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An eco-friendly and new facile method for synthesis of prenylated or geranylated acylphloroglucinol-based xanthenones

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ABSTRACT

The synthesis of prenylated or geranylated acylphloroglucinol-based xanthenones with 15.1–36.9% yield was achieved *via* aldol condensation of 2,4,6-trihydroxy-3-prenylacetophenone (tHPA) or 2,4,6-trihydroxy-3-geranylacetophenone (tHGA) and 2-hydroxybenzaldehyde derivatives in the presence of lithium bis(trimethylsilyl) amide (LiHMDS) as deprotonating agent. This study is one of the first to report on the synthesis of xanthenones using strong non-nucleophilic base catalyst, and the described synthesis not only achieved a compound with a natural product skeleton but also formed in a simple and environmentally friendly specificity.

Introduction

Diversity oriented synthesis which maximizes skeletal diversity and chemical space had demonstrated a powerful approach to design and synthesize compounds with natural product skeleton [1]. Xanthenones are one of the important secondary metabolites produced by many microorganisms which exhibit many biological activities e.g., antiproliferative [2,3], antiviral [4], anti-microbial [5] and anti-inflammatory [6] etc. Furthermore, xanthenone due to the spectroscopic properties have been used as dyes [7], in laser technology and electrographic toners [8], for the visualization of biomolecules and as marker or biological stain [9]. Thus, significant attention has been given to the synthesis of this highly functionalized compound.

Generally, xanthenone synthesis involves a multicomponent reaction using a phenolic compound, an aldehyde, and a cyclic 1,3-dicarbonyl compound [10]. Many Lewis or Bronsted acids [2–4,11–19] have been used as catalysts for xanthenone synthesis (Table 1), however, their uses have been restricted due to several drawbacks including less availability or hard preparation, expensive, harmful, and toxic. In addition, synthesis reaction involving these acid catalysts requires long reaction time, burdensome product isolation procedures, and toxic organic solvents to perform the reaction or purification [20]. In addition, Terra et al., (2017) reported a green methodology by using organic acid (oxalic acid/succinic acid/acetic acid) as catalyst for the synthesis of xanthenones from the one-pot condensation of benzaldehyde derivatives, β-naphthol, and dimedone. This alternative green method provides several advantages including eco-friendly reaction conditions, easy workup procedure, short reaction times and good yields (up to 93 %).

These facts encouraged us to explore other green method using less common base lithium bis(trimethylsilyl)amide (LiHMDS) which is a cheap, mild, non-hazardous, efficient, and eco-friendly reagent [21], for the synthesis of an important class of the xanthenones **3(a-f)**. This work is to explore the use of a non-nucleophilic base catalyst for the synthesis of xanthenones.

In continuation of our previous works on the design and synthesis of compounds based on the isolated natural lead i.e., 2,4,6-trihydroxy-3-geranylacetophenone, a geranylated acylphloroglucinol [22,23], herein has been reported a simple and green method for the synthesis of prenylated and geranylated acylphloroglucinol-based xanthenones **3(a-f)** *via* aldol condensation, using LiHMDS as base catalyst (Scheme 1 and Table 2). Since LiHMDS is moisture sensitive and flammable, we have performed the reaction under nitrogen gas atmosphere, at room

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Table 1
Previously reported work for the synthesis of xanthenones using Lewis or Bronsted acids as catalysts.

Catalyst	Conditions	Time (min)	Yield (%)	Ref.
Calixarenes (CX)	120 °C/stir	60	68–74	2
Ceric ammonium nitrate (CAN)	120 °C	30	85–94	3
Polyethyleneglycol bound sulfonic acid (PEG-SO_3H)	60–65 °C/stir	30	97	4
Tri-methyl tetradecyl ammonium bromide (TTAB)	H_2O/rt/stir	150	85–91	11
Sulfamic acid (NH_2SO_3H)	120 °C/stir	90–129.6	79–84	12
***p*-toluenesulfonic acid (pTSA)**	[bmim]BF_4/ 80 °C/stir	120–210	83–95	13
Indium(III) chloride ($InCl_3$)	120 °C	25–75	68–88	14
Boron trifluoride diethyl etherate (BF_3:OEt_2)	C_2H_5OH/ reflux	180	70–80	15
Functionalized ionic liquids	240 W/heat	10	84	16
Succinimide-N-sulfonic acid (SuSA)	80 °C/heat	30–48	82–96	17
Ytterbium perfluorooctanoate [Yb(PFO)$_3$]	80 °C/ irradiated	5	84–93	18
Thiamine hydrochloride (VB_1)	CTAB-H_2O[a]/rt	25–210	76–92	19

[a] Hexadecyltrimethylammonium bromide.

temperature. The prenylated and geranylated acylphloroglucinol (**1**) were synthesized by direct *C*-alkylation of 2,4,6-trihydroxyacetophenone with prenyl or geranyl bromide as alkylating agent [23].

Experimental

Materials and apparatus

Chemicals, reagents and solvents such as 2′,4′,6′-trihydroxyacetophenone (98 %), prenyl bromide (90 %), geranyl bromide (95 %), LiHMDS (1 M solution in THF/Ethylbenzene), salicylaldehyde (98 %), 2-Chloro-6-hydroxybenzaldehyde (98 %), 4-Diethylamino-2-hydroxybenzaldehyde (98 %), toluene, hydrochloric acid (37 %), dichloromethane, sodium sulphate anhydrous, silica gel 60 (0.063–0.2MM), hexane, ethyl acetate were purchased from Ambeed USA, Sigma, Thermo Fisher Scientific, Merck and Systerm chemical companies. The industrial nitrogen gas (99.9 %) used in the chemical synthesis was purchased from Premium Gases Supply (M) Sdn Bhd. All synthesized compounds were characterized by their spectral (Melting point, ^{1}H NMR, ^{13}C NMR and MS). Melting points were determined by using a hot stage melting point apparatus equipped with microscope, Fisher-Johns and were uncorrected. The ^{1}H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ (99.8 %, Sigma-Aldrich) using Varian 500 MHz NMR spectrometer. Mass analysis was done using Shimadzu QP-2010 Ultra gas chromatography-mass spectrometer (GC–MS) with Electron Ionization (EI) method by using Direct Inlet Probe (DIP) system. Thin layer chromatography (TLC) aluminium sheets, silica gel layer, ALUGRAM XTRA SIL G UV254 was used to monitor the progress of the reactions.

Table 2
Synthesis of prenylated or geranylated acylphloroglucinol-based xanthenones 3 (a-f) under nitrogen gas atmosphere at room temperature using LiHMDS as base catalyst.

Compound	R_1	R_2	R_3	mp (°C)	Yield[a] (%)
3a	C_5H_9	H	H	179	20.9 %
3b	C_5H_9	H	Cl	150	15.1 %
3c	C_5H_9	$(C_2H_5)_2N$	H	162	16.6 %
3d	$C_{10}H_{17}$	H	H	142	28.7 %
3e	$C_{10}H_{17}$	H	Cl	153	22.6 %
3f	$C_{10}H_{17}$	$(C_2H_5)_2N$	H	130	36.9 %

[a] Yields refer to pure isolated compounds characterized by ^{1}H & ^{13}C NMR spectroscopy and mass spectrometry.

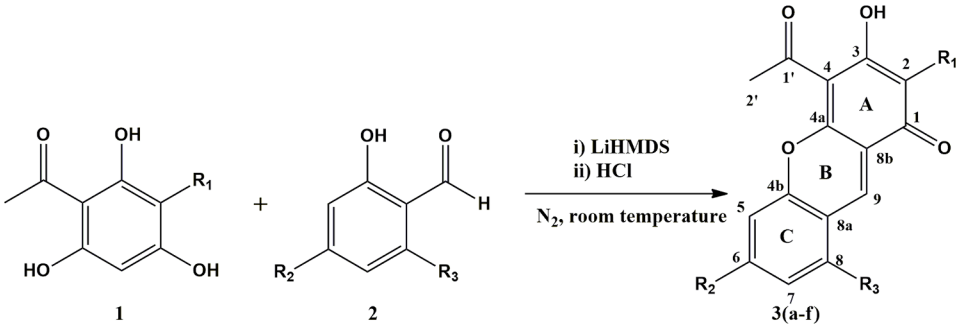
*Synthesis of prenylated or geranylated acylphloroglucinol-based xanthenones **3a-f***

Xanthenones were synthesized using aldol condensation adopted from Rosa et al., (2019) with modifications [24]. The 2,4,6-trihydroxyprenylacetophenone (tHPA)/2,4,6-trihydroxygeranylacetophenone (tHGA) (**1**, 0.0015 mol) was dissolved in dried toluene (10 mL) and mixed with a solution of LiHMDS (10 mL, 0.0099 mol), under nitrogen atmosphere, at room temperature. After 30 min, 2-hydroxybenzaldehyde derivatives (**2**, 0.0015 mol) were added and the reaction mixture was stirred for 5 days at room temperature. The reaction mixture was poured over crushed ice and acidified with HCl (37 %) to pH < 2 following by extraction with dichloromethane (CH_2Cl_2) and purified using column chromatography over silica gel eluted with hexane/ethyl acetate (10:1) gave xanthenones.

Results and discussion

Percentage of yield & mechanism of the reaction

The utilization of the LiHMDS for the synthesis of xanthenones **3(a-f)** in this study showed a low yield (15.1–36.9 %) when used under room temperature (compared to the catalysts: TTAB (85–91 %) and VB_1 (76–92 %) in Table 1). Both TTAB and VB_1 were used to synthesize the tetrahydrobenzo(a)xanthenes-11-one derivatives, while the pronounced catalytic effect of LiHMDS in the synthesis of prenylated or geranylated acylphloroglucinol-based xanthenones **3(a-f)** is described for the first time. Wu et al., (2021) reported the use of LiHMDS in the synthesis of xanthones, particularly dihydroxanthones (DHSx) and tetrahydroxanthones (THXs) [25]. As compared to the mentioned study, the yield of the synthesized xanthones showed comparable and lower yield (29 %) when 3 equivalent LiHMDS was used as catalyst, and reagents used was in 1:1 ratio. In contrast, in micellar solution of TTAB and VB_1, hydrophobic reactants moved towards the hydrophobic core of micelle droplets, causing an increased in the effective concentration of the reactants and efficient collisions between the reactants which leads to an increased in the reaction rate and yield [19,26–28].

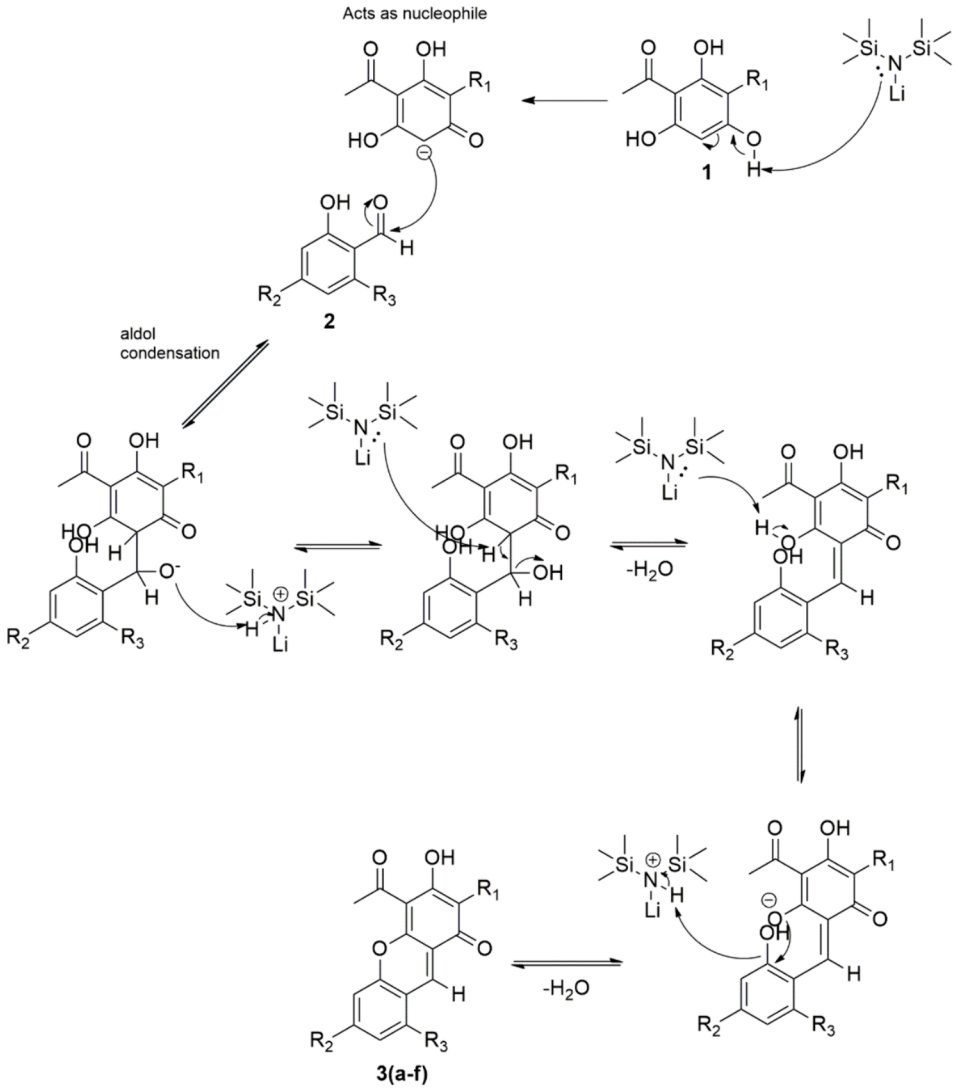


Scheme 1. Synthesis of prenylated or geranylated acylphloroglucinol-based xanthenones.

In accordance with the literature [29–32], the proposed mechanism for the LiHMDS catalyzed one-pot reaction of prenylated or geranylated acylphloroglucinol (**1**), 2-hydroxybenzaldehyde derivative (**2**), is shown in Scheme 2. It is suggested that one of the exposed hydroxyl groups on the phloroglucinol ring of acylphloroglucinol (**1**) is deprotonated by LiHMDS causing the oxygen of the hydroxyl group to become more electron rich and the formation of C = O bond *via* electrons transfer. This activates the "π-donation" causing neighboring α-carbon to be nucleophilic (break of C = C bond). The nucleophilic *α*-carbon attacks the electrophilic carbonyl carbon of the benzaldehyde (**2**), forming an alkoxide intermediate which has a tetrahedral geometry. The LiHMDS H^+ protonates the alkoxide intermediate to afford a dienone adduct and LiHMDS is recycled. Meanwhile, the recycled LiHMDS deprotonates an α-carbon which is adjacent to a carbonyl group of the phloroglucinol core (formation of C = C bond), and a water molecule is removed. The same deprotonating mechanism catalyzed by LiHMDS on the other exposed hydroxyl group of phloroglucinol is observed, causing the formation of carbanion which forms a single bond by eliminating an OH group. This OH group is protonated by LiHMDS H^+, and a water molecule is removed to afford the xanthenone products **3**. Finally, a strong hydrochloric acid (HCl) protonates the recycled LiHMDS to terminate the reaction which allows the LiHMDS decomposed during the water work-up extraction process, and hence cannot be recycled [21].

Spectroscopic data of the synthesized compounds

The structure of xanthenones **3(a-f)** were deduced from their ^{1}H NMR, ^{13}C NMR, and MS spectral data (see supplementary data). The spectroscopic data have been given in the supplementary data. The NMR data for the acylphloroglucinol-based xanthenone core structure and their corresponding prenylated or geranylated derivatives were observed to be similar. In the ^{1}H NMR spectrum of xanthenone (**3a**, Fig. A.1a), a singlet that appeared at δ_H 8.39 ppm is assigned to a methine proton (CH) at C-9 of ring B. The four aromatic protons of ring C appeared in the region of δ_H 7.33–7.62 ppm. Three sharp singlets appeared at δ_H 2.73 ppm, δ_H 1.86 ppm and δ_H 1.70 ppm are assigned to the methyl proton (CH_3) of acetyl and prenyl substituents of ring A, respectively. The ^{13}C NMR spectrum (Fig. A.1b) shows 20 signals and the appearance of the methine signals resonating in the δ_C 110–170 region suggested the presence of an aromatic ring C. The signals occurring at δ_C 202.90 ppm, δ_C 184.57 ppm, δ_C 177,43 ppm, δ_C 156.47 ppm, δ_C 151.79 ppm, δ_C132.76 ppm, δ_C 128.78 ppm, δ_C 119.96 ppm, δ_C 109.07 ppm, and δ_C 107.62 ppm were assigned to quaternary carbons. The signal δ_C 202.90 ppm and δ_C 184.57 ppm correspond to the carbonyl (C = O) moiety while the signal resonating at δ_C 177.43 ppm is characteristic of carbon attached to hydroxyl group within a phenolic ring A. Both NMR spectra (Fig. A.1a & Fig. A.1b) confirmed the C6-C1-C6



Scheme 2. The proposed mechanism for the synthesis of prenylated or geranylated acylphloroglucinol-based xanthenones under nitrogen gas atmosphere catalyzed by non-nucleophilic base LiHMDS.

carbon skeletal of xanthenone (**3a**) with two different types of substituents i.e., acetyl and prenyl group, providing clear evidence that aldol condensation using LiHMDS as deprotonating agent resulted in the cyclized product. The NMR data for xanthenone (**3b**, Fig. A.2a & Fig. A.2b) & (**3c**, Fig. A.3a & Fig. A.3b) were also similar to that of (**3a**) except for the missing proton signals at C-8 of (**3b**), and C-6 of (**3c**); addition proton (Ha and Hb) and carbon signals at C-6 of (**3c**). As in the case of geranylated acylphloroglucinol-based xanthenones (**3d-f**, Fig. A.4a-6a & Fig. A.4b-6b), three singlets appearing in the range from δ_H 1.54–1.86 ppm were integrated for three methyl protons (CH_3) each at C-8″, C-9″ and C-10″ of geranyl group. The methylene proton (CH_2) at C-1″, C-4″ & C-5″ of geranyl group appeared in the region of δ_H 3.36–3.38 ppm and δ_H 1.98–2.06 ppm; methine proton (CH) at C-2″ and C-6″ of geranyl group appeared in the region of δ_H 5.04–5.23 ppm. However, for prenylated compounds, there are only two singlets and one triplet that could be assigned as methyl and methine groups of the alkyl chain, respectively.

4-acetyl-3-hydroxy-2-(3-methylbut-2-en-1-yl)-1H-xanthen-1-one (3a): Dark red amorphous solid; mp 178–180 °C; ^{1}H NMR (500 MHz, Chloroform,- d4) δ_H 8.39 (1H, s, H-9), 7.62 (2H, t, J = 7.5 Hz, H-5,7), 7.39 (1H, d, J = 5.0 Hz, H-8), 7.33 (1H, t, J = 7.5 Hz, H-6), 5.22 (1H, t, J = 7.5 Hz, H-2″), 3.37 (2H, d, J = 5.0 Hz, H-1″), 2.73 (3H, s, H-2′), 1.86 (3H, s, H-5″), 1.70 (3H, s, H-4″); ^{13}C NMR (126 MHz, Chloroform-d_4) δ_C 202.90 (C-1′), 184.57 (C-1), 177.43 (C-3), 156.47 (C-4a), 151.79 (C-4b), 138.03 (C-9), 134.22 (C-7), 132.76 (C-3″), 130.51 (C-5), 128.78 (C-8b), 125.11 (C-6), 120.75 (C-2″), 119.96 (C-4), 116.40 (C-8), 109.07 (C-8a), 107.62 (C-2), 29.99 (C-2′), 25.80 (C-4″), 20.97 (C-1″), 17.93 (C-5″). DIP: m/z 322.10.

4-acetyl-8-chloro-3-hydroxy-2-(3-methylbut-2-en-1-yl)-1H-xanthen-1-one (3b): Dark maroon amorphous solid; mp 148–151 °C; ^{1}H NMR (500 MHz, Chloroform,- d4) δ_H 8.67 (1H, s, H-9), 7.50 (1H, t, J = 7.5 Hz, H-6), 7.34 (1H, d, J = 10.0 Hz, H-7), 7.26 (2H, t, J = 5.0 Hz, H-5), 5.20 (1H, t, J = 7.5 Hz, H-2″), 3.34 (2H, d, J = 5.0 Hz, H-1″), 2.73 (3H, s, H-2′), 1.84 (3H, s, H-5″), 1.69 (3H, s, H-4″); ^{13}C NMR (126 MHz, Chloroform-d_4) δ_C 202.82 (C-1′), 185.04 (C-1), 177.25 (C-3), 155.50 (C-4a), 154.32 (C-4b), 134.96 (C-8), 133.63 (C-6), 133.59 (C-9), 132.96 (C-3″), 125.60 (C-7), 125.47 (C-8b), 120.47 (C-2″), 118.81 (C-4), 115.00 (C-5), 109.16 (C-8a), 108.42 (C-2), 29.77 (C-2′), 25.78 (C-4″), 21.00 (C-1″), 17.93 (C-5″). DIP: m/z 356.05.

4-acetyl-6-(diethylamino)-3-hydroxy-2-(3-methylbut-2-en-1-yl)-1H-xanthen-1-one (3c): Dark purple amorphous solid; mp 160–164 °C; ^{1}H NMR (500 MHz, Chloroform,- d4) δ_H 8.40 (1H, s, H-9), 7.42 (1H, d, J = 5.0 Hz, H-8), 6.65 (1H, d, J = 5.0 Hz, H-7), 6.48 (1H, s, H-5), 5.24 (1H, t, J = 7.5 Hz, H-2″), 3.48 (4H, q, J = 8.3 Hz, H-6a), 3.34 (2H, d, J = 5.0 Hz, H-1″), 2.72 (3H, s, H-2′), 1.86 (3H, s, H-5″), 1.69 (3H, s, H-4″), 1.27 (3H, t, J = 7.5 Hz, H-6b); ^{13}C NMR (126 MHz, Chloroform-d_4) δ_C 203.29 (C-1′), 180.79 (C-1), 177.12 (C-3), 167.74 (C-6), 157.93 (C-4a), 156.89 (C-4b), 153.22 (C-8a), 139.86 (C-9), 132.22 (C-8), 130.86 (C-3″), 121.73 (C-2″), 118.79 (C-4), 110.52 (C-7), 109.29 (C-8b), 104.24 (C-2), 96.35 (C-5), 45.22 (C-6a), 30.34 (C-2′), 25.81 (C-4″), 20.89 (C-1″), 17.90 (C-5″), 12.53 (C-6b). DIP: m/z 393.15.

(E)-4-acetyl-2-(3,7-dimethylocta-2,6-dien-1-yl)-3-hydroxy-1H-xanthen-1-one (3d): Dark red amorphous solid; mp 140–143 °C; ^{1}H NMR (500 MHz, Chloroform,- d4) δ_H 8.39 (1H, s, H-9), 7.62 (2H, dd, J = 10.0, 5.0 Hz, H-5 & H-7), 7.38 (1H, d, J = 5.0 Hz, H-8), 7.33 (1H, t, J = 7.5 Hz, H-6), 5.22 (1H, t, J = 7.5 Hz, H-2″), 5.04 (1H, t, J = 7.5 Hz, H-6″), 3.38 (2H, d, J = 5.0 Hz, H-1″), 2.73 (3H, s, H-2′), 2.06 (2H, q, J = 6.7 Hz, H-5″), 1.98 (2H, t, J = 7.5 Hz, H-4″), 1.86 (3H, s, H-9″), 1.59 (3H, s, H-10″), 1.54 (3H, s, H-8″); ^{13}C NMR (126 MHz, Chloroform-d_4) δ_C 202.89 (C-1′), 184.60 (C-1), 177.48 (C-3), 156.49 (C-4a), 153.75 (C-4b), 137.94 (C-9), 136.26 (C-3″), 134.19 (C-7), 131.36 (C-7″), 130.50 (C-5), 129.98 (C-8b), 125.10 (C-6), 124.13 (C-6″), 120.61 (C-2″), 119.96 (C-4), 116.40 (C-8), 109.07 (C-8a), 107.69 (C-2), 39.71 (C-4″), 29.98 (C-2′), 26.58 (C-5″), 25.59 (C-8″), 20.88 (C-1″), 17.64 (C-10″), 16.27 (C-9″); DIP: m/z 390.

(E)-4-acetyl-8-chloro-2-(3,7-dimethylocta-2,6-dien-1-yl)-3-hydroxy-1H-xanthen-1-one (3e): Dark maroon amorphous solid; mp 152–154 °C; ^{1}H NMR (500 MHz, Chloroform,- d4) δ_H 8.68 (1H, s, H-9), 7.50 (1H, t, J = 7.5 Hz, H-6), 7.34 (1H, d, J = 5.0 Hz, H-7), 7.27 (1H, d, J = 5.0 Hz, H-5), 5.20 (1H, t, J = 7.5 Hz, H-2″), 5.04 (1H, t, J = 5.0 Hz, H-6″), 3.36 (2H, d, J = 5.0 Hz, H-1″), 2.74 (3H, s, H-2′), 2.06 (2H, q, J = 6.7 Hz, H-5″), 1.98 (2H, t, J = 7.5 Hz, H-4″), 1.84 (3H, s, H-9″), 1.60 (3H, s, H-8″), 1.55 (3H, s, H-10″); ^{13}C NMR (126 MHz, Chloroform-d_4) δ_C 202.82 (C-1′), 185.05 (C-1), 177.27 (C-3), 155.53 (C-4a), 154.32 (C-4b), 136.49 (C-3″), 134.96 (C-8), 133.68 (C-9), 133.56 (C-6), 131.41 (C-7″), 125.60 (C-7), 124.95 (C-8b), 124.08 (C-6″), 120.31 (C-2″), 118.81 (C-4), 115.01 (C-5), 109.16 (C-8a), 108.49 (C-2), 39.69 (C-4″), 29.78 (C-2′), 26.56 (C-5″), 25.61 (C-8″), 20.91 (C-1″), 17.66 (C-10″), 16.28 (C-9″); DIP: m/z 424.

(E)-4-acetyl-6-(diethylamino)-2-(3,7-dimethylocta-2,6-dien-1-yl)-3-hydroxy-1H-xanthen-1-one (3f): Dark maroon amorphous solid; mp 129–131 °C; ^{1}H NMR (500 MHz, Chloroform,- d4) δ_H 8.41 (1H, s, H-9), 7.42 (1H, d, J = 5.0 Hz, H-8), 6.65 (1H, d, J = 5.0 Hz, H-7), 6.48 (1H, s, H-5), 5.23 (1H, t, J = 5.0 Hz, H-2″), 5.04 (1H, t, J = 5.0 Hz, H-6″), 3.48 (4H, q, J = 8.3 Hz, H-6a), 3.36 (2H, d, J = 5.0 Hz, H-1″), 2.73 (3H, s, H-2′), 2.05 (2H, q, J = 6.7 Hz, H-5″), 1.98 (2H, t, J = 7.5 Hz, H-4″), 1.86 (3H, s, H-9″), 1.58 (3H, s, H-8″), 1.54 (3H, s, H-10″), 1.27 (6H, t, J = 7.5 Hz, H-6b); ^{13}C NMR (126 MHz, Chloroform-d_4) δ_C 203.96 (C-1′), 180.84 (C-1), 177.01 (C-3), 157.94 (C-4a), 156.90 (C-4b), 153.22 (C-6), 150.67 (C-8a), 139.83 (C-9), 135.44 (C-3″), 132.22 (C-8), 131.31 (C-7″), 124.20 (C-6″), 121.59 (C-2″), 118.80 (C-4), 110.49 (C-7), 110.35 (C-8b), 104.33 (C-2), 96.37 (C-5), 45.21 (C-6a), 39.74 (C-4″), 29.68 (C-2′), 26.65 (C-5″), 25.58 (C-8″), 20.80 (C-1″), 17.64 (C-10″), 16.23 (C-9″), 12.52 (C-6b); DIP: m/z 461.

Conclusion

In summary, we have demonstrated that LiHMDS is able to catalyze the one-pot synthesis of prenylated or geranylated acylphloroglucinol-based xanthenones **3(a-f)** in yield ranging from 15.1 to 36.9 %. Plausible reaction mechanisms have also been described to explain the formation of these compounds. The acylphloroglucinol-based xanthenones could represent interesting new structures for the development of biologically active compounds as they contain two pharmacophores (acylphloroglucinol and xanthenones) possessing a broad spectrum of activities.

CRediT authorship contribution statement

Raaginie Tamil Segaran: Writing – original draft. **Chean Hui Ng:** Writing – review & editing, Supervision, Formal analysis. **Mohd Fadhlizil Fasihi Mohd Aluwi:** Writing – review & editing, Conceptualization. **Kok Wai Lam:** Writing – review & editing, Resources, Methodology, Data curation. **Khozirah Shaari:** Validation, Conceptualization. **Mazura Md Pisar:** Resources.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Supplementary data (in attach files) to this article can be found online.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.rechem.2024.101627.

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