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Mesomorphism and molecular flexibility in novel chalconyl derivatives with two phenyl rings

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ABSTRACT

A novel chalconyl liquid crystalline (LC) homologous series with two phenyl rings and one central bridge RO–C_6H_4–CH=CH–CO–C_6H_4(O–$C_{18}H_{37}$) (meta) has been synthesized and studied with a view to understanding and establishing the effects of molecular structure on thermotropic LC behaviors or properties. Homologous series of present investigation (C_1–C_{18}) consists of thirteen homologues, and thermotropic mesomorphism for nematic and smectic commences from C_3 and C_6 homologues respectively. The C_3, C_4, and C_5 homologues are enantiotropically nematic, whereas, the homologues from C_6 to C_{18} are monotropically smectic plus nematic. Melting points and transition temperatures were determined using a polarizing optical microscope (POM) equipped with a heating stage. The textures of the nematic phase are threaded or Schlieren and that of a smectic phase are fan shaped of smectic-A or smectic-C type. The Cr–I/M, N–I/I–N, and N-Sm transition curves behave in the normal manner, and the Cr-I/M exhibits an odd-even effect. Analytical, thermal, and spectral data confirm the molecular structures. Thermal stabilities of smectic and nematic are low. Mesophase lengths are very short, ranging between 1–8°C and 14°C. The novel compounds are compared with a known series.

KEYWORDS

Chalcone; Liquid crystals; Mesomorphic; Nematic; Smectic

Introduction

Low temperature liquid crystalline (LC) [1] materials are important for applications in electronic devices, thermographic articles, and additionally for chalconyl derivatives in the field of agriculture and pharmaceutical preparations through their bioactive nature [2–10]. The aim of the presently planned investigation is to correlate mesomorphism and the molecular structure [11–16]. In view of the proposed investigation, a novel homologous series of chalcone derivatives are synthesized and characterized through elemental analysis, IR and ^{1}HNMR spectra and thermal data by POM. The evaluated thermometric data will be interpreted and discussed in terms of molecular rigidity and flexibility [17–20] and finally appropriate conclusions will be drawn from present investigation. The group efficiency order will be derived from comparative study of present investigation with structurally similar analogous series. Several homologous series have been reported to date [21–26].

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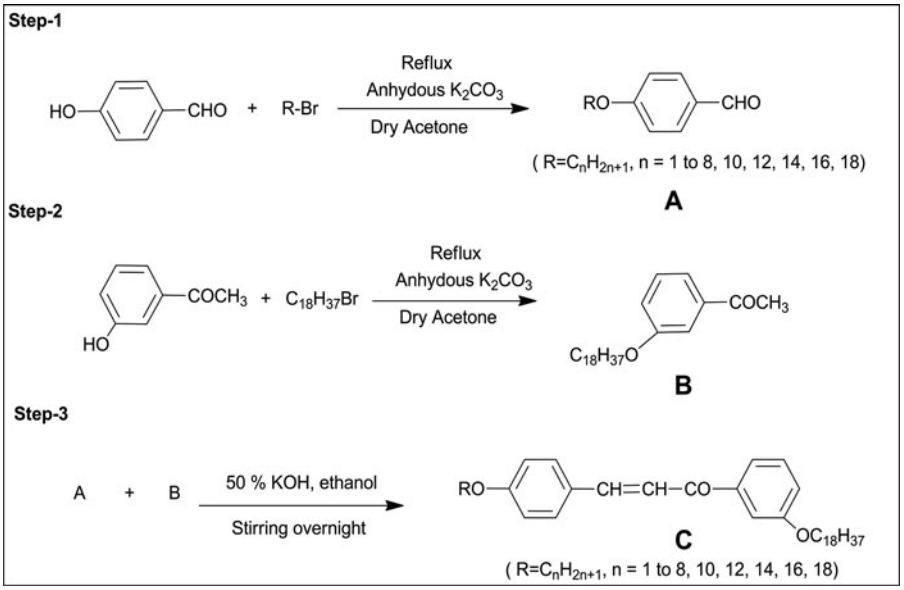


Scheme 1. Synthetic route to the series.

Experimental

Synthesis

Alkylation of 4-hydroxy benzaldehyde to give 4-n-alkoxy benzaldehyde was carried out by a reported method [27] 3-n-Octadecyloxy acetophenone was obtained by alkylation of 3-hydroxy acetophenone by a reported method [28] Thus, the chalconyl homologue derivatives (C) were prepared by a usual established method [29]. Final products were collected and filtered, washed with ethanol solution dried and purified until constant transition temperatures obtained using an optical polarizing microscope, equipped with a heating stage. Alkyl halides, acetone, EtOH, KOH, 3-hydroxy acetophenone, 4-hydroxy benzaldehyde required for synthesis were used as received, except solvents which were dried and distilled prior to use. The synthetic route to the series is shown below in Scheme 1.

Characterization

Representative homologues of a series were characterized by elemental analysis (Table 1), Infrared spectroscopy, ^{1}H NMR spectra. IR spectra were recorded on Perkin-Elmer spectrum GX, ^{1}H NMR spectra were recorded on Bruker using $CDCl_3$ as solvent. Microanalysis was performed on Perkin-Elmer PE 2400 CHN analyzer. Transition temperature (Table 2) and

Table 1. Elemental analysis for (1) propyloxy (2) pentyloxy (3) decyloxy and (4) dodecyloxy derivatives.

		Elements % found		Elements % calculated	
Sr. no.	Molecular formula	C	H	C	H
1	$C_{36}H_{54}O_3$	80.83	10.04	80.89	10.11
2	$C_{38}H_{58}O_3$	81.21	10.38	81.13	10.32
3	$C_{43}H_{68}O_3$	81.55	10.51	81.64	10.75
4	$C_{45}H_{72}O_3$	80.98	10.78	81.81	10.90

Table 2. Transition temperature in°C.

Sr.no	R = n-alkyl group	Transition temperatures in°C		
		Smectic	Nematic	Isotropic
1	C_1	–	–	88.0
2	C_2	–	–	84.0
3	C_3	–	57.0	66.0
4	C_4	–	54.0	69.0
5	C_5	–	48.0	58.0
6	C_6	(38.0)	(44.0)	51.0
7	C_7	(43.0)	(49.0)	60.0
8	C_8	(41.0)	(48.0)	54.0
9	C_{10}	(45.0)	(52.0)	60.0
10	C_{12}	(48.0)	(57.0)	69.0
11	C_{14}	(50.0)	(61.0)	70.0
12	C_{16}	(55.0)	(64.0)	75.0
13	C_{18}	(52.0)	(58.0)	73.0

() Indicates monotropy.

LC properties (Textures) were determined using an optical polarizing microscopy equipped with heating stage and digital camera (POM). Texture images of nematic phase were determined by miscibility method (Table 3).

Analytical data

IR Spectra in cm^{-1} for octyloxy and hexadecyloxy derivatives:

Octyloxy (C_8)

2914 (C–H str. of alkane), 2842 (C–H str. of –(CH_2–)n group of $-OC_8H_{17}$ alkyl chain, 1640 (C=O str. of carbonyl carbon of chalconyl group), 1604 (C=C str. of alkene in chalcone), 1510, 1543 (C=C str. of aromatic ring), 999 (C–H bending of alkene), 1178 (C–O str. of ether linkage), 1288, 1246 (C–O str. of carbonyl (>CO) group), 770 Polymethylene (–CH_2–) of $-OC_{18}H_{37}$, 675 Polymethylene (–CH_2–)n of $-OC_8H_{17}$, 823(–C–H– def. m di-substituted-Para), IR data confirms the molecular structure of comp.C_8.

Hexadecyloxy (C_{16})

2916 (C–H str. of alkane), 2850 (C–H str. of –(CH_2–)n group of $-OC_{16}H_{33}$ group, 1660 (>C=O str. of carbonyl group of chalconyl group), 1604(C=C str. of alkene), 1543 (C=C str. of aromatic ring), 1004, (C–H bending of alkene), 1199 (C–O str. of ether linkage), 1251 (C–O str. of carbonyl group), 770 Polymethylene (–CH_2–) of $-OC_{18}H_{37}$, 823 (–C–H– def. m di-substituted-Para), IR data confirms the molecular structure of comp.C_{16}.

Table 3. Texture of nematic phase of C_4, C_5, C_{12}, C_{14} by miscibility method.

Sr. no.	Homologue	Texture
1	C_4	Schlieren
2	C_5	Threaded
3	C_{12}	Threaded
4	C_{14}	Schlieren

^{1}HNMR spectra in $CDCl_3$ in δ ppm for butyloxy and pentyloxy derivative:

Butyloxy (C_4)

0.88 (t, 6H, $-CH_3$ of polymethylene $-C_6H_{13}$ and $-C_{14}H_{29}$), 1.79(p, 10H, $CH_3-CH_2-CH_2-CH_2-CH_2-$ of $-OC_6H_{13}$ and $-OC_{14}H_{29}$), 1.28 (m, 15H $-CH_2-CH_2-CH_2-$ of $-OC_7H_{15}$ and $-OC_{14}H_{29}$), 1.31 (q, 8H, $-CH_2-CH_3$), 4.06(t, 4H, $-OCH_2-CH_2-$), 7.59 (d, 2H, $-CH=CH-$), 7.43, 7.28, and 7.83 (4H, meta substituted phenyl ring), 7.56 and 7.97 (4H, phenyl ring with alkoxy chain). NMR data confirms the molecular structure of comp.C_4.

Pentyloxy (C_5)

0.88 (t, 6H, $-CH_3$ of $-C_{10}H_{21}$ and $-C_{14}H_{29}$), 1.80(p, 10H, $CH_3-CH_2-CH_2-CH_2-CH_2-CH_2$ of $-OC_{10}H_{21}$ and $-OC_{14}H_{29}$), 1.28 (m, polymethylene 16H $-CH_2-CH_2-CH_2-$ of $-OC_{10}H_{21}$ and $-OC_{14}H_{29}$), 1.34 (q, 8H, $-CH_2-CH_3$), 4.05(t, 4H, $-OCH_2-CH_2-$), 7.59 (d, 2H, $-CH=CH-$),7.43, 7.28, and7.83 (4H, meta substituted phenyl ring), 7.56 and 7.97 (4H, phenyl ring), 6.95 and 7.62 (4H, phenyl ring with alkoxy chain). NMR data confirms the molecular structure of comp.C_5.

Result and discussion

4-n-Alkoxy benzaldehydes on condensation with 3-octadecyloxy acetophenone yielded thirteen chalconyl derivatives (C_1-C_{18}) with C_3 to C_{18} homologues as mesogenic. C_3-C_5 derivatives are enantiotropically nematogenic with absence of smectic property, C_6 to C_{18} derivatives are monotropically nematogenic plus monotropically smectogenic. C_1-C_2 homologues are nonmesogenic. Transition temperatures of homologues as determined by POM on plotting for number of carbon atoms present in n-alkyl chain 'R' of –OR terminal group and subsequently on linking like or related points, transition curves Cr-I/N, I-N or N-I, N-Sm are obtained. Cr-I/N transition curve follows a zigzag path of rising and falling values with an overall falling tendency and so behaves in a normal manner. The N-I or I-N transition curve follows descending tendency up to C_8 homologue and then negligibly ascended up to last homologue with exhibition of odd-even effect. Thus, it behaved in normal manner neglecting minor deviating effect from C_8 or C_9 homologue beyond merging of N-I/I-N curves for higher homologue of longer n-Alkyl chain 'R' of –OR group. N-Sm transition curve gradually rises up to C_{16} homologue and then descended to C_{18} homologue member after passing through maxima at C_{16} member. Thus, it behaved in normal manner with exhibition of odd-even effect up to C_8 homologue; and then monotropic N-Sm transition curve prolonged as a single transition curve. The mesophase length for N-Sm transitions are very short, of the order of one or a fraction of 1°C. The textures of nematic phase are threaded or Schlieren and that of the smectic phase are focal conic fan shaped smectic-A or smectic-C as judged directly from the heating top of the POM. Analytical, thermal and spectral data supported molecular structures of novel homologues. Thermal stabilities for nematic is 64.3°C and that of the smectic phase are minimum from fraction of 1°C or maximum 1°C. Thus, mesophase lengths are very small or shorter. The LC properties of thermotropic novel LC chalcones from homologue to homologue in the same series undergo variation with changing; length of molecules or permanent dipole moment across the longer molecular axis.

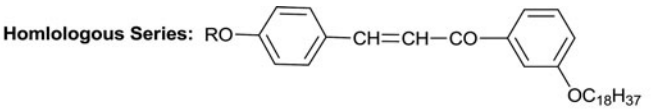
The absence of mesomorphic tendency of C_1 and C_2 homologue is attributed to low magnitudes of intermolecular dispersion forces and low magnitudes of dipole–dipole interactions which induces unsuitable magnitudes of anisotropic forces of end to end or lateral cohesions and closeness as a consequence of unfavorable molecular rigidity and flexibility to induce

mesomorphism. Therefore, their molecules are unable to withstand the intensity of exposed thermal vibrations and sharply transform into isotropic liquid without passing through mesomorphic state. However, the homologues C_6–C_{18} transform from crystalline solid state to isotropic state without showing of any sort of LC state, but, on cooling, a monotropic nematic of considerable temperature range results, and then a monotropic smectic mesophase of very short temperature range. The molecules of C_3,C_4,C_5 under microscopic (POM) examination on heating top resists exposed thermal vibrations and disalign at an angle less than ninety degree due to suitable magnitudes of end to end intermolecular cohesions and closeness, as a result of favorable or suitable magnitudes of dispersion forces, dipole–dipole interactions, molecular rigidity, and flexibility. Thus, the molecules from C_3 to C_5 homologues acquire comfortable thermodynamic environmental situation to set their respective molecules in statistically parallel orientational order for some degrees of temperature range in reversible (heating and cooling both) manner which facilitated enantiotropically nematogenic mesomorphism only. Odd-even effect exhibited by I-N/N-I and N-Sm transition curves of a phase diagram (Fig. 1) is attributed to the sequentially added methylene unit at the n-alkyl chain 'R' of –OR terminal end group. The minor and negligible deviation from normal behavior of transition curves of a phase diagram from and beyond merging of transition curves contributing to odd-even effect of higher homologues of longer n-alkyl chain 'R' and $-C_{18}H_{37}$ hydrocarbon chain bonded through oxygen atom as terminal or lateral groups with phenyl rings; which may coil or bend or flex or couple to lie with major axis of core to modify favorable or unfavorable molecular rigidity and / or flexibility to cause phase behaviors with LC properties. The variations in mesogenic or LC properties and the degree of mesomorphism from homologue to homologue in the same novel series are attributed to the sequentially added carbon atoms in n-alkyl chain 'R' of –OR group which may cause variations in the molecular length, permanent dipole moment across the long molecular axis, length to breadth ratio, the ratio of the molecular polarity to polarizability, intermolecular dispersion forces due to end to end or lateral attractions and the magnitudes of closeness, molecular rigidity and /or flexibility etc. Following Fig. 2 represents some structurally similar analogous series-X [30] and Y [31] chosen for the comparative purpose with presently investigated novel series-1:

Homologous series 1, X, Y under comparative study consisted of two phenyl rings and one central bridge –CH = CH–CO– which contributes identically to the molecular rigidity from series to series for the same homologue and from homologue to homologue in the same series. The terminal group –OR, in which n-alkyl chain 'R' varies with number of methylene units from homologue to homologue in the same series but the number of methylene units of 'R' remains unaltered for the same homologue from series to series, contributing partly towards sharing of total molecular flexibility. Laterally substituted $-OC_{18}H_{37}$(n), $-OC_{16}H_{33}$(n) and $-OC_{14}H_{29}$(n) groups whose molecular flexibility, group polarities and polarizability vary from series to series for the same homologue but remains unaltered from homologue to homologue in the same series respectively due to their respective lateral groups linked to meta position of second phenyl ring but vary with changing -R. Thus, variations in LC properties among the series 1, X and Y can be attributed to the varying features related to mesomorphism due to, even a minute changing features or the total changing flexibility of terminal and / or lateral groups; for the same homologue from series to series. Following Table 4 represents some thermometric data evaluated from present investigation of series-1 and series-X and Y chosen for comparative study.

Above Table 4 indicates that,

- The homologous series 1,X and Y are identical with respect to the exhibition of smectogenic and nematogenic property.



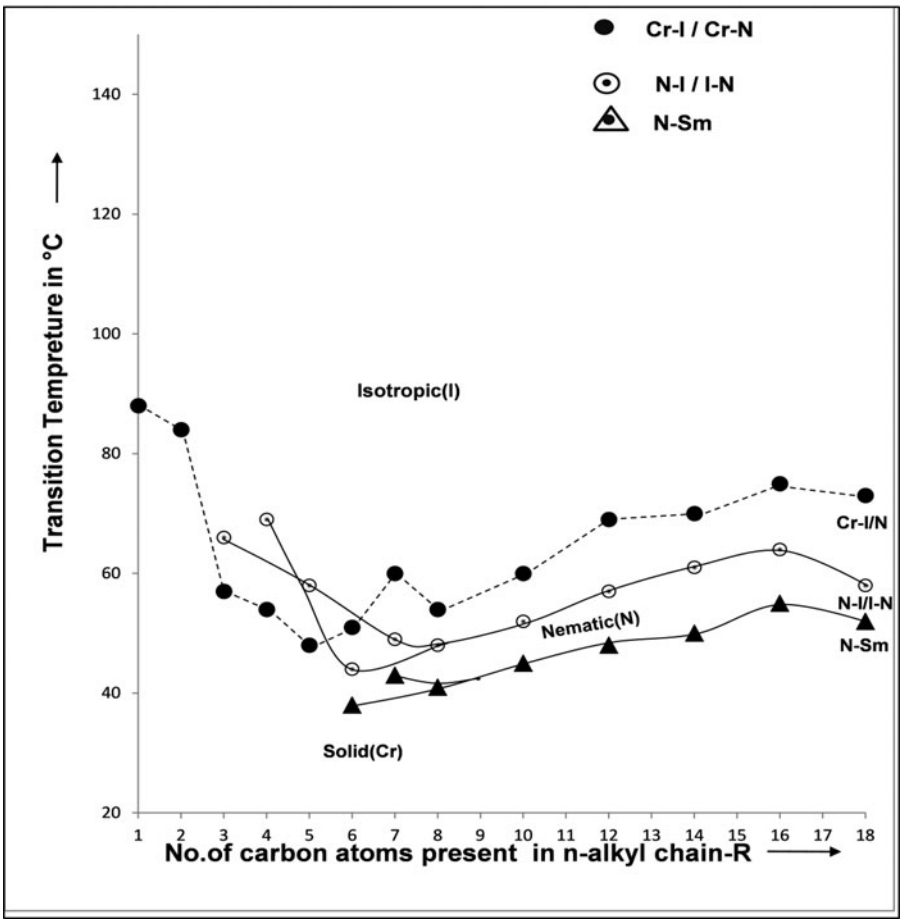


Figure 1. Phase behaviors of series.

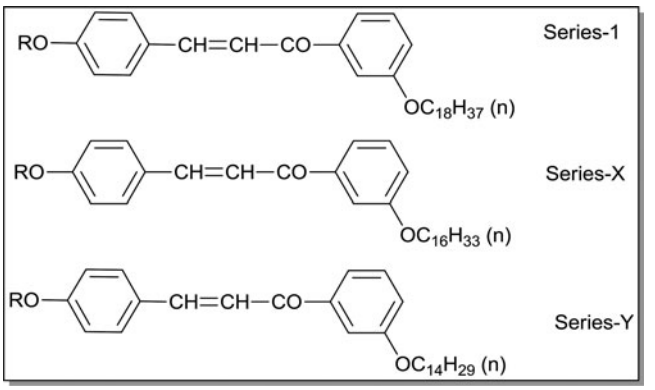


Figure 2. Structurally similar analogous series.

Table 4. Thermal stability in°C.

Series→	Series-1	Series-X	Series-Y
Sm-I or Sm-N or N-Sm commencement of smectic phase	Fraction of 1°C or 1°C (C_6–C_{18}) C_6	1 to 2.0°C (C_7–C_{18}) C_7	1 to 2.0°C (C_6–C_{18}) C_6
N-I or I-N	64.3	72.6	67.5
Commencement of nematic phase	(C_3–C_5) C_3	(C_2–C_6) C_2	(C_2–C_5) C_2
Mesophase length (total)	06.0–15.0°C $C_6/C_7/C_{18}$ C_4	05.0–21.0°C C_{18} C_4	05.0–12.0°C C_8 $C_3/C_5/C_{18}$

- Exhibition of smectogenic mesophase commences from C_6 (series 1 and Y) or C_7 (series-X) homologue with the difference of only one homologue.
- Nematogenic mesophase commences from C_2 (series X and Y) or C_3 (series-1) homologue with the difference of only one homologue.
- Thermal stability for nematic increases from series 1 to Y to X. Whereas, stabilization of smectogenic character is very poor.
- Total mesophase length in higher category follows increasing order as series-X > series-1 > series-Y whereas in lower category are equal for series-X and Y but more for present series 1 by 1°C.

Looking to the geometrical shapes, aromaticity, molecular length or the permanent dipole moment across the long molecular axis, molecular rigidity for the same homologue from series to series under comparative study (Fig. 2) are identical except the length of laterally meta substituted $–OC_{18}H_{37}$ (n), $-OC_{16}H_{33}$ (n) and $–OC_{14}H_{29}$ (n) group of series 1, X and Y, respectively, which differs by $–CH_2$-CH_2- unit from series-1 to series X to series Y affecting molecular flexibility if the status of their n-alkyl chains $–C_{18}H_{37}$ (n), $-C_{16}H_{33}$ (n) and $–C_{14}H_{29}$ (n) are as identical as expected in equal manner. But, longer n-alkyl meta substituted chain bonded through oxygen atom of lateral group also may cause difference in polarity and polarizability due to gradually added $–CH_2$-CH_2- unit and the same longer n-alkyl chains may coil or bend or flex or couple to lye with major axis of core structure which may modify magnitudes of flexibility and the magnitudes of intermolecular cohesions and closeness for the same homologue from series to series under the influence of exposed thermal vibrations when floated on the surface. But experimentally evaluated thermometric data suggest that, there is very little difference in thermal stabilities, commencements of smectic and nematic phase, degrees of mesomorphism or facilitations of upper and lower total mesophase lengths as well as the types of mesophases for the same homologue from series to series, i.e., lengthening of laterally meta substituted n-Alkyl chain bonded through oxygen atom with phenyl ring are less effective in raising or depressing thermotropic mesomorphic properties.

Conclusions

- Chalconyl derivatives consisted of two phenyl rings with laterally meta substituted n-alkoxy group are predominantly nematogenic and partly smectogenic with its low melting type behavior.
- Group efficiency order derived for smectic and nematic on the basis of (a) thermal stabilities (b) early commencement of mesophase and (c) upper and lower mesophase lengths are as under.

(a) **Smectic**
Series 1 = Series X = Series Y
Nematic
Series X > Series Y > Series 1
(b) **Smectic**
Series 1 = Series Y > Series X
Nematic
Series X = Series Y > Series 1
(c) **Total mesophaselengths**
Upper: Series X > Series 1 > Series Y
Lower: Series 1 > Series X = Series Y

- Mesomorphism is very sensitive and susceptible to molecular structure as a consequence of molecular rigidity or/and flexibility.
- Present investigation may be useful for the construction of devices to be operated at low temperature or room temperature. Biological activity of novel compounds may be exploited and studied for pharmaceutical and agricultural production.
- This supported and raised the credibility to the conclusions drawn earlier.

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