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A convenient eco-friendly one-pot synthesis of 14-aryl-14*H*-dibenzo [a, j] xanthenes were developed by the condensation of aromatic aldehydes and β -naphthol with BTADCI (Benzyltrimethylammonium dichloroiodate) as catalyst under solvent free conditions and microwave irradiation with much more rate accelerations and afford very good yields. Developed protocols involve mild reaction conditions, simple isolation of derivatives with inexpensive reagents.

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INTRODUCTION

Condensation reactions are a set of eco-friendly reactions assisted with the elimination of simple molecules such as water or ammonia before yielding products. Xanthenes synthesis is simple condensation reaction that has attracted considerable attention of organic and biochemists in recent past because of its simplicity. In these reactions, isolation of intermediate is not required before the end product is obtained; this reduces time and saves both energy and raw materials. Benzoxanthenes are an extraordinary class of xanthenes, which have an extensive variety of organic and helpful properties, for example, antibacterial, antiviral exercises, and furthermore they are hopefuls in photodynamic therapy [1–3]. Xanthenes dyes tend to be fluorescent, yellow to pink to bluish red, brilliant dyes.

Moreover, benzoxanthenes are utilized as dyes in laser technologies [4] and in fluorescent materials [5-7]. The synthesis of benzoxanthenes schematically represented (Scheme 1), which are obtained by the buildup of aldehydes with β -naphthol by Cyclo-dehydration within the sight of a variety of impetuses, for example, AcOH-H2SO4, p-TSA, MeSO₃H, sulfamic corrosive, ionic fluid, iodine, heteropolyacid, silica sulfuric corrosive, Amberlyst-15, cyanuric chloride, LiBr, Yb(OTf)₃, Sc[N(SO₂C₈F₁₇)₂]₃, NaHSO₄, and Al(HSO₄)₃ [8]. Geng-Chen Li has developed a convenient eco-friendly strategy

for the amalgamation of 14-aryl-14H-dibenzo [a, j] xanthenes subsidiaries by one-pot buildup of 2-naphthol and aryl aldehydes catalyzed by extended graphite under solvent-free conditions [9]. Shaterian et al., have declared ferric hydrogen sulfate and aluminum hydrogen sulfate Al(HSO₄)₃ catalyzed the essential one-pot union of 14-aryl-14H-dibenzo [a, j] xanthenes subordinates under warm and solvent-free conditions by development of 2-naphthol and substituted benzaldehyde [10,11]. The union of dibenzoxanthenes by the buildup of aldehydes with β -naphthol within the sight of CBr₄ as an impetus has been reported [12]. Polyvinyl poly pyrrolidonebolstered boron trifluoride (PVPP-BF₃) is a gentle and productive impetus for the union of 14-aryl-14Hdibenzo [a, j] xanthenes and bis(naphthalene-2-yl-sulfane) solvent-free subsidiaries under system [13,14]. Recoverable heterogeneous AgI nanoparticles effectively catalyzed the one-pot union of 14-aryl-14H-dibenzo [a, j] xanthenes through multi-segment response of aldehydes and 2-naphthol under solvent-free conditions [15]. Microwave helped green mix of dibenzo [a, j] xanthenes using a capable, proficient, and eco-pleasing procedure for the mix of 14-aryl-14H-dibenzo [a, j] xanthenes has been refined by methods for p-dodecylbenzenesulfonic acid (DBSA) under solvent-free microwave conditions [16], [H-NMP][HSO₄] as a Brønsted acidic ionic fluid under solvent-free conditions, at 110°C [17], sulfonic corrosive functionalized silica (SiO₂-Pr-SO₃H) under solvent free



Scheme 1. BTADCI Promoted One-pot synthesis of 14-aryl-14H-dibenzo [a, j] xanthenes.



conditions [18,19]. The one-pot combination of 14-aryl-14*H*-dibenzo [a, j] xanthenes catalyzed by assignment particular ionic fluid a Brønsted-acidic undertaking particular ionic fluid, 1-methyl-3-propane sulfonicimidazolium hydrosulfate ([MIMPS] HSO₄), *via* the one-pot condensation of β -naphthol with aliphatic or sweet-smelling aldehydes [20]. All responses are performed without dissolvable in generally short response times in great to high yields [21]. This prompted a sensational increment in the quantity of distributions utilizing MW in natural synthesis [22–24].

As expanding natural cognizance in synthetic research and industry, the test for a maintainable situation consistently calls for clean procedures [25]. Which are fairly long to grow this kind of pair process that would allow the buildup of the in situ produced orthoquninonemethide with nucleophile other than phenols, naphthol compounds using BTADCI as a catalyst until now has not been explored in synthetic organic chemistry. In our attempt to explore the role, BTADCI is mild and efficient Lewis acid catalyst. A major concern to overcome such problems in organic synthesis has considered the use of solvent free and microwave irradiation as a source of energy.

RESULTS AND DISCUSSION

In view of our ongoing interest in green chemistry, the author has taken up solvent free and microwave irradiated one-pot synthesis of 14-aryl-14*H*-dibenzo [a, j] xanthenes derivatives using condensation of aromatic aldehydes and β -naphthol with BTADCI. The reaction was completed in 70 to 90 min in solvent free condition at 110°C and 8 to 10 min in microwave conditions (Table 1) to obtain14-(3-Fluorophenyl)-14*H*-dibenzo [a, j] xanthenes (Scheme 1).

The most plausible mechanism of is proposed as shown in Scheme 2. First step involves proton abstraction by the arylaldehyde leading to the formation of aryl oxocarbenium ion intermediate (II), which in turn reacts with β -naphthol in a condensation step to afford naphthyloxonium intermediate (III) followed by the removal of water molecule as shown in Scheme 2. The naphthyloxonium intermediate (III), thus, formed reacts with one more β - naphthol to give target molecule assisted by the elimination of water molecule. The synthesized xanthene derivatives were fullycharacterized by FTIR, 1H NMR, 13C NMR, and mass spectrometer (Supplementary data).

EXPERIMENTAL

General remarks. All chemicals used were reagent grade and were used as received without further purification. ¹H NMR and ¹³C NMR spectra were recorded at 25°C at 300 MHz and 75 MHz (Bruker Avance, Switzerland) instrument, respectively, using TMS as internal standard. Chemical shifts are given in parts per million (ppm). The FT-IR spectroscopy of samples was carried out on a Perkin Elmer (Maryland, USA) precisely 100 FT-IR spectrometer in the 400–4000 cm^{-1} region. LCMS mass spectra were recorded on a MASPEC low resolution mass spectrometer operating at 70 eV. Elemental analyses were carried out using a Perkin-Elmer CHNS Elemental Analyzer model 2400. Melting points are uncorrected and were recorded on a hot stage melting point apparatus Ernst Leitz Wetzlar, Germany. The MW-assisted synthesis of titled compounds was carried out in a CEM - 908010 (Smith Farm Rd, Mathews, N.C-28105, US), bench mate model, 300 watts laboratory MW reactor. Reactions were monitored, and the purity of products was checked out on thin layer chromatography (TLC) on aluminum-backed plates coated with Kieselgel 60 F254 silica gel, visualizing the spots under ultraviolet light and iodine chamber.

General method for the synthesis of xanthenes derivatives (3a–j). The reaction mixture was of β -naphthol (2 mmol), 3-fluorobenzaldehyde (1 mmol), and BTADCI (60 mg) was heated at 110°C until completion, the reaction mixture was cooled to room temperature and extracted in to ethyl acetate and washed with 15% Na₂CO₃; the resultant mixture was purified by column chromatography using silica gel (60–120 mesh size) and recrystallized in aqueous EtOH (15%).

General method for the synthesis of xanthenes derivatives under microwave irradiation (3a–j). A mixture of β -naphthol (2 mmol), aldehydes (1 mmol), and BTADCI

S. No.	Aldehydes	Products (3a-j)	Solvent free	Micro wave
1	4-F-C ₆ H ₄ -CHO	3a	75	85
2	2-NO ₂ -C ₆ H ₄ -CHO	3b	70	80
3	4-Me-C ₆ H ₄ -CHO	3c	85	95
4	4-OMe-C ₆ H ₄ -CHO	3d	82	93
5	4-OH-C ₆ H ₄ -CHO	3e	85	95
6	2-Cl-C ₆ H ₄ -CHO	3f	80	95
7	4-Cl-C ₆ H ₄ -CHO	3g	80	90
8	C ₆ H ₄ -CHO	3h	80	90
9	4-Br-C ₆ H ₄ -CHO	3i	75	90
10	3-OH-C ₆ H ₄ -CHO	3j	80	95
11	3-Br-C ₆ H ₄ -CHO	3k	79	94
12	3-OMe-C ₆ H ₄ -CHO	31	80	95

 Table 1

 The synthesis of 14-aryl-14H-dibenzo [a. j] xanthene derivative by BTADCI.

Reaction times: Microwave 8-10 min; solvent free 70-90 min; isolated yields.

Scheme 2. Proposed mechanism of 14-aryl-14H-dibenzo [a, j] xanthenes promoted by BTADCI.



(60 mg) was placed in a laboratory microwave oven (BPL, 800 T model) on a silica gel solid support and heated at 300 W for the appropriate time. The product isolation

and purification methods are same as aforementioned and recrystallized in aqueous EtOH (15%) to obtain pure product.

14-(4-FlroPhenyl)-14H-dibenzo [a,j] xanthenes (3a). White solid; mp 230–232°C; ¹HNMR (400 MHz, CDCl₃): $\delta = 6.49$ (s, 1H), 6.78 (t, J = 8.4 Hz, 2H), 7.38–7.47 (m, 6H), 7.55 (t, J = 8.0 Hz, 2H), 7.77 (m, 4H), 8.31 (d, J = 8.4 Hz, 2H); ¹³CNMR (100 MHz, CDCl₃): $\delta = 37.1$, 115.1, 115.4, 117.1, 118.0, 122.4, 124.3, 126.8, 128.8, 128.9, 129.5, 129.6, 131.0, 131.3, 148.7; IR (KBr cm⁻¹): 3068, 2926, 1591, 1457, 1396, 1243, 1157, 1087, 957, 809, 742; EI-MS: m/z (%) = 376 (M⁺); *Anal.* Calcd for C₂₇H₁₇FO: C, 86.15; H, 4.55; F, 5.05. Found: C, 86.12; H, 4.41, F, 5.01.

14-(2-Nitrophenyl)-14H-dibenzo [a, j] xanthenes (3b).

Yellow solid; mp 211–213°C; ¹HNMR (400 MHz, CDCl₃): δ = 7.06–7.23 (m, 2H), 7.41–7.44 (m, 2H), 7.47–7.83 (m, 11H), 8.51 (d, J = 8.4 Hz, 2H); ¹³CNMR (100 MHz, CDCl₃): δ = 32.5, 117.5, 118.0, 122.5, 124.6, 124.9, 127.3, 127.5, 128.7, 129.4, 130.9, 131.7, 132.2, 134.1, 140.8, 147.0, 149.3; IR (KBr, cm⁻¹): 3400, 3057, 1625, 1593, 1397,, 1305, 817, 750; EI-MS: m/z (%) = 418 (M⁺); *Anal.* Calcd for C₂₇H₁₇NO₃: C, 80.38; H, 4.25; N, 3.47, Found: C, 80.29; H, 4.26, N, 3.53.

14-(4-Methylphenyl)-14H-dibenzo [a, j] xanthenes (3c). Yellow solid; mp 221–223°C; ¹HNMR (400 MHz, CDCl₃): δ = 2.12 (s, 3H), 6.45 (s, 1H), 6.94 (d, J = 8.0 Hz, 2H), 7.38–7.42 (m, 4H), 7.46 (d, J = 8.8 Hz, 2H), 7.55–7.59 (m, 2H), 7.77–8.38 (m, 6H); ¹³CNMR (100 MHz, CDCl₃): δ = 20.8, 37.6, 117.4, 118.0, 122.7, 124.2, 126.7, 128.0, 128.7, 128.7, 129.1, 131.1, 131.4, 135.8, 142.1, 148.7; IR (KBr, cm⁻¹): 3073, 1625, 1513, 1435, 1258, 1123, 1086, 966, 841, 813, 744; EI-MS: m/z (%) = 372 (M⁺); Anal. Calcd for C₂₈H₂₀O: C, 90.29; H, 5.41. Found: C, 90.36; H, 5.46.

14-(4-Methoxyphenyl)14H-dibenzo[a, j]xanthenes (3d). Off-white solid; mp 203–205°C; ¹HNMR (400 MHz, CDCl₃): δ = 3.61 (s, 3H), 6.44 (s, 1H), 6.57–6.67 (m, 2H), 7.38–7.48 (m, 6H), 7.55–7.83 (m, 6H), 8.39 (d, J = 8.4 Hz, 2H); ¹³CNMR (100 MHz, CDCl₃): δ = 37.1, 55.0, 113.8, 117.5, 118.0, 122.6, 124.2, 126.7, 128.7, 128.8, 129.1, 131.1, 131.4, 137.3, 148.7, 157.8; IR (KBr cm⁻¹): 3062, 1594, 1509, 1458, 1398, 1247, 960, 814, 744. EI-MS: m/z (%) = 388 (M⁺); *Anal.* Calcd for C₂₈H₂₀O₂: C, 86.57; H, 5.19. Found: C, 86.60; H, 5.23.

14-(4-Hydroxyphenyl)-14H-dibenzo [a, j] xanthenes (3e). Pink solid; mp 139–141°C; ¹HNMR (400 MHz, CDCl₃): δ = 6.43 (s, 1H), 6.57–6.59 (m, 2H), 7.35–7.59 (m, 8H), 7.76–7.83 (m, 4H), 8.35 (d, J = 8.4 Hz, 2H); ¹³CNMR (100 MHz, CDCl₃): δ = 37.0, 115.2, 117.4, 117.9, 122.6, 124.2, 126.7, 128.7, 128.8, 129.3, 131.0, 131.3, 137.4, 148.6, 153.8; IR (KBr, cm⁻¹): 3393, 1596, 1514, 1437, 1404, 1257, 958, 818, 748; EI-MS: m/z (%) = 374 (M⁺); *Anal.* Calcd for C₂₇H₁₈O₂: C, 86.61; H, 4.85. Found: C, 86.56; H, 4.82.

14-(2-Chlorophenyl)-14H-dibenzo [a, j] xanthenes (3f). Off-white solid; mp 178–180°C; ¹HNMR (400 MHz, CDCl₃): δ = 7.25 (s, 1H), 7.34 (m, 2H), 7.70–8.28 (m,

12H), 9.18 (d, J = 8.4 Hz, 2H); ¹³CNMR (100 MHz, CDCl₃): $\delta = 33.5$, 116.9, 117.0, 122.3, 123.3, 125.8, 126.7, 126.8, 127.5, 128.0, 128.5, 129.0, 129.8, 130.7, 142.5, 147.9; IR (KBr cm⁻¹): 3069, 1695, 1591, 1430, 1243, 747, 692; EI-MS: m/z (%) = 394 (M⁺). Anal. Calcd for C₂₇H₁₇ClO: C, 82.54; H, 4.36; Cl, 9.02. Found: C, 82.60; H, 4.32, Cl, 9.04.

14-(4-Chlorophenyl)-14H-dibenzo [a, j] xanthenes (3g). Yellow solid; mp 289–291°C; ¹HNMR (400 MHz, CDCl₃): $\delta = 6.46$ (s, 1H), 7.08 (d, J = 8.0 Hz, 2H), 7.42–7.57 (m, 8H), 7.78–7.84 (m, 4H), 8.30 (d, J = 8.0 Hz, 2H); ¹³CNMR (100 MHz, CDCl₃): $\delta = 37.4$, 116.8, 118.0, 122.4, 124.3, 126.9, 128.6, 128.9, 129.0, 129.4, 131.1, 131.3, 132.1, 143.4, 148.7; (IR KBr cm⁻¹): 3066, 1622, 1514, 1456, 1397, 1245, 1209, 1065, 959, 812, 775, 746. EI-MS: m/z (%) = 392 (M⁺). Anal. Calcd for C₂₇H₁₇ClO: C, 82.54; H, 4.36. Found: C, 82.60; H, 4.39.

14-Phenyl-14H-dibenzo [a, j] xanthenes (3h). Off-white solid; mp 185–187°C; ¹HNMR (400 MHz, CDCl₃): δ 6.45 (s, 1H), 6.96 (t, J = 7.2 Hz, 1H), 7.12 (t, J = 7.2 Hz, 2H), 7.37–7.58 (m, 8H), 7.74–7.81 (m, 4H), 8.34 (d, 2H); ¹³CNMR (100 MHz, CDCl₃): $\delta = 38.0$, 117.3, 118.0, 122.6, 124.2, 126.3, 126.7, 128.2, 128.4, 128.7, 128.8, 131.0, 131.4, 148.7; IR (KBr cm⁻¹): 3070, 1585, 1445, 1250, 1080, 965; EIMS, 70 eV, m/z: 358 (M+); Anal. Calcd for C₂₇H₁₈O: C, 90.47; H, 5.06. Found: C, 90.43; H, 5.08.

14-(4-Bromophenyl)-14H-dibenzo [a, j]-xanthene (3i). Off-white solid; mp 295–296°C; ¹HNMR (400 MHz, CDCl₃): δ 8.32 (d, J = 8.4 Hz, 2H), δ 7.85–7.78 (m, 4H), δ 7.60–7.55 (m, 2H), δ = 7.48–7.37 (m, 5H), δ 7.25–7.23 (m, 3H), δ 6.46 (s, 1H); ¹³CNMR (75 MHz, CDCl₃): d 147.9, 144.8, 131.2, 130.7, 130.6,130.0, 129.2, 128.6, 127.0, 124.6, 123.2, 119.4, 117.6, 116.8, 36.8; IR (KBr, cm⁻¹): 3056, 2922, 1622, 1590, 1514, 1456, 1392, 1243, 1231, 1207, 1143,1062, 957, 816, 772, 741; EIMS, 70 eV, m/z: 437 (M+).

14-(3-Hydroxyphenyl)-14H-dibenzo [a, j]-xanthene (3j). Off-white solid; mp 242–243°C. ¹HNMR (400 MHz, CDCl₃): δ 8.37 (d, J = 8.7 Hz, 2H), δ 7.83–7.76 (m, 4H), δ 7.59–7.54 (m, 2H), δ 7.47–7.37 (m, 4H), δ 7.18 (d, J = 7.8 Hz, 1H), δ 7.03 (t, J = 8.1 Hz, 1H), δ 6.86 (s, 1H), δ 6.64–6.43 (m, 2H), δ 4.54 (s, 1H); ¹³CNMR (100 MHz, CDCl₃): d 154.8, 147.9, 145.9, 130.6, 130.2,128.6, 128.1, 128.0, 126.0, 123.5, 121.9, 120.1, 117.2, 116.3, 114.6, 112.8, 37.0; IR (KBr, cm⁻¹): 3412, 1588, 1507, 1413, 1258, 1237, 813; EIMS, 70 eV, m/z: 374 (M+); *Anal.* Calcd for C27H18O2: C, 86.61; H, 4.85. Found: C, 86.58; H, 4.81.

14-(3-BromoPhenyl)-14H-dibenzo [a, j] xanthenes (3k).

Off-white solid; m.p. 197–199; ¹HNMR (400 MHz, CDCl₃): $\delta = 6.45$ (s, 1H), 7.0–7.13 (m, 2H), 7.41–7.62 (m, 8H), 7.80–8.31 (m, 6H); ¹³CNMR (100 MHz,

CDCl₃): δ = 37.7, 116.5, 118.0, 122.7, 124.3, 126.8, 126.9, 128.9, 129.1, 129.6, 129.9, 131.0, 131.1, 131.2, 147.1, 148.8 IR (KBr cm⁻¹): = 3066, 3020, 1628, 1594, 1398, 1249, 810, 740; EI-MS: m/z (%) = 436 (M⁺); *Anal.* Calcd for C₂₇H₁₇BrO: C, 74.15; H, 3.92. Found: C, 74.17; H, 3.99.

14-(3-Methoxyphenyl)14H-dibenzo[a, j]xanthenes (31). Off-white solid; mp 160–162°C; ¹HNMR (400 MHz, CDCl₃): δ = 3.63 (s, 3H), 6.46 (s, 1H), 6.51–6.53 (m, 1H), 7.02–7.17 (m, 3H), 7.38–7.83 (m, 10H), 8.38 (d, J = 8.4 Hz, 2H); ¹³CNMR (100 MHz, CDCl₃): δ = 37.9, 54.9, 110.9, 114.8, 117.9, 120.7, 122.6, 124.1, 126.7, 128.7, 128.8, 129.2, 131.0, 131.4, 146.4, 148.7, 159.5; IR (KBr cm⁻¹): 3015, 2951, 2940, 1594, 1490, 1455, 1439, 1400, 1250 cm⁻¹; EI-MS: m/z (%) = 388 (M⁺); *Anal.* Calcd for C28H20O2: C, 86.45; H, 5.06. Found: C, 86.58; H, 5.12.

CONCLUSION

In brief, the authors have developed a convenient ecofriendly procedure for one-pot synthesis of 14-aryl-14*H*dibenzo [a, j] xanthene derivatives by the condensation of β -naphthol and aromatic aldehydes catalyzed by BTADCI under solvent free and micro wave conditions. The developed methodologies offer advantages such as excellent yields, shorter reaction times, and low cost of the catalyst. Thus, the present method provides Green-Reaction conditions with a simple work-up procedure.

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