 76.69; H, 3.58. Found: C, 76.48; H, 3.62%.

14-(4-Methoxyphenyl)-14H-dibenzo[a,i]xanthene-8,13-dione (4c): Yellow powder, mp 279-280 °C; ^1H NMR (CDCl_3 , 400 MHz) δ 8.16 (d, 1H, J 8.0 Hz), 8.12 (d, 1H, J 7.6 Hz), 7.98 (d, 1H, J 8.4 Hz), 7.89-7.76 (m, 4H), 7.60-7.43 (m, 5H), 7.31 (d, 2H, J 8.4 Hz), 5.90 (s, 1H), 3.69 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 178.4, 158.3, 157.0, 147.2, 135.5, 135.1, 131.9, 131.2, 131.0, 130.0, 129.6, 129.4, 128.5, 127.4, 125.5, 124.5, 123.8, 117.1, 116.8, 113.9, 55.1, 34.3; MS (ESI): m/z 419 $[\text{M}+\text{H}]^+$; Anal. Calc. for $\text{C}_{28}\text{H}_{18}\text{O}_4$: C, 80.37; H, 4.34. Found: C, 80.50; H, 4.27%.

14-(4-Methylphenyl)-14H-dibenzo[a,i]xanthene-8,13-dione (4d): Yellow powder; mp 255-256 °C; ^1H NMR (CDCl_3 , 400 MHz) δ 8.16 (d, 1H, J 8.0 Hz), 8.11 (d, 1H, J 7.6 Hz), 7.99 (d, 1H, J 8.0 Hz), 7.89-7.76 (m, 3H), 7.60-7.42 (m, 4H), 7.29 (d, 2H, J 8.0 Hz), 7.00 (d, 2H, J 7.6 Hz), 5.90 (s, 1H), 2.21 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 178.3, 178.2, 157.1, 147.2, 140.3, 136.5, 135.1, 131.9, 131.2, 131.0, 130.9, 120.0, 129.4, 129.3, 129.2, 128.5, 128.4, 127.4, 125.5, 124.5, 123.8, 117.0, 116.8, 116.7, 34.7, 21.0; MS (ESI): m/z 403 $[\text{M}+\text{H}]^+$; Anal. Calc. for $\text{C}_{28}\text{H}_{18}\text{O}_3$: C, 83.57; H, 4.51. Found: C, 83.49; H, 4.63%.

14-(4-Nitrophenyl)-14H-dibenzo[a,i]xanthene-8,13-dione (4e): Yellow powder, mp 332-333 °C; ^1H NMR (CDCl_3 , 400 MHz) δ 8.20 (d, 1H, J 7.6 Hz), 8.15 (d, 1H, J 7.6 Hz), 8.06 (d, 2H, J 8.8 Hz), 7.97-7.81 (m, 4H), 7.66-7.49 (m, 6H), 6.06 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 178.2, 178.0, 157.8, 150.0, 147.3, 146.7, 135.3, 132.0, 131.7, 130.7, 130.4, 130.3, 130.0, 129.6, 128.8, 127.9, 125.9, 124.8, 123.9, 123.3, 116.8, 115.4, 115.0, 35.3; MS (ESI): m/z 434 $[\text{M}+\text{H}]^+$; Anal. Calc. for $\text{C}_{27}\text{H}_{15}\text{NO}_5$: C, 74.82; H, 3.49; N, 3.23. Found: C, 74.91; H, 3.38; N, 3.29%.

14-(3-Nitrophenyl)-14H-dibenzo[a,i]xanthene-8,13-dione (4f): Yellow powder, mp 304-305 °C; ^1H NMR (CDCl_3 , 400 MHz) δ 8.22 (d, 1H, J 8.0 Hz), 8.15 (d, 1H, J 8.0 Hz), 8.12 (s, 1H), 8.00-7.82 (m, 6H), 7.66-7.61 (m, 2H), 7.52-7.41 (m, 3H), 6.06 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 178.1, 178.0, 157.7, 148.6, 147.3, 145.1, 135.3, 135.1, 132.0, 131.7, 130.6, 130.4, 130.1, 129.6, 129.4, 128.9, 127.8, 125.9, 124.9, 123.3, 122.1, 117.0, 115.3, 115.2, 35.2; MS (ESI): m/z 434 $[\text{M}+\text{H}]^+$; Anal. Calc. for $\text{C}_{27}\text{H}_{15}\text{NO}_5$: C, 74.82; H, 3.49; N, 3.23. Found: C, 74.76; H, 3.56; N, 3.25%.

14-(2,4-Dichlorophenyl)-14H-dibenzo[a,i]xanthene-8,13-dione (4g): Yellow powder, mp 301-302 °C; ^1H NMR (CDCl_3 , 400 MHz) δ 8.20 (d, 1H, J 8.4 Hz), 8.16-8.11 (m, 2H), 7.89-7.80 (m, 3H), 7.65-7.45 (m, 4H), 7.31-7.27 (m, 2H), 7.07-7.05 (m, 1H), 6.14 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 178.1, 157.6, 147.1, 139.2, 135.2, 134.0, 133.1, 132.6, 131.8, 131.5, 131.1, 130.6, 130.1, 130.0, 129.8, 129.5, 128.7, 127.7, 127.5, 125.7, 124.7, 123.7, 116.8, 33.2; MS (ESI): m/z 457 $[\text{M}+\text{H}]^+$; Anal. Calc. for $\text{C}_{27}\text{H}_{14}\text{Cl}_2\text{O}_3$: C, 70.91; H, 3.09. Found: C, 70.82; H, 3.11%.

14-(3,4-Dichlorophenyl)-14H-dibenzo[a,i]xanthene-8,13-dione (4h): Yellow powder, mp 260-261 °C; ^1H NMR (CDCl_3 , 400 MHz) δ 8.19 (d, 1H, J 7.6 Hz), 8.16 (d, 1H, J 7.6 Hz), 7.95-7.82 (m, 4H), 7.65-7.43 (m, 5H), 7.31-7.30 (m, 2H), 5.92 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 178.2, 178.0, 157.6, 147.3, 143.2, 135.2, 132.7, 132.0, 131.6, 131.1, 130.7, 130.5, 130.4, 130.1, 129.6, 128.8, 128.2, 127.8, 125.8, 124.7, 123.4, 116.8, 115.6, 115.4, 34.6; MS (ESI): m/z 457 $[\text{M}+\text{H}]^+$; Anal. Calc. for $\text{C}_{27}\text{H}_{14}\text{Cl}_2\text{O}_3$: C, 70.91; H, 3.09. Found: C, 70.95; H, 3.02%.

14-(2-Chlorophenyl)-14H-dibenzo[a,i]xanthene-8,13-dione (4i): Yellow powder, mp 281-282 °C; ^1H NMR (CDCl_3 , 400 MHz) δ 8.25-8.22 (m, 2H), 8.14 (d, 1H, J 7.2 Hz), 7.89-7.83 (m, 3H), 7.65-7.29 (m, 6H), 7.08-7.04 (m, 2H), 6.22 (s, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 178.2, 178.1, 157.4, 147.1, 135.2, 133.3, 131.8, 131.4, 131.3, 130.8, 130.2, 130.1, 129.8, 129.4, 128.6, 128.2, 127.6, 127.1, 125.6, 124.7, 124.0, 116.8, 116.7, 115.6, 33.5; MS (ESI): m/z 423 $[\text{M}+\text{H}]^+$; Anal. Calc. for $\text{C}_{27}\text{H}_{15}\text{ClO}_3$: C, 76.69; H, 3.58. Found: C, 76.79; H, 3.41%.

Supplementary Information

^1H NMR, ^{13}C NMR spectra of compounds **4a-i** are available free of charge at <http://jbc.sbcq.org.br>, as pdf file.

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