



RESEARCH ARTICLE

SYNTHESIS, CHARACTERIZATION AND MESOMORPHISM OF CHALCONE MESOGENS: A-4-ALKOXYBENZOYL B-4/-HEPTYLOXY PHENYL ETHYLENE

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ABSTRACT

A new mesogenic homologous series : RO-C₆H₆-CH=CH-CO-C₆H₆-OC₇H₁₅ have been synthesized and studied with a view to understand establish the effect of molecular structure on mesomorphic properties and characterized by a combination of elemental analysis and standard spectroscopic methods (IR, ¹HNMR and elemental analysis). In present series mesomorphism commences from C₄ homologue, C₄ and C₅ exhibit only enantiotropic nematic mesophase whereas C₆ to C₁₈ exhibit enantiotropic nematic as well as smectic mesophase. The thermometric behaviours of novel homologue substances and their textures were determined by an optical polarizing microscopy (POM) equipped with a heating stage. Textures of a nematic phases are threaded or schlieren. The textures of smectic phase are directly judged from the heating top as smectic-A. The mesomorphic properties of present series were compared with other two structurally analogous series.

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INTRODUCTION

The Combination of thermotropic LC property (Reinitzer, 1888) and biological activity possessing property have been matched in chalconyl derivatives. There are many kinds of liquid crystalline (LC) materials with different molecular structures and shape. The Chalconyl derivatives due to their geometrical shapes may exhibit LC properties with lower thermometric transitions and bioactivity. Chalconyl derivatives with two ring and -CH=CH-COO- as central linkage exhibit thermotropic liquid crystal property (LC) property with biological activity which is very useful for society in the manufacture of LC devices and the pharmaceutical preparations (Collings and Hird, 1997; Calliste *et al.*, 2001; Rajesh *et al.*, 2008; Jain Upendra *et al.*, 2014; Imrie and Luckhurst, 1998; ImranTadwee *et al.*, 2012). Therefore present investigation is planned with a view to understand and establish the relation between thermotropic LC properties and the molecular structure of a substance or substances (Collings and Hird, 1997; Hird *et al.*, 1993; Gaikwad Prajkata and Desai Maya, 2013; Gray, 1974; Gray, 1962; Sashidhara *et al.*, 2015) through synthesis of novel chalconyl series of homologues.

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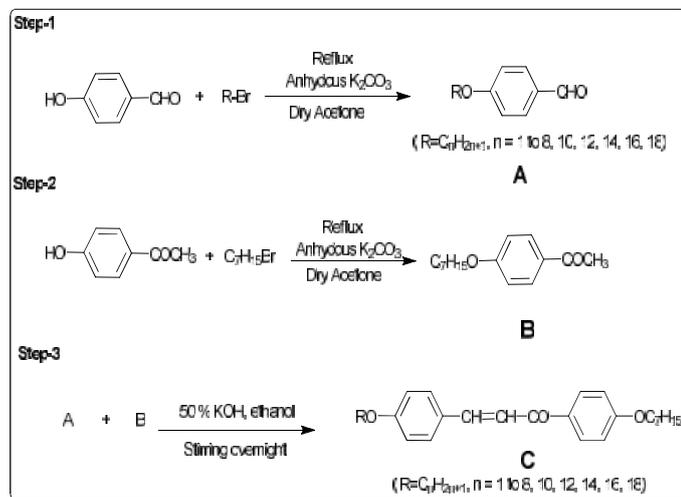
In present investigation synthesizing a novel homologous series which consist of a chalconyl group, which link two phenyl rings and varying n- alkoxy group -OR and fixed -OC₇H₁₅ left and right terminal end groups, of low transition temperature. Thermometric data will be derived using an optical polarising microscopy (POM) equipped with a heating stage and will be discussed and interpreted in terms of molecular rigidity and flexibility in relation (Gray *et al.*, 1989; Gray and Winster (Eds); Imrie, 1999; Dermus, 1998; Hird *et al.*, 1993) to molecular structure geometry, size, polarity and polarizability etc. LC properties of present novel series will be compared with the structurally similar analogous series. Number of homologous series have been reported till the date (Macros *et al.*, 1992; Doshi *et al.*, 2013; Patel *et al.*, 2012).

MATERIALS AND METHODS

Synthesis

Alkylation of 4-hydroxy benzaldehyde to give 4-n-alkoxy benzaldehyde (A) is carried out by reported method (Nikitin and Andryukhova, 2004) and 4-n-heptyloxy acetophenone (B) is obtained by alkylation of 4-hydroxy acetophenone by reported method (Aurangzeb Hasan *et al.*, 2011) Thus, the chalconyl homologue derivatives were prepared by usual establish method (Furniss *et al.*, 1989). Homologues were

filtered, washed with ethanol solution dried and purified till constant transition temperatures obtained using an optical polarising microscope equipped with a heating stage. Alkyl halides, EtOH, KOH, 4-Hydroxy acetophenone, 4-Hydroxy benzaldehyde etc., required for synthesis were used as received except solvents which were dried and distilled prior to use. The synthetic route to the series is mentioned below as Scheme 1.



Scheme 1. Synthetic route to the series

Characterization

Representative homologues of a series were characterised by elemental analysis, Infrared spectroscopy, ^1H NMR spectra, IR spectra were recorded on Perkin-Elmer spectrum GX, ^1H NMR spectra were recorded on Bruker using CDCl_3 as solvent. Microanalysis was performed on Perkin-Elmer PE 2400 CHN analyser (Table-1). Transition temperature (Table-3) and LC properties (Textures) were determined using an optical polarising microscopy equipped with heating stage and digital camera (POM). Texture images of nematic phase were determined by miscibility method (Table-2), thermodynamic quantities enthalpy (H) and entropy ($S=H/T$) are qualitatively discussed.

Analytical Data

Table 1. Elemental Analysis for (1) Octyloxy (2) Dodecyloxy (3) Tetradecyloxy and (4) Hexadecyloxy derivatives

S.No.	Molecular formula	% Elements found		% Elements Theoretical	
		C	H	C	H
1	$\text{C}_{30}\text{H}_{42}\text{O}_3$	80.02	9.30	80.00	9.33
2	$\text{C}_{34}\text{H}_{50}\text{O}_3$	80.62	9.86	80.63	9.88
3	$\text{C}_{36}\text{H}_{54}\text{O}_3$	80.85	10.10	80.89	10.11
4	$\text{C}_{38}\text{H}_{58}\text{O}_3$	81.10	10.30	81.13	10.32

Table 2. Texture of Nematic Phase of C_7 , C_{12} , C_{16} , C_{18} by miscibility method

S. No.	Homologue	Texture
1	C_7	Threaded
2	C_{12}	Schlieren
3	C_{16}	Schlieren
4	C_{18}	Schlieren

IR Spectra in cm^{-1} for Octyloxy & Tetradecyloxy Derivatives

Octyloxy : 2910 (C-H str. of alkane), 2848 (C-H str. of $-(\text{CH}_2)_n$ group of $-\text{OC}_8\text{H}_{17}$ alkyl chain), 1608-1650 (C=O str. of carbonyl carbon of chalconyl group), 1610 (C=C str. of alkene), 1511, 1565 (C=C str. of aromatic ring), 996 (C-H bending of alkene), 1180 (C-O str. of ether linkage), 1283, 1242 (C-O str. of carbonyl group), 778 Polymethylene $(-\text{CH}_2-)$ of $-\text{OC}_7\text{H}_{15}$, 681 Polymethylene $(-\text{CH}_2-)_n$ of $-\text{OC}_8\text{H}_{17}$, 831 (C-H def. m di-substituted-Para)

Tetradecyloxy : 2912 (C-H str. of alkane), 2846 (C-H str. of $-(\text{CH}_2)_n$ group of $-\text{OC}_{14}\text{H}_{29}$ group), 1605-1657 (C=O str. of carbonyl group of chalconyl group), 1612 (C=C str. of alkene), 1512, 1569 (C=C str. of aromatic ring), 998 (C-H bending of alkene), 1182 (C-O str. of ether linkage), 1282, 1246 (C-O str. of carbonyl group), 775 Polymethylene $(-\text{CH}_2-)$ of $-\text{OC}_7\text{H}_{15}$, 679 Polymethylene $(-\text{CH}_2-)_n$ of $-\text{OC}_8\text{H}_{17}$, 827 (C-H def. m di-substituted-Para)

^1H NMR spectra in CDCl_3 in ppm for Octyloxy & Octadecyloxy Derivative:

Octyloxy: 0.87 (t, $-\text{CH}_3$ of polymethylene $-\text{C}_{10}\text{H}_{21}$ and $-\text{C}_7\text{H}_{15}$), 1.76 (p, $\text{CH}_3\text{-CH}_2\text{-CH}_2\text{-CH}_2\text{-CH}_2-$ of $-\text{OC}_{10}\text{H}_{21}$ and $-\text{OC}_7\text{H}_{15}$), 1.25 (m, $-\text{CH}_2\text{-CH}_2\text{-CH}_2-$ of $-\text{OC}_{10}\text{H}_{21}$ and $-\text{OC}_7\text{H}_{15}$), 1.32 (q, $-\text{CH}_2\text{-CH}_3$), 3.98 (t, $-\text{OCH}_2\text{-CH}_2-$), 7.62 (d, $-\text{CH}=\text{CH}-$), 7.40, 7.27 & 7.83 (para substituted phenyl ring), 7.52 & 7.97 (phenyl ring with alkoxy chain).

Octadecyloxy: 0.86 (t, $-\text{CH}_3$ of $-\text{C}_{18}\text{H}_{37}$ and $-\text{C}_7\text{H}_{15}$), 1.81 ($\text{CH}_3\text{-CH}_2\text{-CH}_2\text{-CH}_2\text{-CH}_2\text{-CH}_2-$ of $-\text{OC}_{18}\text{H}_{37}$ and $-\text{OC}_7\text{H}_{15}$), 1.27 (polymethylene $-\text{CH}_2\text{-CH}_2\text{-CH}_2-$ of $-\text{OC}_{18}\text{H}_{37}$ and $-\text{OC}_7\text{H}_{15}$), 1.30 (q, $-\text{CH}_2\text{-CH}_3$), 4.04 (t, $-\text{OCH}_2\text{-CH}_2-$), 7.59 (d, $-\text{CH}=\text{CH}-$), 7.43, 7.26 & 7.85 (para substituted phenyl ring), 7.55 & 7.96 (phenyl ring with alkoxy chain)

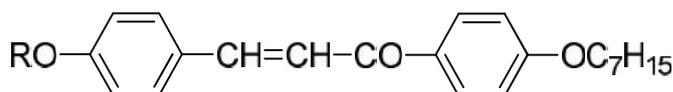


Table 3. Transition temperature in $^{\circ}\text{C}$

S.No.	R= n-alkyl group	Transition temperatures in $^{\circ}\text{C}$		
		Smectic	Nematic	Isotropic
1	C_1	-	-	77.0
2	C_2	-	-	79.0
3	C_3	-	-	72.0
4	C_4	-	56.0	68.0
5	C_5	-	52.0	61.0
6	C_6	40.0	47.0	59.0
7	C_7	38.0	46.0	54.0
8	C_8	43.0	50.0	60.0
9	C_{10}	39.0	45.0	53.0
10	C_{12}	44.0	52.0	62.0
11	C_{14}	42.0	51.0	63.0
12	C_{16}	46.0	56.0	67.0
13	C_{18}	49.0	60.0	71.0

RESULTS AND DISCUSSION

A novel chalconyl homologous series with two phenyl rings, two terminal group (-OR and -OC₇H₁₅) bonded through one central bridge (-CH=CH-CO-) have been synthesized by condensation reaction between 4-n-alkoxy benzaldehyde and 4-octyloxy acetophenone which include thirteen homologues. Liquid crystal property started from from C₄ homologue and remain upto C₁₈ homologue. C₄ and C₅ exhibit enantiotropic nematic property, C₆ to C₁₈ exhibit both enantiotropic nematic and smectic property and C₁ to C₃ does not exhibit any kind of liquid crystal property.

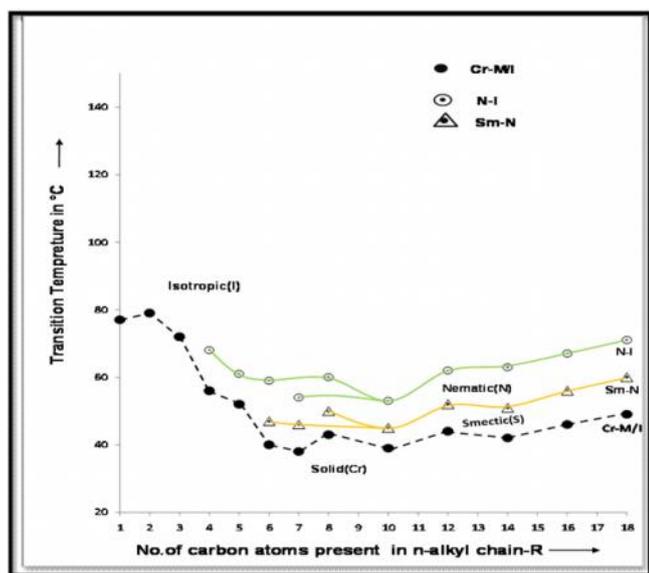
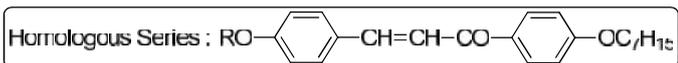


Figure 1. Phase behavior of series

The transition temperatures (Table-3) determined with the help of polarizing optical microscope (POM) equipped with the hot stage plotted against the number of carbon atoms present in n-alkyl chain 'R' of -OR group and then transition curves Cr-M/I, N-I and Sm-N are obtained on linking like or related transition points as shown in a phase diagram (Figure-1). Sm-N or N-Sm transition curve initially descended to C₁₀ homologue and then ascended to upto C₁₈ homologue and slightly descended from C₁₀ to C₁₂ homologue with exhibition of odd-even effect from C₆ to C₉ homologues of a series. The Cr-N/I transition curve adopted zigzag path of rising and falling with overall descending manner. N-I or I-N transition curve descended from C₄ to C₁₀ and then ascended by negligible magnitudes upto C₁₈ homologue with exhibition of odd-even effect from C₄ to C₇ homologue. The variations in mesogenic properties and behaviours from homologue to homologue in the same series is observed with changing number of carbon atoms in n-alkyl chain 'R' of -OR group, keeping rest of the molecular part unaltered throughout a series. Thermal stabilities are 50.87°C and 61.80°C for smectic and nematic respectively whose maxima to minimum phase lengths for smectic and nematic are 09 °C to 13.0 °C and 06 °C to 11.0 °C respectively. Thus, novel homologous series is low melting type series. Analytical, spectral and thermal data

supported molecular structures of homologues. The exhibition of mesomorphism by C₄ to C₁₈ homologues either as enantiotropic manner is attributed to suitable magnitudes of anisotropic forces of intermolecular end to end and lateral attractions as a result of favourable magnitudes of molecular rigidity and flexibility; induced by dispersion forces and dipole-dipole as well as electronic interactions. The intermolecular cohesive forces on heating a sample arrange the molecules of C₄ and C₅ homologues in stastically parallel orientational order. Therefore the molecules of C₄ and C₅ homologues exhibited enantiotropically nematic property. However, the molecules of higher homologues C₆ to C₁₈ of longer n-alkyl chain 'R' of -OR end group exhibited both nematic and smectic property. The lowering of transition temperature from lower to higher homologue is attributed to weakening of gradual intermolecular forces, with increasing addition of methylene unit or units. The diminishing of odd-even effect from and beyond C₅ for nematic and C₈ for smectic is attributed to the coiling or bending or flexing or coupling of longer n-alkyl chains with principal axis of the core structure of molecules from 'R' and terminal para -C₇H₁₅ which induces uncertainty in molecular polarity and polarizability, affecting factors governing intermolecular cohesion and closeness. However, disappearance of odd-even effect for higher homologues of longer n-alkyl chain 'R' of -OR group and -OC₇H₁₅(n) terminal group is attributed to the coiling or bending or flexing or coupling of n-alkyl chain with the major axis of core structure of a molecule. Thus, unusual status of n-alkyl chain may deviate the magnitudes of anisotropic forces of intermolecular attractions and may favour or disfavour the exhibition of mesomorphism and may alter the normal sequential order of enantiotropy in a homologous series. The variations in thermometric mesogenic properties of presently investigated novel series -1 are compared with other structurally similar analogous series -A (Jain and Patel, 2016) and B (Jain and Patel, 2016) as mentioned below in Figure 2.

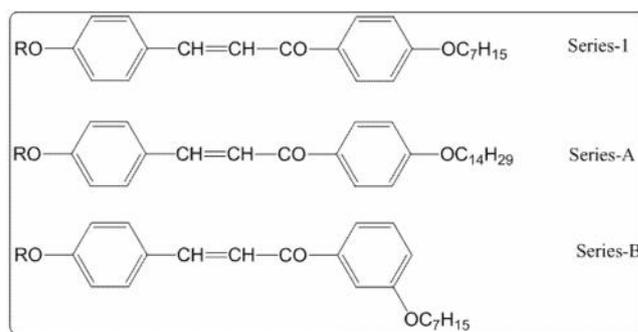


Figure 2. Structurally analogous series

Homologous series-1, A and B under comparative study are identical with respect to two phenyl rings bonded through chalconyl central bridge (-CH=CH-CO) which fully contribute to total molecular rigidity of series-1, A and B and the left n-alkoxy terminals -OR for the same homologue from series to series which partly contributes to the total molecular flexibility for same homologue and homologue to homologue in the same series.. Homologous series 1, A and B differs with tailed end group viz. para -OC₇H₁₅ (n), para -OC₁₄H₂₉ (n), meta -OC₇H₁₅ (n), for the same homologue from series to series and homologue to homologue in the same series which partly

contributes to the total molecular flexibility. Therefore, variations in liquid crystal (LC) properties are due to differing features of their molecular structures which differs with respect to flexibility of tailed end groups and their position of substitutions para or meta as shown in figure-2. Following table-4 represents some mesogenic properties in comparative manner for series-1, A and B.

Table 4. Thermal Stability in °C

Series	Series-1		Series-A		Series-B	
	Para	-OC ₇ H ₁₅	Para	-OC ₁₄ H ₂₉	Meta	-OC ₇ H ₁₅
Sm-I or Sm-N Commencement of smectic phase	50.87 (C ₆ -C ₁₈)		59.0 (C ₁₀ -C ₁₈)		Fraction of 1 °C or 1 °C (C ₁₀ - C ₁₈)	
	C ₆		C ₁₀		C ₁₀	
N-I Commencement of nematic phase	61.80 (C ₄ -C ₁₈)		73.0 (C ₅ -C ₁₈)		66.0 (C ₇ - C ₈)	
	C ₄		C ₅		C ₇	
Total mesophase lengths in °C minimum to maximum	09.0 to 23.0 C ₅ C ₁₆		13.0 to 27.0 C _{5/7} C _{10/16}		07.0 to 15.0 C _{10/14/16} C ₁₂	

Above Table 4 represents that, homologous series 1, X and Y are:

- A homologous novel series - 1 and A are enantiotropically smectic plus nematic and series-B is enantiotropically nematic and monotropically smectic plus nematic
- Nematic mesomorphism commences from C₄ homologue in series-1 whereas it commences from C₅ and C₇ homologue in series -A and B respectively.
- Smectic mesomorphism commences from C₆ homologue in series-1 whereas it commences C₁₀ homologue in series-A and B respectively
- Lower mesophase length of Series-A is longest compare to series-1 and B as well as upper mesophase length increases from Series-B to 1 to A.
- Thermal stability for nematic and smectic are more for series-A as compared to series 1 and series-B.

The suitable magnitudes of anisotropic forces of intermolecular attractions caused by suitable magnitudes of dispersion forces, dipole-dipole interactions, molecular polarity and differing polarizability offered by differing para substituted -OC₇H₁₅(n), -OC₁₄H₂₉(n) and meta substituted -OC₇H₁₅(n) groups which induces magnitudes of molecular flexibility to facilitates and stabilize the molecular arrangement required to exhibit nematic and smectic mesophase formation in enantiotropic manner under floating condition. The early or late mesophase or mesophases appearances are attributed to the extent of molecular planarity and noncoplanarity exerted from respective molecular structures and the status of n-alkyl chains of the para-OC₇H₁₅(n), -OC₁₄H₂₉(n) and meta -OC₇H₁₅(n) terminal and lateral groups of series 1, A and B respectively. The alternation of nematic thermal stability or mesophase lengths are related with the combined effects of molecular rigidity in combination with flexibility and the unexpected status of both ended n-alkoxy end groups, which may fluctuate with the status of flexible groups.

Series-1 and B has para substituted -OC₇H₁₅ and -OC₁₄H₂₉ group whereas Series-B have meta substituted lateral-OC₇H₁₅ groups so there are difference in the geometry of series-1 and A as compared to series-B. Thus, the two opposing effects of intermolecular attractions due to para and meta substituted tail ended n- alkoxy groups play role in facilitating smectic and/or nematic mesophase formation through predominancy of either molecular polarity or polarizability factor.

Conclusion

- Novel chalconyl homologous series of two phenyl rings and one central bridge is predominately enantiotropic nematic and partly enantiotropic smectic whose degree of mesomorphism, mesophase lengths and thermal stabilities are low and of low ordered melting type.
- The group efficiency order derived for smectic and nematic on the basis of (a) thermal stability (b) early commencement of mesophase and (c) total mesophase lengths are as under.

(1) Smectic



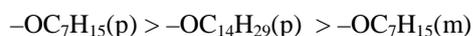
Nematic



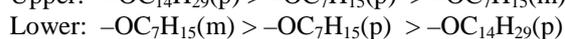
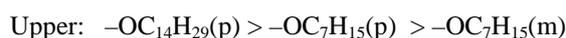
(2) Smectic



Nematic



(3) Sm + N



- Mesomorphism is very sensitive and susceptible to the molecular structure based on positional substitution of a functional group and its polarizability.
- Mesomorphism is depended on suitable magnitudes of molecular rigidity and flexibility.
- Present investigation may be useful for LC devices, Their pharmaceutical or biological activity may be exploited for pharmaceutical preparation and agricultural production to reduce the consumption of insecticides and pesticide as they are antibacterial and antifungal.
- Homologous series of novel chalconyl derivatives are predominantly nematogenic and partly smectogenic whose total mesophase lengths vary between 09.0 to 23.0 C⁰ and low melting type of series.
- Present study supports and raises credibility to the conclusions drawn earlier.

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