

SYNTHESIS OF HETEROCYCLIC PYRIDINE-BASED CHALCONES
WITH DIMERIC STRUCTUREPui-Mun Lai¹, Sie-Tiong Ha¹, ✉<https://doi.org/10.23939/chcht16.01.001>

Abstract. Three new heterocyclic chalcones containing pyridine moiety were synthesized and their chemical structures were determined *via* IR, ¹H NMR and ¹³C NMR spectroscopy. General name of these compounds are α,ω -bis{3-(pyridin-3-yl)-1-(phenyl-4-oxy)prop-2-en-1-one}alkanes. The chalcones are dimers having a symmetrical structure and they can be differed by the alkyl spacer length (C_nH_{2n}, where n = 8, 10 or 12). Differential scanning calorimetry (DSC) technique was employed to study their phase transition behaviors. DSC thermograms displayed direct isotropization and recrystallization during heating and cooling processes, respectively. The crystal phase turned into isotropic phase without exhibiting any mesophase. Influence of structure-liquid crystalline property relationships of the symmetrical dimers was examined in order to explain the reasons for the non-liquid crystalline properties in the current chalcones.

Keywords: heterocycle, pyridine, chalcone, dimer, synthesis.

1. Introduction

Chalcone is a compound comprising two aromatic rings connected by an unsaturated α,β -ketone, with various substituents on the two aromatic rings. Chalcone occurs naturally in most of the plants and is an intermediate precursor of flavonoids and isoflavonoids.¹ It was reported to have comprehensive applications in the fields of biology and biochemistry^{2,3} as anti-tumor,^{4,5} anti-inflammatory⁶⁻⁸ and anti-malaria⁹ agent. In addition, it is also reported for its photochemical and photophysical properties, including being used as photo-alignment and photo-crosslinking unit in polymerization process,¹⁰ fluorescent dyes, light-emitting diodes (LEDs), *etc.*¹¹

Liquid crystals with a chalcone central linkage are relatively rare. In the literature there are several reports of

mesomorphic compounds having chalcone linkage. However, many years ago Chudgar and Shah¹² and Yeap *et al.*¹³ reported homologous series containing ester-chalcone linkages. Thaker and co-workers¹⁴ have also studied mesomorphic compounds containing Schiff base-chalcone linkage. Ha and Low¹⁵ had studied the properties of chalcone with different terminal polar substituents (F, Cl, Br and NO₂). The previous studies were mainly on the aromatic-based chalcone and having calamitic (non-dimeric) structure. In our attempt to incorporate heterocyclic ring into chalcone skeleton, we have recently synthesized the chalcone with heterocyclic pyridine-based liquid crystal, 4-[3-(pyridin-4-yl)prop-2-enoyl]phenyl 4-alkyloxybenzoates.¹⁶ In this continuation work, our aim is to synthesize the dimers of heterocyclic pyridine-based chalcone. Here, we report three analogues of symmetrical dimers, α,ω -bis{3-(pyridin-3-yl)-1-(phenyl-4-oxy)prop-2-en-1-one}alkanes (Fig. 1).

2. Experimental

2.1. Materials and Methods

All reagents used were purchased from commercial suppliers without further purification. Intermediate, 1-(4-hydroxyphenyl)-3-(pyridin-3-yl)prop-2-en-1-one was prepared according to a previously reported method.¹⁵ The synthetic route for the title compounds is illustrated in Fig. 1. Compound purity was confirmed using TLC on silica gel 60 F254. Melting point determination was done using the Stuart SMP10 digital melting point apparatus and was recorded in °C. Electron ionization mass spectrum was recorded by a Finnigan MAT95XL-T mass spectrometer operating at 70 eV ionizing energy. FT-IR spectra were acquired on Perkin Elmer 2000-FTIR spectrophotometer in the frequency range of 4000–400 cm⁻¹ with samples prepared as KBr pellets. NMR spectra were recorded in CDCl₃ by utilizing JEOL 400M Hz NMR Spectrometer with TMS as internal standard. The phase transition

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temperatures were measured by Mettler Toledo DSC823 Differential Scanning Calorimeter (DSC) at the scanning rate of 10°C/min.

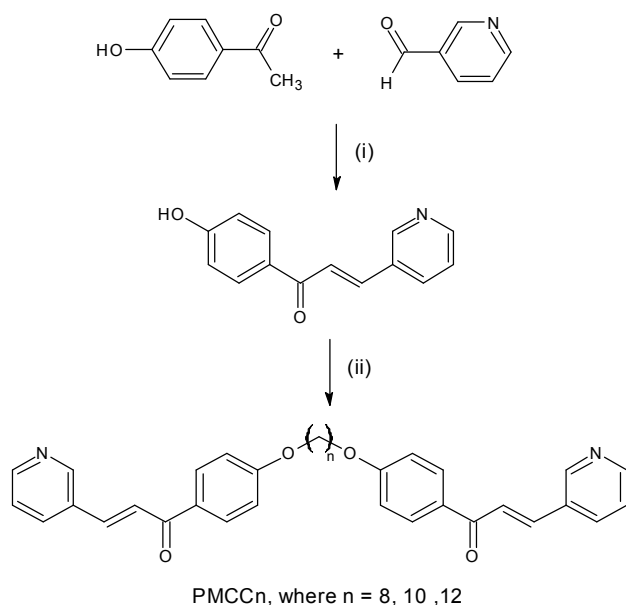


Fig. 1. Synthetic route towards formation of the title compounds. Reagents and conditions: (i) EtOH/H₂O (1:1), KOH, stirred at r.t., 24 h, neutralized with 2M HCl; (ii) Dibromoalkane (C_nH_{2n}Br₂, n = 8, 10 and 12), K₂CO₃, KI, acetone, reflux 24 h

2.2. Synthesis of α,ω -bis{3-(pyridin-3-yl)-1-(phenyl-4-oxy)prop-2-en-1-one} alkanes

1-(4-Hydroxyphenyl)-3-(pyridin-4-yl)prop-2-en-1-one (2 mmol) and dibromoalkane (1 mmol) were added into 30 mL of acetone. Then, the mixture was stirred and heated under reflux for 24 h in the presence of potassium carbonate and potassium iodide. Upon completion of the reaction, the mixture was then cooled to room temperature and followed by addition of distilled water (80 mL). The resulting precipitate was filtered and washed with water. The final products were recrystallized with ethanol until constant transition temperature was obtained. Melting points of the products are given as follow: PMCC8 (168.2 °C), PMCC10 (157.2 °C) and PMCC12 (153.0 °C). Percentage yields of the products are listed as follow: PMCC8 (50 %), PMCC10 (56 %) and PMCC12 (60 %).

The IR and NMR (¹H and ¹³C) data of the representative compound, PMCC12 are summarized as below.

IR (KBr) ν_{\max} cm⁻¹: 2937, 2849 (C–H aliphatic), 1659 (C=O), 1637 (C=C olefinic), 1604, 1474 (C=C aromatic), 1256 (C–O); ¹H NMR (400 MHz, CDCl₃, δ ppm): 1.26–1.38 {m, 12H, 2 x –O(CH₂)₃(CH₂)₃}, 1.46 {p, *J* = 6.9 Hz, 4H, 2 x –O(CH₂)(CH₂)(CH₂)}, 1.80 {p,

J = 7.1 Hz, 4H, 2 x –O(CH₂)(CH₂)(CH₂)}, 4.03 (t, *J* = 6.4 Hz, 4H, 2 x –OCH₂), 7.60 (d, *J* = 15.6 Hz, 1H, olefinic-H), 7.76 (d, *J* = 16.0 Hz, 1H, olefinic-H), 6.96 (d, *J* = 8.7 Hz, 4H, Ar-H), 8.02 (d, *J* = 8.0 Hz, 4H, Ar-H), 7.34 (dd, *J* = 7.8 Hz, 4.6 Hz, 2H, pyridine protons), 7.93 (dt, *J* = 7.8 Hz, 1.8 Hz, 2H, pyridine protons), 8.60 (dd, *J* = 4.6 Hz, 1.8 Hz, 2H, pyridine protons), 8.84 (d, *J* = 1.8 Hz, 2H, pyridine protons); ¹³C NMR (100 MHz, CDCl₃ δ ppm): 26.07, 29.18, 29.43, 29.63 for {–O(CH₂)(CH₂)₅}, 68.42 for (–OCH₂), 114.50, 123.87, 130.45, 131.02, 134.72, 149.90, 150.94, 163.44 for aromatic and pyridine carbons, 123.82, 140.07 for olefinic carbons, 188.08 (C=O keto).

3. Results and Discussion

3.1. FT-IR, ¹H NMR and ¹³C NMR Spectra

The heterocyclic chalcones were synthesized in two-steps reaction. First, the chalcone skeleton was obtained through base-catalyzed Claisen-Schmidt condensation method. Second, the formation of alkyl spacer was through Williamson etherification. These reactions are summarized in Fig. 1.

The chalcones were obtained as yellow powder. The chemical structures of the synthesized chalcones were confirmed using FTIR, ¹H NMR and ¹³C NMR.

The FTIR spectrum of heterocyclic chalcone gave absorptions at 1659 and 1637 cm⁻¹, corresponding to the C=O and C=C functional groups that are characteristic of the α,β -unsaturated ketone moiety. Strong absorption bands emerged at 2937 and 2849 cm⁻¹ confirmed the presence of aliphatic C–H in alkyl chain. A sharp band at 1256 cm⁻¹ can be ascribed to C–O bonds of aromatic ether linking group. Absorption bands appeared at 1604 and 1474 cm⁻¹ designate the aromatic C=C linking group.

The ¹H NMR spectrum is shown in Fig. 2. The successful completion of a base-catalyzed Claisen-Schmidt condensation reaction in the synthesis of chalcone results in an enone system known as the α,β -unsaturated ketone moiety. This was proven by the characteristic pair of doublets at δ = 7.60 and 7.76 ppm due to vicinal coupling between H _{α} and H _{β} with a coupling constant of *J* = 16 Hz pointing to a *trans* configuration. Triplet signal observed at δ = 4.03 ppm, is attributed to the methylene protons attached to oxygen atom (–OCH₂). The remaining methylene protons of the long chain appeared as multiplet, pentet and pentet signals at the respective chemicals shifts, δ = 1.26–1.38, 1.46, and 1.80 ppm. Two distinct doublets detected at δ = 6.96 and

8.02 ppm which each doublet integrated as two protons belong to the resonance of the *para*-substituted aromatic protons. Both signals are coupled with each other with a

coupling constants of 8 Hz. Signals attributed to the four pyridine protons were observed at $\delta = 7.34, 7.93, 8.60$ and 8.84 ppm.

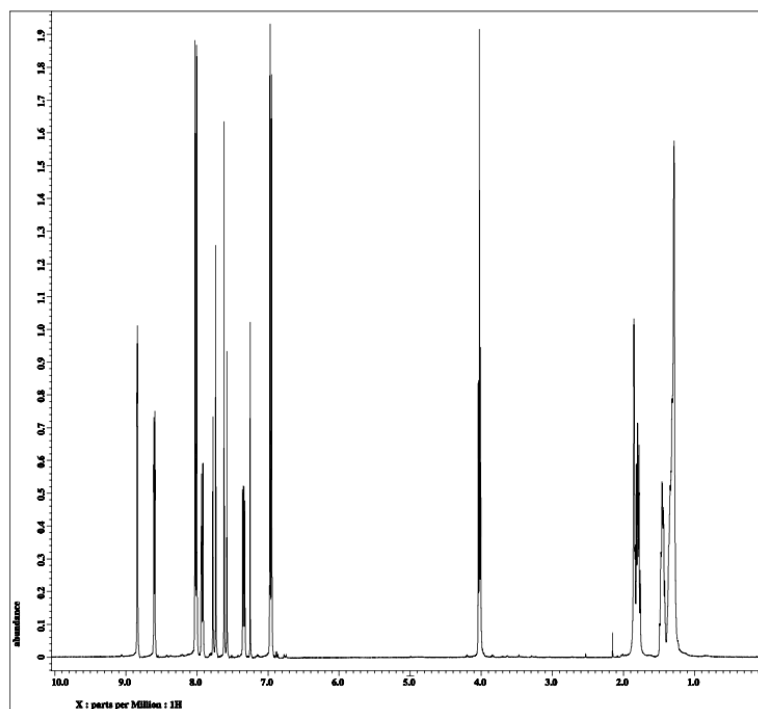


Fig. 2. ^1H NMR spectrum of symmetrical dimer, PMCC12

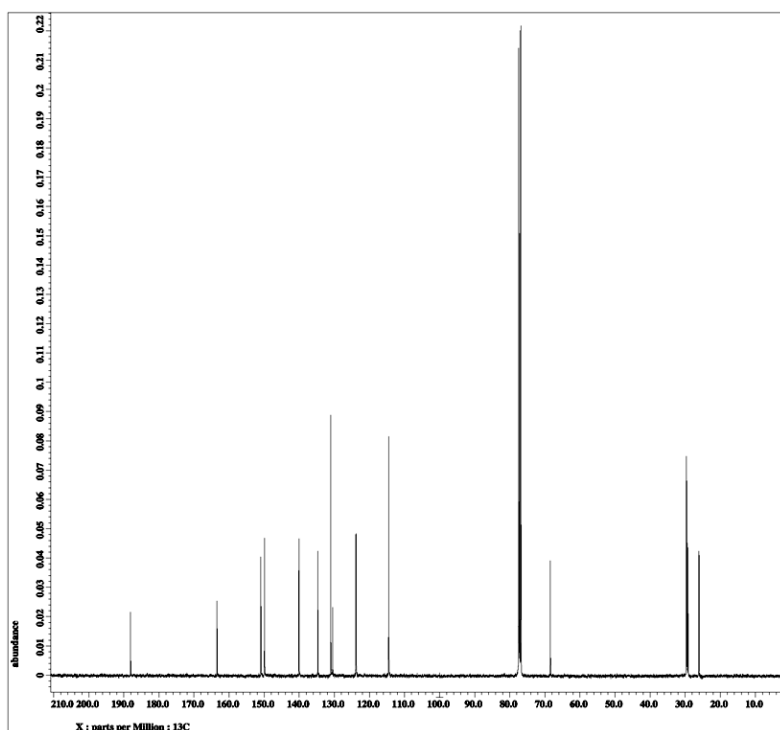


Fig. 3. ^{13}C NMR spectrum of symmetrical dimer, PMCC12

The ^{13}C NMR spectrum (Fig. 3) displayed a signal at the most downfield region of the spectrum, $\delta = 188.08$ ppm corresponding to the presence of the carbonyl carbon of their α,β -unsaturated ketone moiety. Olefinic carbons appeared at $\delta = 123.82$ and 140.07 ppm confirmed the presence of enone linking unit. A peak at $\delta = 68.42$ ppm is attributed to the methylene carbons attached to oxygen atom ($-\text{OCH}_2-$). The remaining methylene carbons were resonated at $\delta = 26.07, 29.18, 29.43,$ and 29.63 ppm. ^{13}C signals at $\delta = 114.50-$ 163.44 ppm is assigned to aromatic and pyridine carbons.

3.2. Phase Transition Behaviour of Chalcone Derivatives

The thermal properties of new chalcones were studied using DSC. All homologous members were non-mesogenic compounds. The representative DSC thermogram of PMCC12 (Fig. 4) shows single endotherm and exotherm, respectively, during both heating and cooling cycles. This observation indicates direct melting of the crystal phase to the isotropic liquid phase (Cr-to-I) and vice versa (I-to-Cr). Therefore, no liquid crystal texture was observed during heating and cooling processes. Other members, PMMCn (where $n = 8, 10$) showed the similar characteristics as those discussed for PMMC12.

3.3. Effect of Alkyl Spacer Length on Transition Temperatures

A further inspection on DSC data (Table 1) also illustrates the evolution of the Cr-to-I transition with the lengthening of spacers. This study revealed that the melting temperatures illustrate a gradual descending trend ($168.2\text{ }^\circ\text{C} \rightarrow 157.2\text{ }^\circ\text{C} \rightarrow 153.0\text{ }^\circ\text{C}$) with the increase in the length of alkyl spacer from $n = 8$ to $n = 12$. This phenomenon can be explained in terms of dilution of the core units which was influenced by the increase in the flexibility of spacer.^{17,18}

Table 1. Transition temperatures, enthalpy changes and phase transitions of symmetrical dimers

Compound	Transition temperatures, $^\circ\text{C}$ (ΔH , kJ mol^{-1})	
	Heating	Cooling
PMCC8	Cr 168.2 (40.6) I	Cr 137.5 (38.7) I
PMCC10	Cr 157.2 (57.4) I	Cr 135.3 (57.2) I
PMCC12	Cr 153.0 (50.2) I	Cr 140.3 (55.3) I

3.4. Structure-Mesomorphic Property Relationships

The mesomorphic behavior of organic compounds is sensitive to its molecular architecture; a slight alteration

of molecular geometry brings about considerable change in its mesomorphic behaviour.¹⁹ Investigation of these factors by experimental or theoretical means should hopefully lead to a better understanding of structure-property relationships on the synthesized compounds in liquid crystals.

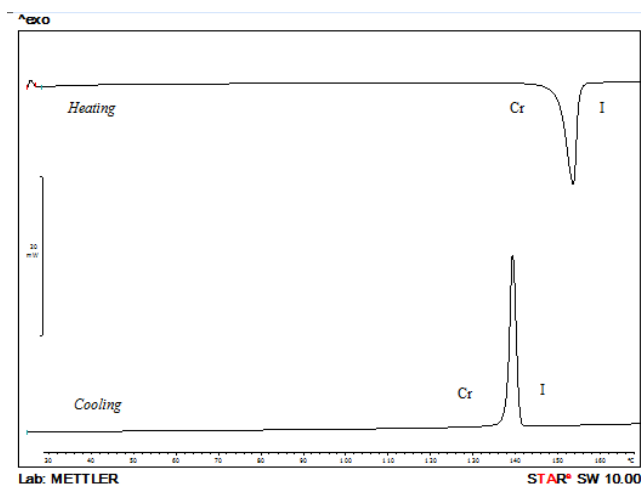


Fig. 4. DSC thermogram of symmetrical dimer, PMCC12

In order to relate the transitional properties with the chemical constitution of molecules, the present dimers are compared with the related liquid crystal dimers. The general molecular structure for these dimers labeled as nBAm,^{20,21} 10BABZ¹⁸ and nBABTP²² and their phase sequences and transition temperatures are shown in Table 2.

Terminal groups showed strong influence on the mesomorphic properties of a molecule as presented in Table 2. It can be seen that the dimers with terminal alkyl chain, nBAm exhibited mesomorphic properties and dimers without terminal group, PMCCm are non-mesogens. Terminal chains bring flexibility into the molecule which is required to stabilize mesophases.²³ Dimers with short terminal chain, 12BA6 and 12BA7 are nematogens and longer terminal chain promotes smectic phase in 12BA10.

Presence of pyridine ring in PMCCn is not the factor that prevents the current dimers from exhibiting mesophases. This can be evidenced from the case of 10BABZ and 10BATP wherein these compounds having heterocyclic benzothiazole and thiophene, respectively, are mesogens. However, it needs to complement with terminal polar chloro substituent like in the case of 10BABZ or having a longer molecular core through additional aromatic ring in 10BATP. Increasing the core length by inserting short unsaturated linkages (COO, ester) between phenyl and thiophene rings enhances the clearing temperature of 10BATP.

Table 2. Comparison of mesomorphic behavior of PMCCn with structurally related dimers

Compound	Structure	Phase Transition, °C
PMCC10 [Current work]		Cr 157.2 I
nBAm ^{20,21}		12BA6: Cr 129.1 N 132.2 I 12BA7: Cr 127.0 N 133.0 I 12BA10: Cr 118.2 SmG 131.3 I
10BABZ ¹⁸		Cr 144.2 SmA 160.7 N 173.2 I
nBATP ²²		10BATP: Cr 149.6 N 207.6 I

4. Conclusions

In this paper, we report the synthesis of new heterocyclic chalcones, α,ω -bis{3-(pyridin-3-yl)-1-(phenyl-4-oxy)prop-2-en-1-one}alkanes. The spectral results matched with the molecular structures of the new chalcones. However, DSC analysis found that the chalcones did not exhibit liquid crystal properties. The influence of dimer spacer length, terminal chain and core rings on mesomorphic properties was discussed and compared with the reported dimer liquid crystals. The reason of the chalcone dimers for not exhibiting liquid crystal phase could be due the lack of polar group at the terminal position and lack of extra aromatic ring as the core system in a liquid crystal molecule. Therefore, the current dimers need to be modified in order to generate liquid crystal phase. These findings can serve as reference for future researches of heterocyclic pyridine chalcone-based dimer liquid crystals. In addition, the presence of the pyridine group makes this system a suitable component (or intermediate) for building charge transfer-based liquid crystals with an appropriate choice of acceptor molecules in future studies.

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СИНТЕЗ ГЕТЕРОЦИКЛІЧНИХ ХАЛКОНІВ ДИМЕРНОЇ СТРУКТУРИ НА ОСНОВІ ПІРИДИНУ

Анотація. Синтезовано три нові гетероциклічні халкони, що містять піридиновий фрагмент. За допомогою ІЧ-спектроскопії, ^1H ЯМР та ^{13}C ЯМР визначено їх хімічну структуру, і встановлено, що вони відносяться до сполук із загальною назвою α,ω -біс{3-(піридин-3-іл)-1-(феніл-4-окси)проп-2-ен-1-он}алкани. Халкони – це димери, що мають симетричну структуру, і відрізняються довжиною алкільного проміжку (C_nH_{2n} , де $n = 8, 10$ або 12). Для визначення фазових переходів синтезованих сполук застосовано метод диференціальної скануючої калориметрії (DSC). Термограми DSC відображають пряму ізотропізацію та перекристалізацію під час процесів нагрівання та охолодження відповідно. Кристалічна фаза перетворюється в ізотропну фазу, не виявляючи жодної мезофази. Вивчено вплив структурно-рідких кристалічних властивостей симетричних димерів, щоб пояснити причини нерідких кристалічних властивостей у діючих халконах.

Ключові слова: гетероциклі, піридин, халкон, димер, синтез.