

A new type of banana shape bifunctional monomer of ester chalcones

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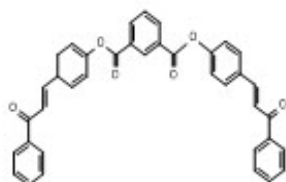
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GRAPHICAL ABSTRACT



ABSTRACT

A synthesis of a new type of chalcone analogue banana-shaped liquid crystals compounds is described. The approach involved the reaction of hydroxyl chalcones with isophthaloyl dichloride to form benzene-1,3-dicarbonyl-bis-1-(4-alkoxyphenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one (**7a-c** and **8a-c**) having two chalcone units *via* esterification reaction. The hydroxyl chalcones, which differ in the length of alkyl group, C_nH_{2n+1} , where $n=10, 12$ and 14 were synthesised *via* Claisen Schmidt condensation prior to the reaction with isophthaloyl dichloride. The reaction was a catalyst dependence. No sign of product was formed by employing NaH (60%). It was only occurred in the presence of NaH (95%) to afford banana shape bi-functional monomer of ester chalcone **7a-c** and **8a-c**. The synthesized compound was characterized by 1H and ^{13}C Nuclear Magnetic Resonance (NMR), and Fourier Transform Infrared (FTIR).

Keywords: Chalcones, banana shape liquid crystals, alkyl chains, ester

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1. INTRODUCTION

Liquid crystal technology has been widely applied in device technology. Both fluidity and crystalline properties of liquid crystal facilitate better control over alignment when applied to device. The advantages of liquid crystals for these applications such as faster switching times, a wider viewing angle, continuous gray level, improved transmittance of the clear state, approximately no threshold voltage and low power consumption [1].

Series of linear shape of mesogens based on chalcone has been studied for their liquid crystal behavior [2,3]. Chalcone derivatives were reported for excellent nonlinear optic property [4] and liquid crystal displays [5,6]. Chalcones has been used in promoting light transmittance [7] and crystallability [8].

In recent years, banana-shaped liquid crystals have been given much attention in various aspects such as fast polarization reorientation, ferroelectricity and antiferroelectricity, and small threshold voltage when applied to liquid crystal display mode [9]. Banana-shaped liquid crystals have also been identified as interesting compounds due to their unexpected electro-optical properties, polarity and chirality [10].

In this paper, we describe the preparation of a new type banana shape compound of benzene-1,3-dicarbonyl-bis-1-(4-alkoxyphenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one **5a-c** possessing alkyl chains of varying length from C_{10} to C_{14} which potentially used for liquid crystal studies.

2. EXPERIMENTAL

2.1 Materials and characterizations

4-hydroxybenzaldehyde, 4-hydroxyacetophenone, 1-bromoalkane, 4-hydroxychalcone and isophthaloyl dichloride were obtained from Merck Company and used as received. THF was dried over sodium and benzophenone. All other reagents and solvent were used as received. Infrared spectra were recorded on a Perkin Elmer 1605 Fourier Transform Infra-Red Spectrophotometer. 1H and ^{13}C NMR spectra were recorded using JEOL ECA 500 Spectrometer operating at 500 MHz for 1H and 125 MHz for ^{13}C with chemical shifts reported relative to $CDCl_3$ as standard reference.

2.2 Synthesis of 4-hydroxychalcones

Potassium hydroxide (KOH) (1.68 g, 30 mmol) in ethanol (90 mL, 95%) was stirred for 30 min. 4-hydroxyacetophenone (4.08 g, 30 mmol) and benzaldehyde (3.66 g, 30 mmol) were added to the solution mixture respectively. The reaction mixture was stirred at room temperature for 18 h. The mixture was cooled in an ice bath and acidified with hydrochloric acid (HCl) (12 M). The precipitate was filtered, washed and dried. Recrystallization was performed from hexane: ethanol (7:1) to afford compound **1** as light yellow solid, 2.02 g, 30 %, m.p. 175.0-

177.0°C. ν_{\max} (KBr / cm^{-1}) 3140 (-OH), 1646(C=O), 980(trans CH=CH) and 823(*para* disub benzene). δ_{H} (500 MHz, CDCl_3) 8.10 (d, J 8.6, 2H, H₁₋₃), 7.85 (d, J 15, 1H, H_{1-a}), 7.81-7.83 (m, 3H, H_{1-3'} & 4'), 7.75 (d, J 15, 1H, H_{1-b}), 7.43 (d, J 6.9, 2H, H₁₋₂), 6.96 (d, J 8.05, 2H, H_{1-2'}).

2.3 Synthesis of benzene-1,3-dicarbonyl-bis-4-chalcone (2)

Attempted Strategy 1

4-hydroxychalcone (0.11 g, 0.50 mmol) and tetrahydrofuran (THF) (30 mL) was added to sodium hydride (NaH) (0.01 g, 0.50 mmol, 60 %) and stirred for 1 h at room temperature. Benzene-1,3-dicarbonyl dichloride (0.05 g, 0.25 mmol) in THF (20 mL) was added to the mixture and stirred for 6 h at room temperature. The TLC showed no sign of product formed after prolonged stirring for 24 h. The reaction was continued no further.

Strategy 2

4-hydroxychalcone (0.11 g, 0.50 mmol) and THF (30 mL) was added to sodium hydride (NaH) (0.01 g, 0.50 mmol, 95 %) and stirred for 1 h at room temperature. Benzene-1,3-dicarbonyl dichloride (0.05 g, 0.25 mmol) in THF (20 mL) was added to the mixture. The mixture was refluxed for 8 h. The mixture was cooled to room temperature, filtered, and washed with distilled water and methanol. Dichloromethane (DCM) and distilled water were used to extract the organic layer. The organic layer was dried, filtered and concentrated *in vacuo*. The crude was purified from hexane: DCM (3:1) to give **2** as pale yellowish oil (0.10 g, 34.9 %). ν_{\max} (KBr / cm^{-1}) 1727(C=O ester), 1646(C=O carbonyl), 1222(C-O-C), 980(C=C), 823(*para* disub benzene). δ_{H} (500 MHz, $\text{DMSO}-D_6$) 6.88 (d, J 8.6, 4H, Ar-H₂'), 7.44 (d, 4H, Ar-H₃'), 7.57 (t, J 7.45, 1H, Ar-H₄'), 6.96 (d, 2H, J 15 Hz, 2 x *trans*-olefinic H), 7.73 (d, J 6.85, 1H, Ar-H₂''), 7.85-7.87(m, 6H, Ar-H₃' & 4''), 7.89(d, 2H, J 15 Hz, 2 x *trans*-olefinic H), 8.08(d, J 8.6, 2H, Ar-H₃), 8.49(s, 1H, Ar-H₁).

2.4 Synthesis of alkyloxyphenyl-ethanone (3a-3c)

General procedure

Bromoalkane (72 mmol), 4-hydroxyacetophenone (72 mmol), K_2CO_3 (72 mmol), and TBAI (6 mmol) in MEK (200 mL) were heated at reflux for 5 h. The mixture was filtered and cooled to room temperature. Water (30 mL) was added to the filtrate, and the layers separated. The aqueous layer was extracted with dichloromethane (2 x 30 mL). The combined layers were washed with water (2 x 20 mL), dried (MgSO_4), filtered, and concentrated *in vacuo*. The crude was recrystallized from ethanol to give **3a-3c**.

1-(4-Decyloxyphenyl)-ethanone (3a)

Bromodecane (12.40 mL, 60 mmol). Yield: 9.08 g, 66 %. FTIR and NMR data were consistent with the reported literature [11].

1-(4-Dodecyloxyphenyl)-ethanone (3b)

Bromododecane (14.38 mL, 60 mmol). Yield: 13.55 g, 89%. FTIR and NMR data were consistent with the reported literature [11].

1-(4-Tetradecyloxyphenyl)-ethanone (3c)

Bromotetradecane (14.38 mL, 60 mmol). Yield: 13.80 g, 83%. FTIR and NMR data were consistent with the reported literature [11].

2.5 Synthesis of (E)-1-[4-(alkyloxy)phenyl]-3-[4-hydroxyphenyl]prop-2-en-1-one (4a-4c)

General procedure

A mixture of 4-hydroxybenzaldehyde (12.5 mmol) and **3a-c** (12.5 mmol) in 35 mL of methanol was added under stirring to a solution of KOH (2.52 g) in methanol (10 mL). The mixture was heated at reflux for 10 h. The reaction was cooled to room temperature and acidified with cold diluted HCl (2 N). The resulting precipitate was filtered, washed, and dried. The crude was recrystallized from hexane:ethanol (7:1) to give **4a-4c**.

((E)-1-(4-decyloxyphenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one(4a)

Compound **4a** was obtained as yellow crystals (5.74 mL, 20 mmol). Yield (2.66 g, 35%). FTIR and NMR data were consistent with the reported literature [11].

(E)-1-[4-(Dodecyloxy)phenyl]-3-[4-hydroxyphenyl]prop-2-en-1-one (4b)

Compound **4b** was obtained as yellow crystals. Yield: 44%. FTIR and NMR data were consistent with the reported literature [11].

(E)-1-[4-(Tetradecyloxy)phenyl]-3-[4-hydroxyphenyl]prop-2-en-1-one (4c)

Compound **4c** was obtained as yellow crystals. Yield: 39%. FTIR and NMR data were consistent with the reported literature [11].

2.6 Synthesis of alkyloxybenzaldehyde (5a-5c)

General procedure

Bromoalkane (60 mmol), 4-hydroxybenzaldehyde (60 mmol), K_2CO_3 (60 mmol), and TBAI (6 mmol) in MEK (200 mL) were heated at reflux for 5 h. The mixture was filtered and cooled to room temperature. Water (30 mL) was added to the filtrate, and the layers separated. The aqueous layer was extracted with dichloromethane (2 x 30 mL). The combined layers were washed with water (2 x 20 mL), dried (MgSO_4), filtered, and concentrated *in vacuo*. The crude product was purified by column chromatography (eluting

with 1:20 ethyl acetate:petroleum ether). The same general procedure gave compounds **5a–c**.

4-Decyloxybenzaldehyde (5a)

Bromododecane (12.40 mL, 60 mmol). Yield: 8.69 g, 66%. FTIR and NMR data were consistent with the reported literature [12].

4-Dodecyloxybenzaldehyde (5b)

Bromododecane (14.38 mL, 60 mmol). Yield: 13.03 g, 90%. The FTIR and NMR data were consistent with the reported literature [12].

4-Tetradecyloxybenzaldehyde (5c)

Bromotetradecane (16.31 mL, 60 mmol). Yield: 11.78 g, 74%. The FTIR and NMR data were consistent with the reported literature [12].

2.7 Synthesis of (E)-3-(4-alkyloxyphenyl)-1-(4-hydroxyphenyl)prop-2-en-1-one (6a–6c)

General procedure

A mixture of 4-hydroxyacetophenone (30 mmol) and **6a–c** (30 mmol) in 90 mL of methanol was added under stirring to a solution of KOH (6.06 g, 108 mmol) in methanol (10 mL). The mixture was heated at reflux for 10 h. The reaction was cooled to room temperature and acidified with cold diluted HCl (2 N). The resulting precipitate was filtered, washed, and dried. The crude was recrystallized from hexane:ethanol (7:1) to give **6a–6c**.

(E)-3-(4-Decyloxyphenyl)-1-(4-hydroxyphenyl)prop-2-en-1-one (6a)

6a (5.25 mL, 20 mmol) Yield: 6.83 g, 54%. The FTIR and NMR data were consistent with the reported literature [12].

(E)-3-(4-Dodecyloxyphenyl)-1-(4-hydroxyphenyl)prop-2-en-1-one (6b)

6b (5.81 mL, 20 mmol). Yield: 8.32 g, 52%. The FTIR and NMR data were consistent with the reported literature [12].

(E)-3-(4-Tetradecyloxyphenyl)-1-(4-hydroxyphenyl)prop-2-en-1-one (6c)

6b (5.81 mL, 20 mmol). Yield: 8.32 g, 52%. The FTIR and NMR data were consistent with the reported literature [12].

2.8 Synthesis of bifunctional monomer of ester chalcones (7a–7c) and (8a–8c)

Benzene-1,3-dicarbonyl-bis-1-(4-decyloxyphenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one (7a)

NaH (0.03 g, 1.2 mmol, 95 %) was added into a solution of **4a** (0.38 g, 1.0 mmol) in THF (20 mL). The mixture was stirred for 2 h at room temperature. A solution of isophthaloyl dichloride (0.10 g, 0.50 mmol) in THF (10 mL) was added and the mixture was refluxed for 24 h. The mixture was cooled to room temperature. Water (30 mL)

was added to the filtrate, and the layers separated. The aqueous layer was extracted with dichloromethane (2 x 30 mL). The combined layers were washed with water (2 x 30 mL), dried (MgSO₄), filtered, and concentrated *in vacuo*. The crude was recrystallized from hexane: DCM (3:1) to afford compound **7a** (0.07 g, 15%) as sticky yellow oil. ν_{\max} (nujol mull/cm⁻¹): 2919, 2850, 1746, 1660, 1602, 1506, 1465, 1419, 1307, 1286, 1245, 1168, 1079, 1018, 989, 807. ¹H NMR (CDCl₃, 500 MHz), δ : 0.87 (t, *J* 7 Hz, 6H, 2 x CH₃), 1.27-1.84 (m, 32H, 16 x CH₂), 4.04 (t, *J* 7 Hz, 4H, 2 x OCH₂), 6.96-8.04 (m, 16H, Ar-H), 7.53 (d, 2H, *J* 15 Hz, 2 x *trans*-olefinic H), 7.71 (t, 1H, Ar-H), 7.79-7.82 (d, 2H, *J* 15 Hz, 2 x *trans*-olefinic H), 8.47-8.49 (d, 2H, *J* 9 Hz, Ar-H), 9.02 (s, 1H, Ar-H). The same general procedure to afford **7b–7c** with the scale (mL, mmol, [**4b–4c**]) and yields are shown below.

Benzene-1,3-dicarbonyl-bis-1-(4-dodecyloxyphenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one (7b)

4b (0.41 g, 1 mmol). Yield: (0.08 g, 17%) as sticky yellow oil. ν_{\max} (nujol mull/cm⁻¹): 2918, 2849, 1730, 1677, 1606, 1579, 1508, 1463, 1361, 1277, 1253, 1178, 1111, 1026, 964, 842. ¹H NMR (CDCl₃, 500 MHz), δ : 0.86 (t, *J* 7 Hz, 6H, 2 x CH₃), 1.38-1.79 (m, 40H, 20 x CH₂), 4.01 (t, 4H, *J* 7 Hz, 2 x OCH₂), 6.93-8.01 (m, 16H, Ar-H), 7.57 (d, 2H, *J* 15 Hz, 2 x *trans*-olefinic H), 7.69 (t, 1H, Ar-H), 7.78 (d, 2H, *J* 15 Hz, 2 x *trans*-olefinic H), 8.20-8.21 (d, 2H, Ar-H), 8.67 (s, 1H, Ar-H).

Benzene-1,3-dicarbonyl-bis-1-(4-tetradecyloxyphenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one (7c)

4c (0.44 g, 1.0 mmol). Yield: (0.09 g, 18%) as sticky yellow oil. ν_{\max} (nujol mull/cm⁻¹): 2918, 2850, 1743, 1651, 1604, 1546, 1509, 1255, 1225, 1169, 1036, 980, 921. ¹H NMR (CDCl₃, 500 MHz), δ : 0.84 (t, *J* 7 Hz, 6H, 2 x CH₃), 1.24-1.81 (m, 48H, 24 x CH₂), 4.04 (t, *J* 7 Hz, 4H, 2 x OCH₂), 6.96-8.04 (m, 16H, Ar-H), 7.53 (d, 2H, *J* 15 Hz, 2 x *trans*-olefinic H), 7.70 (t, 1H, Ar-H), 7.80 (d, 2H, *J* 15 Hz, 2 x *trans*-olefinic H), 8.37 (d, 2H, Ar-H), 8.85 (s, 1H, Ar-H).

Benzene-1,3-dicarbonyl-bis-3-(4-decyloxyphenyl)-1-(hydroxyphenyl)prop-2-ene-1-one (8a)

6a (0.38g, 1.0 mmol). Yield: (0.10 g, 11.6 %) as a pale yellowish oil ν_{\max} (KBr / cm⁻¹) 2921(-CH₂), 2851(-CH₃), 1733(C=O ester), 1644(C=O carbonyl), 1222(C-O-C), 987(C=C), 825(*para* disub benzene). δ_{H} (500 MHz, CDCl₃) 0.86 (t, 6H, H₃-10^{''}), 1.26-1.78 (m, 32H, H₂-2^{''}-9^{''}), 3.99 (t, 4H, H₂-1^{''}), 6.89-6.96 (m, 8H, H₁-2^{''}), 7.36-7.37 (t, 1H, H₁-4), 7.42 (d, *J* 15.45, 2H, H₁-b), 7.57-7.60 (m, 4H, H₁-3^{''}), 7.78(d, *J* 16.60, 2H, H₁-a), 8.09-8.11 (d, 2H, H₁-3), 8.31, 8.37(dd, 8H, H₁-3^{''} & 2^{''}), 8.85(s, 1H, H₁-1).

Benzene-1,3-dicarbonyl-bis-3-(4-dodecyloxyphenyl)-1-(hydroxyphenyl)prop-2-ene-1-one (8b)

6b (0.41 g, 1.0 mmol). Yield: (0.21 g, 23.2 %) as pale yellowish oil. ν_{\max} (KBr / cm⁻¹) 2924(-CH₂), 2853(-CH₃), 1694(C=O ester), 1661(C=O carbonyl), 1278(C-O-C), 940(C=C), 763(*para* disub benzene). (500 MHz, CDCl₃)

0.86 (t, 6H, H₃-12''), 1.26-1.71 (m, 40H, H₂-2''-11''), 3.97 (t, 4H, H₂-1''), 6.88-6.92 (m, 4H, H₁-2''), 7.39-7.40 (t, 1H, H₁-4), 7.45 (d, *J* 15.45, 1H, H₁-b), 7.55-7.57 (m, 4H, H₁-2), 7.77 (d, *J* 16.60, 2H, H₁-a), 7.87-7.90 (d, 4H, H₁-3), 7.94, 7.98 (dd, 8H, H₁-3'' & 2''), 8.07 (s, 1H, H₁-1).

Benzene-1,3-dicarbonyl-bis-3-(4-tetradecyloxyphenyl)-1-(hydroxyphenyl) prop-2-ene-1-one (8c)

6c (0.44 g, 1 mmol). Yield: (0.20 g, 21.5%) as a pale yellowish oil. ν_{\max} (KBr / cm^{-1}) 2917(-CH₂), 2849(-CH₃), 1729(C=O ester), 1655(C=O carbonyl), 1259(C-O-C), 1021(C=C), 799(*para* disub benzene). δ_{H} (500 MHz, CDCl₃) 0.86 (t, 6H, H₃-14''), 1.24-1.78 (m, 48H, H₂-2''-13''), 3.99 (t, 4H, H₂-1''), 6.89-6.92 (m, 4H, H₁-2''), 7.35-7.37 (t, 1H, H₁-4), 7.37 (d, *J* 15.45, 2H, H₁-b), 7.57-7.61 (m, 4H, H₁-3''), 7.78 (d, *J* 15.45, 2H, H₁-a), 8.09-8.11 (d, 4H, H₁-3), 8.31, 8.38 (dd, 8H, H₁-3'' & 2''), 8.86 (s, 1H, H₁-1).

3. RESULTS & DISCUSSION

Initial study on the preparation of **2** was performed following typical procedure, where earlier synthesized 4-hydroxychalcone, **1**, in tetrahydrofuran (THF) was added into sodium hydride (NaH) (60 %) and stirred for 1 h at room temperature. Benzene-1,3-dicarbonyl dichloride in THF was added to the mixture and stirred at room temperature. TLC analysis, however, showed no sign of product formed after 24 h.

Several attempts were made with slight modifications on the reaction condition (solvent, time, temperature), however no evidence for formation of the desired product.

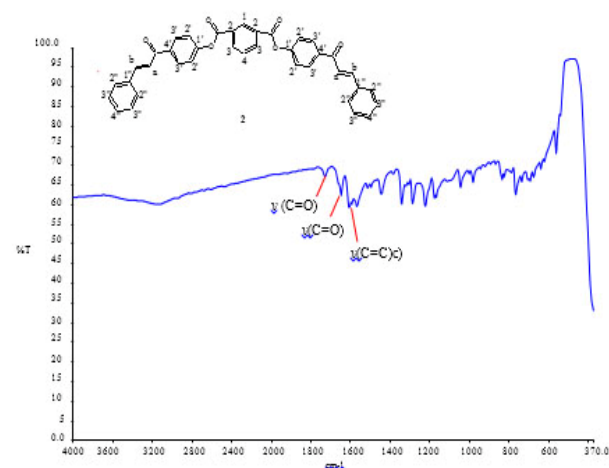
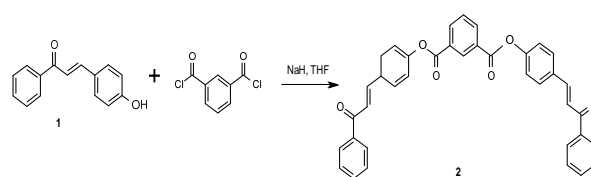


Fig. 1 IR Spectrum of compound (**2**) using NaH (95%)

NaH (95%) was chosen over NaH (60%) as a catalyst and successfully afforded **2** with 50% yield. The preparation of **2** is depicted in Scheme 1.

The chemical structure of **2** was found to be consistent with FT-IR and ¹H NMR spectroscopic methods and showed the peaks corresponded to the structures.

The IR spectrum of **2** is shown in Fig. 1. The IR spectrum showed the disappearance of -OH peak at 3140 cm^{-1} and the appearance of absorption band at 1727 cm^{-1} , which were attributed to the formation of ester bond. The structure of **2** were further confirmed by ¹H NMR spectrum (Fig. 2), which showed peaks attributed to *trans* vinylic proton at δ 6.96 and 7.89, with a coupling constant, *J*ab 15 Hz. New peak attributed to center aromatic H₁ appeared as singlet at δ 8.49. The peaks corresponded to H_{3&4} were appeared at δ 7.57-8.08. This proton resonates more downfield from other resonances due to the attachment with two chalcones moieties.



Scheme 1 Model study on the preparation of chalcone ester **2**

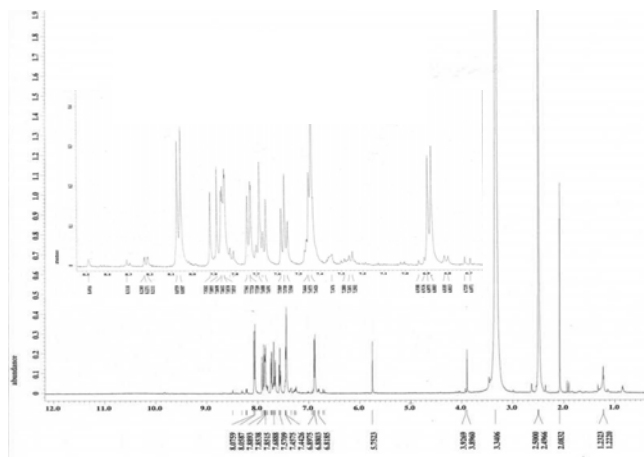


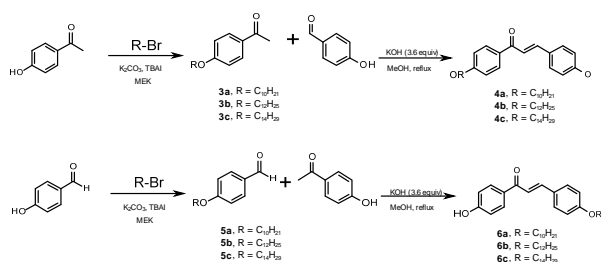
Fig. 2 IR Spectrum of compound (**2**) using NaH (95%)

Several methods have been employed in the synthesis of chalcones due to ease of chalcone structure itself to be substituted [13, 14]. A series of chalcone derivatives

(*E*)-1-[4-(alkyloxy)phenyl]-3-[4-hydroxyphenyl] prop-2-en-1-one (**4a-4c**) and (*E*)-3-(4-alkyloxyphenyl)-1-(4-hydroxyphenyl)prop-2-en-1-one (**6a-6c**) were prepared *via* Claisen-Schmidt condensation by the route depicted in Scheme 2.

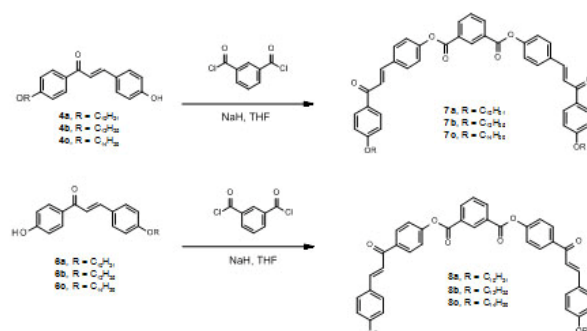
Compound **3a-c** and **5a-c** were reacted with 4-hydroxybenzaldehyde and 4-hydroxyacetophenone, respectively, under refluxing methanol to afford hydroxylated chalcones **4a-c** and **6a-c** as yellow crystals. IR

spectra showed the presence of ν_{OH} at 3262 cm^{-1} , while ν_{CH_2} and ν_{CH_3} bands were observed at 2922 and 2851 cm^{-1} respectively.



Scheme 2 Synthesis of chalcone derivatives **4a-c** and **6a-c**

resonance as a triplet and a singlet at δ 7.37 and 8.85 respectively.



Scheme 3 Synthesis of banana-shaped chalcones ester **7a-c** and **8a-c**

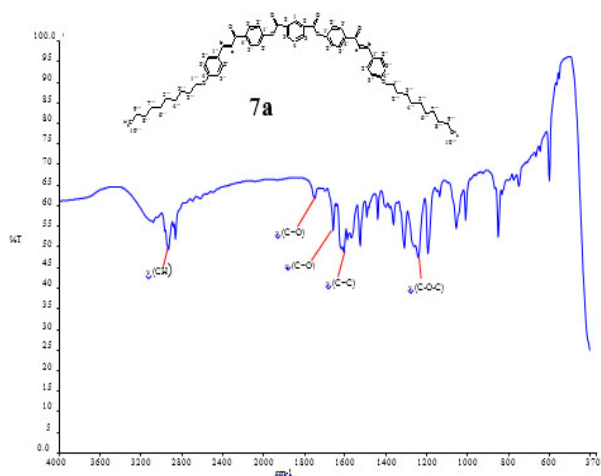


Fig. 3 IR Spectrum of **7a**

The structures of compound **4a-c** and **6a-c** were further verified using ^1H NMR and ^{13}C NMR analysis. ^1H NMR spectra showed the presence of resonances attributed to CH_3 and CH_2 at δ 0.87 and 1.26-1.83. The *trans* vinylic proton α and β centered at δ 7.42 and 7.74 as two doublets peak with *J*ab 15.45 Hz. The ^{13}C NMR spectra showed the peak attributed to $\text{C}=\text{O}$ at δ 189.1 and $-\text{COH}$ at δ 162.9.

Compounds **7a-c** and **8a-c** with two chalcone units were finally prepared by the reaction of **4a-c** and **6a-c** with isophthaloyl dichloride, respectively. The reactions were performed in the presence of NaH (95%) under refluxing THF to afford **7a-c** and **8a-c** as viscous yellow oil. The preparation of **7a-c** and **8a-c** is depicted in Scheme 3.

The IR spectrum of **7a** (Fig 3) showed the presence of $\nu_{\text{C}=\text{O}}$ for ester at 1746 cm^{-1} and $\nu_{\text{C}-\text{O}-\text{C}}$ attributed to alkyl aryl ether at 1245 cm^{-1} . The ^1H NMR spectrum of **7a** (Fig. 4) showed peaks attributed to CH_3 and CH_2 at δ 0.85 and 1.24-1.63, while peak attributed to OCH_2 at δ 3.97. The *trans* vinylic proton α and β were assigned at δ 7.60 and 7.78 with *J*ab 15.45 Hz. The attachment of two chalcone units onto the benzene ring was supported by the appearance of a

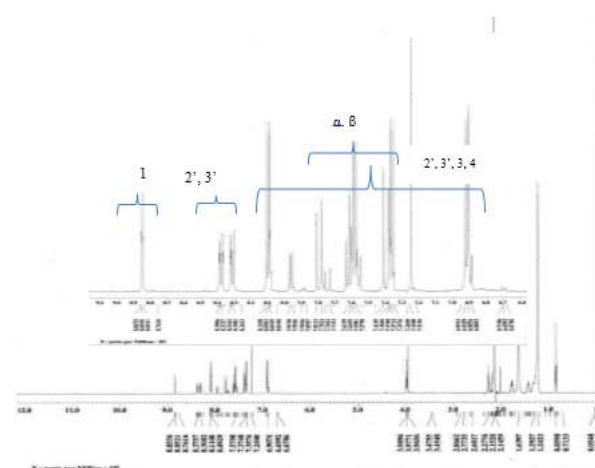


Fig. 4 ^1H NMR spectrum of **7a**

4. CONCLUSION

We have synthesised new homologues series of banana-shaped compounds **7a-c** and **8a-c** comprising bifunctional chalcone units in the presence of NaH (95%). The low yield of **7a-c** and **8a-c** might due to the steric effect of hydroxylated chalcone **4a-c** bearing long alkyl chains. Compounds **7a-c** and **8a-c** could be useful in the study of banana shaped liquid crystals properties.

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