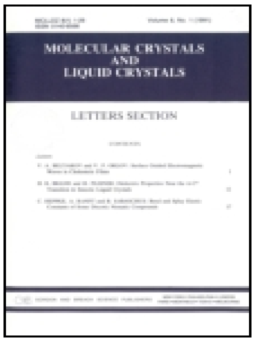

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Mesomorphic dependence on molecular rigidity by central bridge and homo–hetero ring systems

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ABSTRACT

A novel homologous series of thermotropic derivatives containing a heterocyclic ring was synthesized and studied with a view to understanding and establishing a relation between liquid crystal (LC) properties and the molecular structure of chalconyl derivative in general. The series comprises 12 homologues, of which smectogenic LC property commences from the decyloxy (C_{10}) homologue and continues for C_{12}, C_{14}, and C_{16} homologues. Homologues C_1 to C_8 are non-liquid crystals. Exhibition of nematogenic mesophase is totally absent. Transition and melting temperatures were determined by an optical polarizing microscope equipped with a heating stage. Spectral and analytical data confirmed the molecular structures of homologues. The textures of smectic mesophase are A or C, and are determined by a miscibility method. The average thermal stability of the smectic phase is 113.57°C. The mesogenic phase length ranges between 4.1°C and 11.1°C. Thus, it is a middle-ordered melting-type series of shorter range of liquid crystallinity with absence of nematogenic character. Some LC properties of the present series are compared with a structurally similar known homologous series.

KEYWORDS
Liquid crystal; mesomorphism; nematic; smectic, thermotropic

Introduction

The liquid crystalline (LC) state was discovered in 1888 [1], and the thermotropic type is useful for liquid crystal devices (LCDs) and other purposes. Moreover, the bioactive lyotropic type of LC is useful in pharmaceutical and medicinal fields to cure some diseases [2–8]. Therefore, the present investigation is planned with a view to understanding and establishing the effects of molecular structure on LC behavior by synthesizing a novel homologous series of chalconyl derivatives [9–11]. Chalconyl derivatives being biologically active molecules can be studied lyotropically to measure their pharmaceutical and medicinal potency. The present investigation will be limited to the synthesis of novel LC substances and the characterization of some LC properties, and a comparison with a structurally similar known series. Thus, the present investigation will be useful to many research groups working on LC (thermotropic and lyotropic) with various aims and objectives. Numerous homologous series are reported [12–15] to date. Results of our research reported here will be discussed and interpreted in terms of molecular rigidity and flexibility [16–19].

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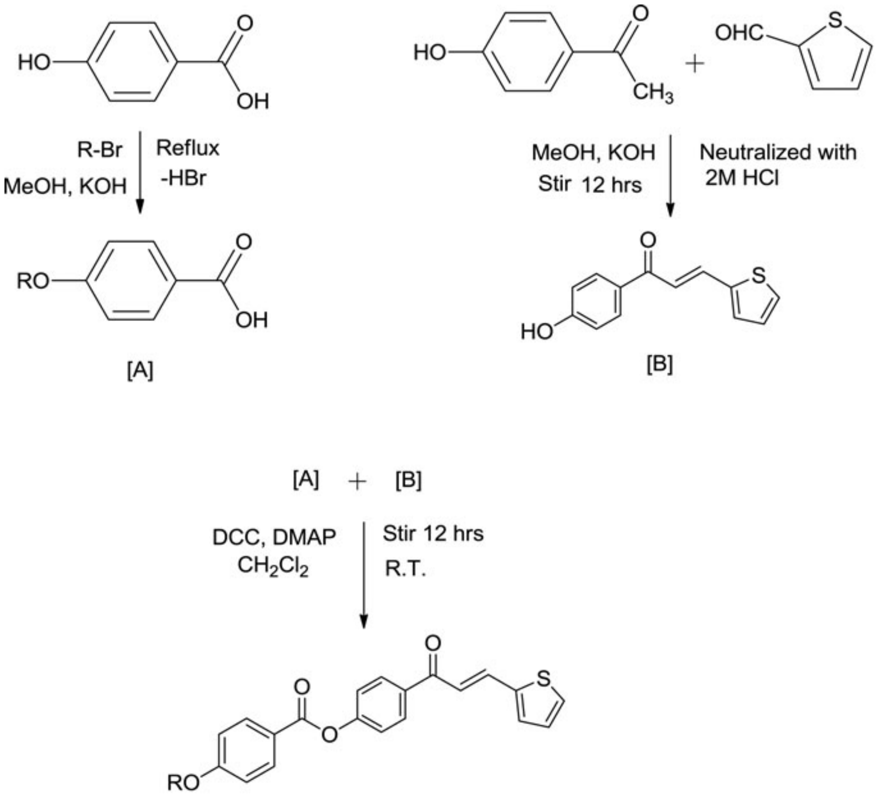
Experimental

Synthesis

4-Hydroxy benzoic acid was alkylated by suitable alkylating agents by the modified method of Dave and Vora [20]. 3-(2-Thiophenyl)-1-(4-hydroxyphenyl) 2-propen-1-one (B) was prepared by an established method [21]; m.p.: 170–172°C, yield: 64.8%. Coupling of compounds A and B is done by the Steglich esterification to yield α-4(4'-n-alkoxy benzoyloxy) benzoyl-β-(2"-thiophenyl) ethylene [22].

The synthetic route to the novel homologous series of ethylene derivatives is shown in Scheme 1.



Where, R= C_nH_{2n+1} and n = 1 to 8 and 10,12,14,16

α-4(4'-n-Alkoxy benzoyloxy) benzoyl-β-(2"-thiophenyl) ethylene

Scheme 1. Synthetic route to the novel series.

Characterization

Representative members of a novel series were characterized by elemental analysis (Table 1), infrared (IR) spectroscopy, ^{1}H Nuclear magnetic resonance (H NMR) spectroscopy, and mass spectroscopy (MS). Microanalysis was performed on Perkin–Elmer PE 2400 CHN analyzer. IR spectra were recorded on Shimadzu FT-IR-8400, ^{1}H NMR spectra were recorded on Bruker

Table 1. Elemental analysis for (1) ethyloxy, (2) pentyloxy, and (3) hexadecyloxy derivatives.

		Elements found (%)	Elements calculated (%)
Sr. No.	Molecular formula	C, H, O	C, H, O
1.	$C_{22}H_{18}O_4S$	69.82, 4.79, 16.91	69.78, 4.34, 17.48
2.	$C_{25}H_{24}O_4S$	71.40, 5.75, 15.22	71.28, 5.64, 15.18
3.	$C_{36}H_{46}O_4S$	75.22, 8.07, 11.10	75.10, 8.00, 11.13

spectrometer using $CDCl_3$ as solvent, and mass spectra were recorded on Shimadzu GC-MS Model No. QP-2010. The mesogenic behavior of homologue derivatives was observed through polarizing microscopy. The textures of smectogenic mesophase were determined by miscibility method.

Analytical data

Spectral data

^{1}H NMR in ppm for hexyloxy derivative

^{1}H NMR (400 MHz, $CDCl_3$) δ 0.89–0.93 (t, J = 7.8 Hz, 3H), 1.32 (m, 4H), 1.43–1.46 (m, 2H), 1.71–1.78 (m, 2H), 4.10–4.13 (t, J = 7.5 Hz, 2H), 7.11–7.15 (m, 2H), 7.20–7.22 (t, J = 7.5 Hz, 1H), 7.47–7.49 (m, 2H), 7.62–7.64 (d, J = 15.0 Hz, 1H), 7.73–7.75 (dd, J = 1.5, 7.5 Hz, 1H), 7.81–7.83 (dd, J = 1.5, 7.5 Hz, 1H), 7.92–7.96 (d, J = 15.0 Hz, 1H), 8.10–8.12 (m, 2H), 8.21–8.26 (m, 2H).

^{1}H NMR in ppm for heptyloxy derivative

^{1}H NMR (400 MHz, $CDCl_3$) δ 0.86–0.88 (t, J = 7.6 Hz, 3H), 1.26–1.28 (m, 8H), 1.42–1.44 (m, 2H), 1.74–1.76 (m, 2H), 4.10–4.14 (t, J = 7.4 Hz, 2H), 7.09–7.17 (d, J = 7.5 Hz, 2H), 7.20–7.22 (m, 1H), 7.47–7.49 (d, J = 8.1 Hz, 2H), 7.60–7.64 (d, J = 15.2 Hz, 1H), 7.72–7.74 (m, 1H), 7.82–7.84 (m, 1H), 7.93–7.97 (d, J = 15.1 Hz, 1H), 8.09–8.12 (s, 2H), 8.21–8.23 (d, J = 7.6 Hz, 2H).

^{1}H NMR in ppm for tetradecyloxy derivative

^{1}H NMR (400 MHz, $CDCl_3$) δ 0.85–0.94 (t, J = 7.4 Hz, 3H), 1.22–1.36 (m, 24H), 1.42–1.46 (m, 2H), 1.74–1.78 (m, 2H), 4.08–4.12 (t, J = 7.5 Hz, 2H), 7.05–7.11 (m, 2H), 7.18–7.22 (t, J = 7.5 Hz, 1H), 7.30–7.34 (m, 2H), 7.52–7.54 (d, J = 15.0 Hz, 1H), 7.78–7.80 (dd, J = 7.5, 1.5 Hz, 1H), 7.96–7.99 (d, J = 15.0 Hz, 1H), 8.07–8.13 (m, 2H), 8.26–8.32 (m, 2H).

IR in cm^{-1} for heptyloxy derivative

IR (KBr): 3070 (Ar str.), 2946, 2852 (–CH_3 str.), 1728 (–C=O str.), 1651, 1589, 1510 (–C=C–Ar str.), 1415, 1271, 1213, 1074 (–C–O str.), 844, 707 (–CH Ar bend) cm^{-1}.

IR in cm^{-1} for butyloxy derivative

IR (KBr): 3038 (Ar str.), 2930 (–CH_3 str.), 1750 (–C=O str.), 1656, 1544, 1508 (–C=C– Ar str.), 1056 (–C–O str.), 880, 742 (–CH Ar bend) cm^{-1}.

IR in cm^{-1} for methyloxy derivative

IR (KBr): 3068 (Ar str.), 2955 (–CH_3 str.), 1719, 1728 (–C=O str.), 1649, 1560, 1506 (–C=C–Ar str.), 1415, 1269, 1064 (–C–O str.), 885, 732 (–CH Ar bend) cm^{-1}.

Table 2. Transition temperatures in °C.

Compound No.	n-alkyl chain CnH2n+1 (n)	Sm	Nm	Isotropic
1	1	–	–	142.2
2	2	–	–	140.5
3	3	–	–	151.2
4	4	–	–	145.6
5	5	–	–	132.0
6	6	–	–	113.4
7	7	–	–	115.2
8	8	–	–	118.3
9	10	103.8	–	114.9
10	12	110.5	–	118.0
11	14	108.6	–	115.5
12	16	101.8	–	105.9

Mass of hexyloxy derivative

m/z (rel. int%): 434 $(M)^+$, 149, 121, 93, 65, 44.

Mass of octyloxy derivative

m/z (rel. int%): 462 $(M)^+$, 205, 121, 93, 65, 44.

Mass of dodecyloxy derivative

m/z (rel. int%): 518 $(M)^+$, 219, 121, 93, 57, 43.

Result and discussion

The chalconyl derivative of the present homologous series was synthesized by the condensation of dimeric 4-n-alkoxy benzoic acids and non-mesomorphic 3-(2-thiophenyl)-1-(4-hydroxyphenyl) 2-propen-1-one as shown in Scheme 1. Dimerization disappears on esterification by the lowering of transition temperatures than the corresponding n-alkoxy acids. Alternation of Cr-I temperatures are observed. Mesomorphism of smectogenic character commences late from the C_{10} member of the series enantiotropically with absence of nematic property. Transition and melting temperatures as determined using an optical polarizing microscope equipped with a heating stage (Table 2) were plotted against the number of carbon atoms present in the n-alkyl chain. A phase diagram (Fig. 1) is obtained, showing phase behavior of novel series by linking like or related points. Cr-I or Cr-Sm transition curve follows a zigzag path of rising and falling with overall descending tendency as series is ascended. Sm-I transition curve initially rises and then descends after passing through maxima at C_{12} member of a series without exhibition of odd-even effect. Thus, both transition curves behaved in normal established manner. The Sm-I transition curve is extrapolated to C_8 homologue [23–26] to determine and predict its latent transition temperature for smectic (109°C) and its latent ability to induce mesomorphism, following the trend of Sm-I transition curve. However, it is hypothetical and not realizable. Therefore, C_1 to C_8 homologues of a series are non-liquid crystals (NLC). Hence, the present series is partly smectogenic with the absence of nematic property, middle-ordered melting-type, and with low mesophase length range.

The dimerization of n-alkoxy benzoic acids disappears by breaking of hydrogen bonding between two molecules of n-alkoxy benzoic acids, and subsequently esterification process with 4-hydroxy chalcone derivative. Thus, the condensation process increases molecular length, lateral to terminal attractions, and molecular polarity and polarizability due to

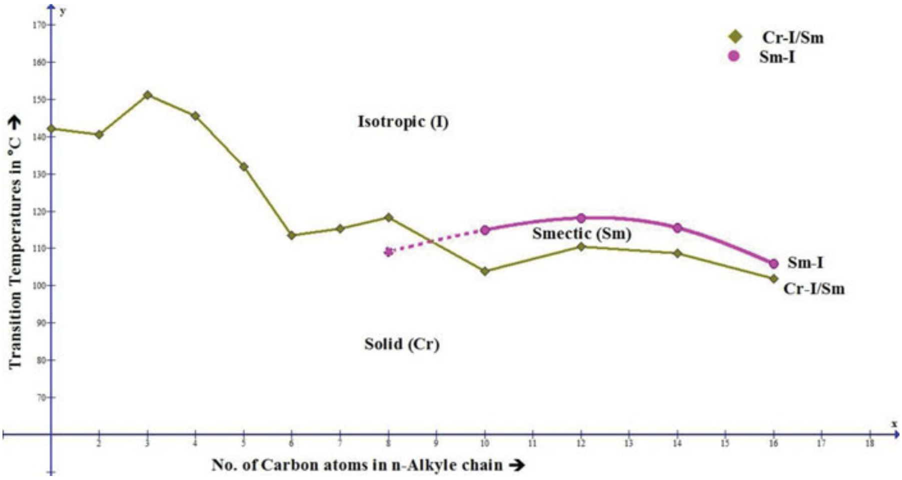


Figure 1. Phase behavior of series.

increase in permanent dipole moment across the long molecular axis, dipole–dipole interactions, electronic interaction, dispersion forces, etc. But suitable magnitudes of anisotropic forces of intermolecular attraction commence from decyloxy homologues (C_{10}) of preoccupied lamellar packing of molecules in their crystal lattices as a consequence of favorable molecular rigidity and flexibility required to cause smectic mesophase formation. The end-to-end intermolecular attractions failed to maintain the statistically parallel orientational order of molecules under the influence of exposed thermal vibrations in floating condition throughout the entire present series. Thus, nematogenic mesophase formation is totally hindered. Only sliding layered molecular arrangement occurs under thermal vibrations for C_{10}, C_{12}, C_{14}, and C_{16} homologues under floating condition of preoccupied lamellar molecular packing and layered structure, which induces smectic mesophase in enantiotropic manner. The non-mesomorphic behaviors of C_1–C_8 homologues is attributed to their high crystallizing tendency, which arises due to unsuitable magnitudes of anisotropic forces of intermolecular attractions. These intermolecular attractions immerge from low dipole–dipole interactions and low magnitudes of dispersion forces by interactions between instantaneous dipoles produced by the spontaneous oscillations of electron clouds of molecules that hinder the thermal environment of molecule arrangement and required to cause either smectic or/and nematic mesophase formation under floating condition on surface. The absence of odd-even effect in Sm-I transition curve is attributed to the absence of mesogenic phase formation either smectic or nematic in the initial (C_1 to C_8) homologues of the series under discussion. However, alternations in transition temperature are observed in isotropic temperatures due to odd and even number of carbon atoms in n-alkyl chain of n-alkoxy group. The changing trend of mesomorphic property and the degree of mesomorphism from homologue to homologue in the present series are attributed to the varying number of methylene unit or units present in left n-alkoxy terminal end group keeping right-handed thiophene ring as an intact end of molecules. The shorter phase length range or degree of mesomorphism is attributed to low resistivity (low enthalpy value, ΔH) of smectogenic homologues toward exposed thermal vibrations.

Variation in some mesomorphic properties of presently investigated series 1 is compared with structurally similar other known homologous series X [27] and Y [28] as mentioned in Fig. 2.

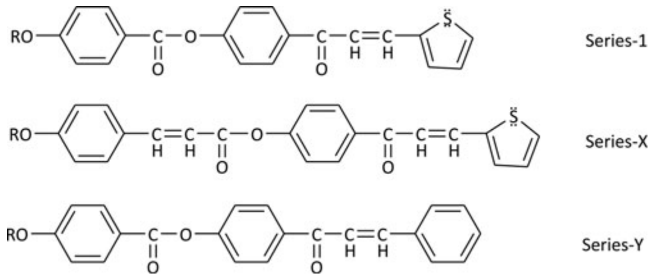


Figure 2. Structurally similar series.

Homologous series 1 of the present investigation and the homologous series X and Y chosen for comparative study are identical to each other with respect to major structural similarity, including minor changes in their molecular structures. Homologous series 1 and X are identical with respect to two phenyl rings, heterocyclic thiophene ring, and a central bridge –CO–CH=CH– linking middle phenyl ring and heterocyclic thiophene ring. Moreover, these are identical with respect to flexible n-alkoxy end group for the same homologue from series 1 to series X. However, these differ with respect to central bridges –COO– and –CH=CH–COO– contributing to the molecular rigidity of the same homologue from series 1 to series X. Homologous series 1 and series Y are identical with respect to two phenyl (first and middle) rings, two central bridges –COO– and –CO–CH=CH–, and the left n-alkoxy flexible end group for the same homologue from series 1 to series Y. However, they differ in the ring system, viz. phenyl ring and heterocyclic ring linked to –CO–CH=CH– at the right-sided end of molecules in series Y and series 1 respectively, which contribute to the molecular rigidity of the same homologue from series to series, and for homologue to homologue in the same series. Thus, variations in mesomorphic properties and the degree of mesomorphism for the same homologue from series to series and from homologue to homologue in the same series will vary with the changing features of series 1, X, and Y. Table 3 represents some mesomorphic properties of series 1, X, and Y under comparative study as mentioned below.

Table 3 indicates the following:

- Homologues series 1 and Y are only smectogenic and only nematogenic respectively, but series X is nematogenic in addition to smectogenic in character.
- Smectic mesophase commences from the C_{10} homologue of series 1 and from the C_4 homologue of series X, but it does not commence till the best member of series Y.

Table 3. Average thermal stability in °C.

Series→	1	X	Y
Smectic–isotropic Or Smectic–nematic	113.57 (C_{10}–C_{16})	136.56 (C_6–C_{12})	– –
Commencement of smectic phase	C_{10}	C_4	
Nematic–isotropic	–	152.78 (C_1–C_{18})	164.5 (C_6–C_{16})
Commencement of nematic phase	–	C_1	C_6
Total mesophase length range °C (Smectic + nematic)	4.1 to 11.1 C_{16}, C_{10}	4.4 to 26.0 C_3, C_8	7.0 to 45.0 C_{16}, C_{10}

- Nematic mesophase commences from the very first member (C_1) of series X and from sixth (C_6) member of series Y, but it does not commence till the last (C_{16}) member of series 1 of the present investigation.
- Smectic thermal stability of the present series 1 (113.57) is lower than that of series X (136.56), whereas smectic mesophase is totally destabilized for series Y.
- Nematic thermal stability (164.5) of series Y is greater than that of series X (152.78), whereas the same does not stabilize through the present series 1.
- The total mesophase length range (minimum to maximum) bears increasing order from series 1 to X to Y.

The energy (ΔH) stored in the molecules of a substance depends upon its quantity of mass and characteristics during its formation as heat of formation per mole. Therefore, the thermodynamic quantity, ΔH, varies for every substances from homologue to homologue in the same series and for the same homologue from series to series depending upon molecular rigidity and flexibility. The molecular rigidity in the present comparison varies with either one of the central bridges (series 1 and X) or with the ring system (series 1 and Y) for the same homologue from series to series. Therefore, the thermal resistivities toward exposed thermal vibrations vary for the same homologue from series to series. Comparing the thermal stability for smectic, which is higher for series X than for the present series 1, the vinyl carboxylate $-CH{=}CH-COO-$ central bridge of series X and the carboxylate $-COO-$ central bridge of series 1, although closely similar to each other, differ in length and added rigidity because of the presence of multiple bond of $-CH{=}CH-$ unit. Therefore, the vinyl carboxylate group, because of its longer length, causes more non-coplanarity by the twist obtained as the oxygen atom of the vinyl carboxylate group bump into the non-bonded adjacent hydrogen atoms of aromatic rings. On account this difference, the smectic–isotropic thermal stability of series 1 is lower than the smectic–nematic thermal stability of series X (or vice versa). Absence of preoccupied lamellar packing of molecular rigidity in the crystal lattices of series Y hinders the stabilization of smectic phase formation till the last member of series Y. Absence of nematic phase formation is attributed to the weaker end-to-end unsuitable magnitudes of anisotropic forces of intermolecular attractions and closeness as compared with series X and Y chosen for comparative study as a consequence of unfavorable molecular rigidity. The late commencement of mesophase (C_{10}) in the present series 1 and other series X and Y is attributed to the corresponding difference in the extent of non-coplanarity of molecules related to the molecular rigidity of each series. The observed difference in the total mesophase length range after acquiring suitable magnitudes of anisotropic forces of intermolecular attractions induces molecular resistivity toward exposed thermal vibrations, thus increasing from series 1 to series X to series Y. Hence, the stabilization of total mesophase length range is progressively enhanced from minimum to maximum for the series under comparison.

Conclusions

- A novel homologous series of 12 chalconyl ester homologues containing heterocyclic ring as end group has been synthesized, whose only smectogenic mesophase formation commences late from C_{10} homologue without exhibition of nematic property throughout the series.
- Group efficiency order derived for smectic and nematic on the basis of (i) thermal stability, (ii) commencement of mesophase, and (iii) total mesophase length range is as follows:

(i) **Smetic**
–CH=CH–COO– –COO– –COO–
with heterocyclic > with heterocyclic > with homocyclic
thiophene ring thiophene ring benzene ring
Nematic
–COO– –CH=CH–COO– –COO–
with homocyclic > with heterocyclic > with heterocyclic
benzene ring thiophene ring thiophene ring
(ii) **Smectic**
–CH=CH–COO– –COO– –COO–
with heterocyclic > with heterocyclic > with homocyclic
thiophene ring thiophene ring benzene ring
Nematic
–CH=CH–COO– –COO– –COO–
with heterocyclic > with homocyclic > with heterocyclic
thiophene ring benzene ring thiophene ring
(iii) **Sm + N**
–COO– –CH=CH–COO– –COO–
with homocyclic > with heterocyclic > with heterocyclic
benzene ring thiophene ring benzene ring

- Molecular rigidity and flexibility are the controlling factors to induce mesomorphism.
- Mesomorphism is very sensitive and susceptible to molecular structure.
- Chalconyl derivatives are biologically active, and sulfur-containing heterocyclic ring is helpful for dermatological treatment.
- Study of binary systems thermotropically may be useful for the manufacture of LC devices at desired temperature.

Acknowledgments

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