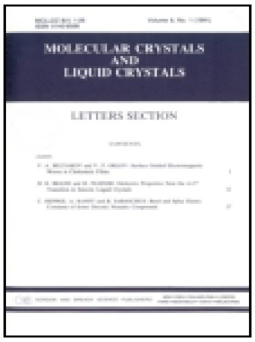

Molecular Crystals and Liquid Crystals

ISSN: 1542-1406 (Print) 1563-5287 (Online) Journal homepage: http://www.tandfonline.com/loi/gmcl20

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To cite this article: Priya K. Patel & R. R. Shah (2016) Dependence of thermotropic mesomorphism on flexible tail group, Molecular Crystals and Liquid Crystals, 630:1, 130-138, DOI: 10.1080/15421406.2016.1146933

To link to this article: http://dx.doi.org/10.1080/15421406.2016.1146933

MOLECULAR CRYSTALS AND LIQUID CRYSTALS
2016, VOL. 630, 130–138
http://dx.doi.org/10.1080/15421406.2016.1146933

Dependence of thermotropic mesomorphism on flexible tail group

Priya K. Patel and R. R. Shah

Chemistry Department, K. K.Shah Jarodwala Maninagar Science College, Gujarat University, Ahmedabad, Gujarat, India

ABSTRACT

A novel homologous series RO-C_6H_4-COO-C_6H_4-CO-CH:CH-C_6H_4-$OC_{12}H_{25}$ of chalcones have been synthesized and studied with a view to understand and establish the effects of molecular structure on liquid crystal (LC) properties of a substance. Liquid crystal properties of a novel series commences from C_6 homologue and continue upto the last C_{18} homologue derivative of a series, as enantiotropically nematogenic without exhibition of smectogenic characteristic. The rest of the homologues (C_1 to C_5) are nonliquid crystals (NLC). Transition temperatures of homologues (C_1 to C_{18}) are determined by an optical polarizing microscopy equipped with a heating stage. Textures of a nematic phase are threaded or schlieren. Transition curves of a phase diagram behaved in normal manner except for C_{18} member of a series, which drastically behaved with abnormal manner. N-I transition curve exhibits odd–even effect. Average thermal stability for nematic is 110.25°C and mesophase length ranges from 10°C to 39°C. Analytical and spectral data supported the molecular structures of homologues. LC properties of present novel series are compared with structurally similar other known homologous series. Thus, present novel series of chalcones is partly nematogenic and of middle-ordered melting type.

KEYWORDS

Liquid Crystal; mesomorphism; nematic; odd-even effect; smectic

Introduction

The study of an intermediate state between crystalline solid and isotropic liquidous state, which is called as anisotropic liquid or liquid crystalline (LC) state [1] has become popular among the scientific and technological community to carry out research with different aims, objects and views at different angles including applications [2–5]. Every field of researchers always need novel LC materials for their novel investigation, either as thermotropic LC or lyotropic LC. Present investigation is planned with a view to understand and establish the effects of molecular structure on LC properties [6–9] of thermotropic LC, excluding the biological study of novel LCs in lyotropic condition. However, the present study will include the synthesis of novel chalconyl ester derivatives, whose, molecular structures will be characterised and to evaluate thermotropic mesogenic properties. Then, the mesogenic properties of proposed novel series will be compared with structurally similar series with reference to changing flexibility of tail end groups, keeping molecular rigidity unchanged. Results of the novel investigation will be recorded and discussed on the basis of molecular rigidity and

CONTACT Priya K. Patel ✉ pp2541991@gmail.com Chemistry Department, K. K. Shah Jarodwala Maninagar Science College, Gujarat University, Ahmedabad, Gujarat,India.

flexibility [10–14]. Number of ester derivatives have been reported till the date [15–21] but present novel series of chalconyl derivatives are important as biologically active compounds which may be studied separately as lyotropical by pharmacist and medicinal chemist [22–26].

Experimental

Synthesis

4- Hydroxy Benzoic acid was alkylated using suitable alkylating Agent (R-X) to convert it into 4-n alkoxy benzoic acids by modified method of Dave and Vora [27].

α-4-hydroxyl benzoyl β-4′-dodecyloxy phenyl ethylene (B) was prepared by usual established method [28]. Acid chlorides component of each 4-n-alkoxy benzoic acid (A) were condensed with α-4′-hydroxyl benzoyl β-4-dodecyloxy phenyl ethylene (B) in dry cold pyridine by usual established method [29] carefully. Thus, chalconyl ester homologue derivatives were finally decomposed, filtered, washed, dried and purified till constant transition temperature obtained using an optical polarising microscope, equipped with a heating stage. 4-Hydroxy benzoic acid, Alkyl halides, Thionyl chloride, MeOH, EtOH, KOH, Acetone, Pyridine, 4-Hydroxy acetophenone, 4- Hydroxy benzaldehyde, Dodecyl halide 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.

Characterization

Selected representative homologues of a titled novel series were characterised by IR, ^{1}HNMR technique and elemental analysis (Table 1). IR spectra were recorded on Perkin Elmer spectrum GX and ^{1}HNMR spectra were recorded using $CDCl_3$ as solvent. Liquid Crystal properties i.e. transition and melting temperatures of homologues were investigated by polarising optical microscopy with heating stage. Textures of the novel homologues were determined by miscibility method (Table 2). Thermodynamic quantities enthalpy (ΔH) and entropy (ΔS) are qualitatively discussed.

Analytical data

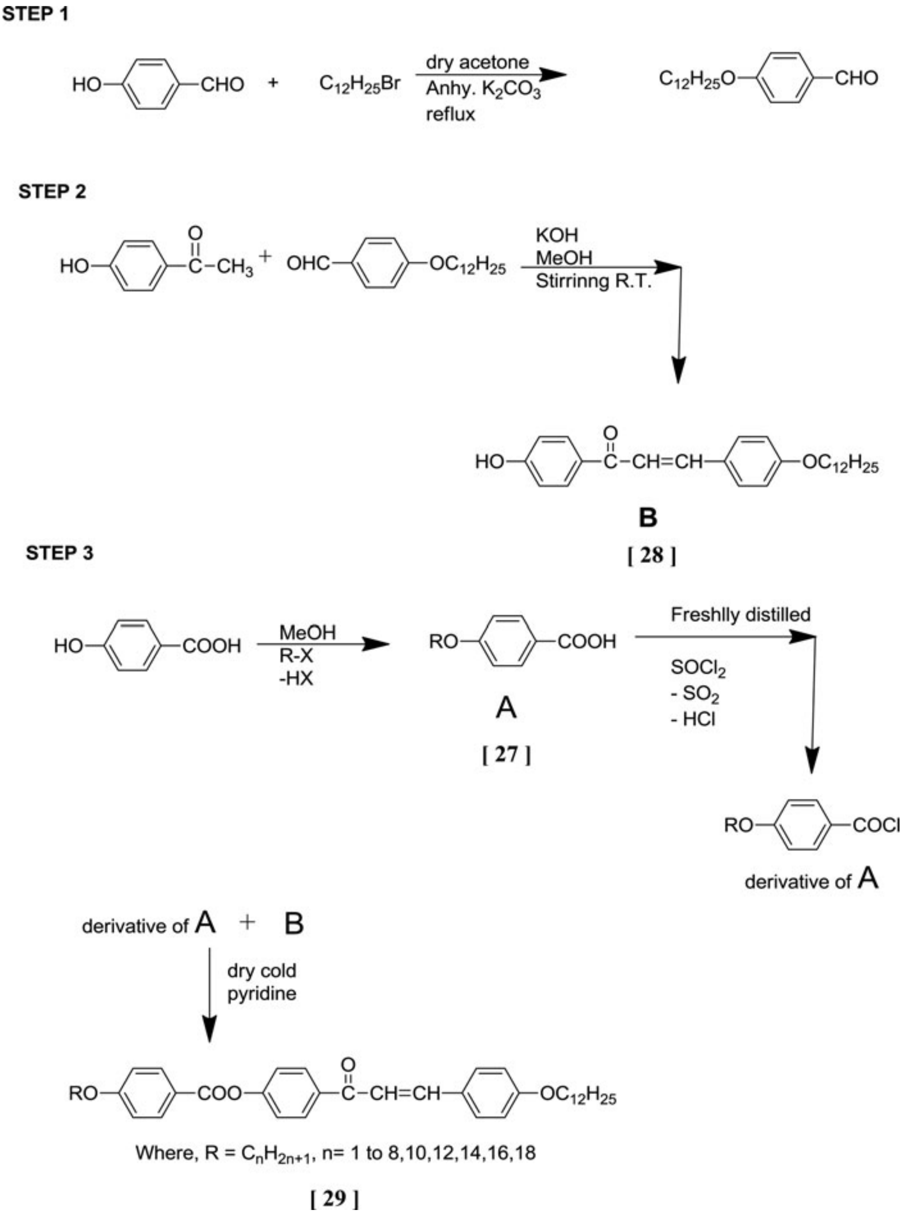
IR spectra in cm^{-1}: For octyloxy and octadecyloxy derivatives

Octyloxy: 763(polymethylene $(-CH_2-)_n$ of $-OC_8H_{17}$, 839 (–OH def., m,p– di substituted), 954 (–CH def. hydrocarbon), 1024 & 1060 (–C–O stre.), 1116,1164,1253 (–C–O stre. in $(-CH_2)_n$ chain),1500 & 1590 (–C–C stre.), 1676 ($-C=O$ group), –COO ester group, 2856 & 2923 (–CH str. in $-CH_3$).

Octadecyloxy:707, 759 (polymethylene $(-CH_2-)_n$ of $-OC_{18}H_{37}$, 842 (–OH def., m,p – disubstituted), 939 (–C–H def. hydrocarbon), 1097 & 1058 (–C–O stre.), 1305, 1253 & 1176 (–C–O stre. in $(-CH_2)_n$ chain), 2921, 2858, 2696 (–C–H stre.), 3087 (= C–H stre.)

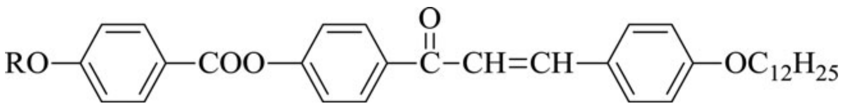
^{1}H– NMR spectra in $CDCl_3$ in δ ppm: For pentyloxy and decyloxy derivatives

Pentyloxy: 0.61, 0.67, 0.83 (t, $-CH_3$ of $-OC_5H_{11}$), 1.08 (m, n-polymethylene group of $-OC_5H_{11}$), 2.00 (s, $-COCH_3$ group), 2.39 (s, $-CH_3$ group), 3.46 to 3.78 (s, $-OCH_2-CH_2$ of $-OC_{18}H_{37}$), 4.3 to 4.4 ($-CH_3$), 8.30 (s, p-disubstituted phenyl ring).



Scheme 1. Synthetic route of the series-1.

Decyloxy: 0.99 (t, $-CH_3$ of $-C_{10}H_{21}$), 1.14 to 1.00 (m, n-polymethylene group of $-OC_{10}H_{21}$), 1.91(m, n-polymethylene group of $-OC_{18}H_{37}$), 2.82 to 2.08 (Ar−C−H), 11.29 (s, p-disubstituted phenyl ring).



Homologous series: α-4 [4′-n-alkoxy benzyloxy] benzoyl β-4″ dodecyloxy phenyl ethylenes.

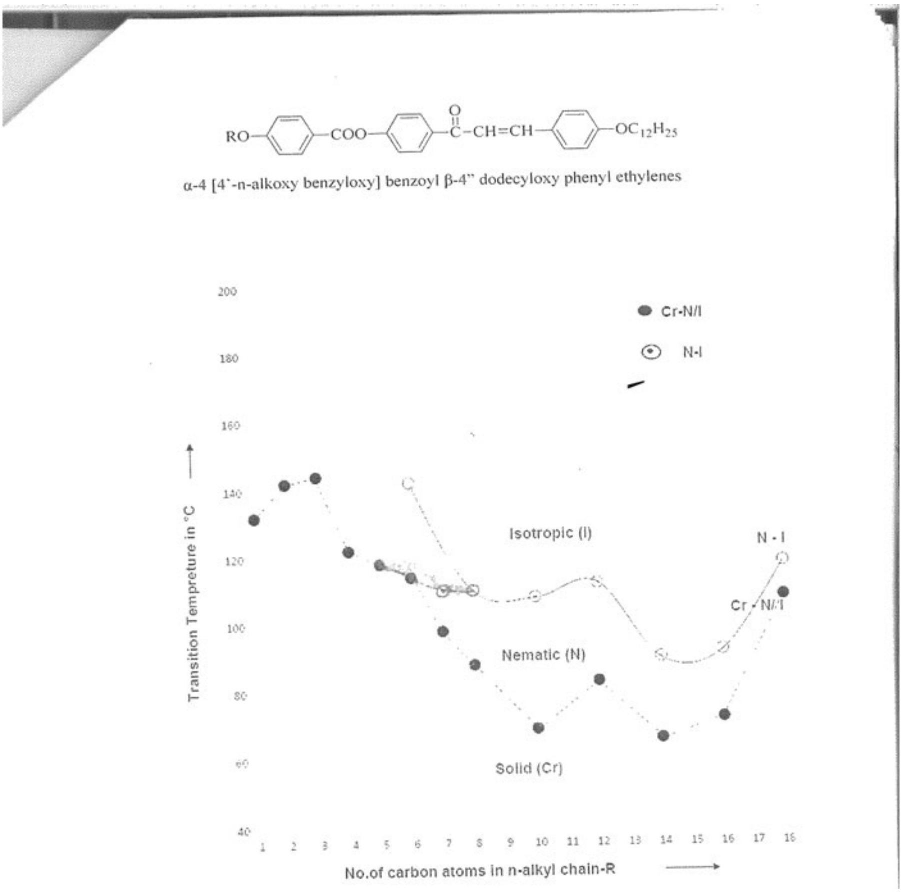


Figure 1. Phase Behaviours of Series.

Results and discussion

α-4-Hydroxy benzoyl β-4′-Dodecyloxy phenyl ethylene is a nonliquid crystal (M.P. is 92°C and yield is 65%) component, which on condensation with dimeric 4-n-alkoxy benzoic acids yielded nematogenic mesomorphs from C_6 homologue to C_{18} homologue with relatively lower transition, than corresponding 4-n-alkoxy benzoic acids. Transition temperatures (Table 1) of homologues were plotted against the number of carbon atoms present in n-alkyl chain bonded to first phenyl ring through oxygen atom. A phase diagram (Fig. 1) consisted of Cr-N/I and N-I transition curves which are formed on linking, like or related points showing phase behaviours of series. Cr-N/I transition curve adopts a zigzag path of rising and falling with overall descending tendency and behaved in normal manner. N-I transition curve descended from C_6 to C_{16} homologues and behaved in normal manner, but it drastically rises for C_{18} and showed abnormal behaviour. N-I transition curve exhibited late odd-even effect in very short range of temperature. Transition curves (N-I) for odd and even members of a series merged into each other at the octyloxy (C_8) homologue and then it propagated as a single curve. The mesomorphic properties are absent from C_1 to C_5 homologues. N-I transition curve for odd members occupy lower position than even member's N-I transition curve. N-I transition curve is extrapolated from C_7 to C_5 to determine its nonmesogenic behaviour. The variations in mesogenic properties are observed from homologue to homologue in the same series. Lowering of transition temperatures of novel chalconyl homologue derivatives and the disappearance of dimerization of 4-n-alkoxy benzoic acids are attributed to the breaking of hydrogen bonding between two molecules of corresponding n-alkoxy benzoic acid by esterification process, through their acid chlorides. The nonmesogenic behaviour of C_1 to C_5 homologues is attributed to their high crystallising tendency, which arises from low dipole-dipole interactions and the low magnitudes of dispersion forces through interactions between instantaneous dipole produced by the spontaneous oscillations of electron clouds of the molecules. Hence, the combine effect of molecular rigidity and flexibility fail to induce mesomorphic property but abruptely breaks the crystal lattices and sharply transform rigid crystals into isotropic state without passing through LC state. The exhibition of nematogenic character by C_6 to C_{18} homologues is attributed to the suitable magnitudes of end to end anisotropic forces of intermolecular attractions and closeness caused as a consequence of favorable molecular rigidity and flexibility which maintained the floating of the molecules on the surface with statistically parallel orientational order only within definite range of temperature under exposed thermal vibrations. However on further continued heating from and above the isotropic temperature, the molecules are randomly oriented in all possible directions with high order of disorder or high entropy [$\Delta S = \Delta H/T$]. But on cooling the same with proper care, the molecules of a homologue under microscopic examination reversibly adopt reappearance of nematic mesophase formation from and below isotropic temperature. However none of the nonmesomorphic (C_1 to C_5) homologues did show exhibition of nematic mesophase in irreversible manner as monotropic mesophase. The exhibition of odd–even effect is observed due to odd and even number of methylene units present in homologues at left n-alkoxy terminal end group. The disappearance of odd-even effect from and beyond C_8 homologue in N-I transition curve is due to the coiling or bending or flexing or coupling of longer n-alkyl chain with the principal axis of the core structure of a molecule. The abnormal behaviour of C_{18} homologue may be attributed to the uncertainity in the status of the longest n-alkyl chain of the left n-alkoxy end group. The extrapolation of N-I transition curve [30–32] from C_7 to C_5 which matches to isotropic temperature of C_5 indicating that, there is no possibility of nematic mesophase formation either in monotropic or enantiotropic condition. The variations in mesomorphic properties or behaviour from homologue to homologue in the same series is attributed to the changing member of carbon atoms which increases gradually the molecular length, length to breadth ratio, permanent dipole moment across the long molecular axis, unsuitable or suitable magnitudes of intermolecular end to end anisotropic forces or dispersion forces of cohesion and closeness, molecular polarity and polarizability as a consequence of resultant molecular rigidity and flexibility etc., keeping the rest of the molecular part unchanged. The absence of smectogenic property in presently investigated novel series may be due to the absence of lamellar packing of molecules in crystal lattices of rigid crystals which hinders the sliding layered molecular arrangement under floating condition on the surface. The extent of molecular noncoplanarity occured due to longer -$OC_{12}H_{25}$ right sided end group, which do not facilitate layered structure in crystal lattices of any of the homologues of present series. Thus, smectogenic mesophase does not occure. The mesogenic behaviours of presently investigated series-1 are compared with the structurally similar other known homologous series as shown in following figure-2.

Homologous series 1 of present investigation and the series X [33] and Y [34] selected for comparative study are identical with respect to three phenyl rings bonded through two central groups –COO– and –CO–CH = CH– and left n-alkoxy terminal end group (–OR) for the same homologue from series to series. But they differ from each other with respect to changing right terminal tail end group –$OC_{12}H_{25}$, –H and m,p-dimethoxy groups for the same homologue from series to series. Thus, variations in mesogenic properties and the degree of

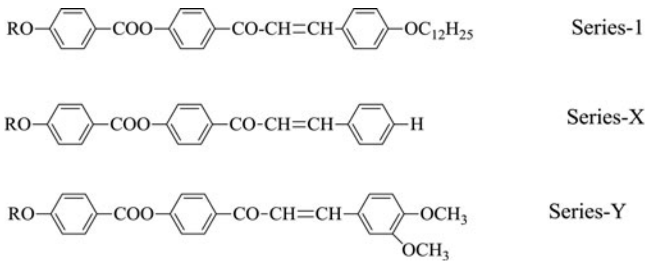


Figure 2. structurally similar series.

Table 1. Elemental analysis for butyloxy, pentyloxy, hexadecyloxy, and octadecyloxy.

	Element % found		Element % calculated	
Molecular formula	C	H	C	H
$C_{38}H_{48}O_5$	78.08	8.21	77.04	7.26
$C_{39}H_{50}O_5$	78.26	8.36	76.25	8.00
$C_{50}H_{72}O_5$	79.78	9.57	77.95	8.98
$C_{52}H_{76}O_5$	80.00	9.74	79.15	9.00

mesomorphism will depend upon changing tail groups from series to series. Table 3 represents some thermometric properties of series 1, X and Y as mentioned below.

Table 4 indicates that,

- Homologous series-1 of present investigation and a series- X are only nematogenic and a series-Y is smectogenic in addition to nematogenic.
- The smectogenic mesophase formation does not commence till the last member of series 1 and X, but it commence from C_6 member of a series-Y.
- Nematic mesophase commences from C_6 member of the Series 1 and X, but it commences early from second member (C_2) of series-Y.
- Nematic thermal stability of the series under comparative study are in the decreasing order as series-X $>$ Y $>$ 1. i.e. thermal stability for nematic is the lowest for presently investigated series.
- Smectic mesophase facilitated and stabilized by series-Y only.
- Total mesophase length range wonderfully varies from maximum to minimum between C_{10} to C_{16} or C_{18} last homologues for all the series under comparison. However, numerical values of total phase length ranges differ from series to series.

The observed difference in the mesogenic or physical properties for the same homologue from series to series among the series 1, X and Y are attributed to the flexibility difference of tail end groups $-OC_{12}H_{25}$, $-H$ and m,p-dimethoxy groups, whereas the difference in mesogenic or physical properties from homologue to homologue in the same series is attributed to the changing molecular flexibility due to sequentially added methylene unit or units at n-alkyl chain of left terminal keeping tail end groups unchanged for each structurally similar series

Table 2. Textures of nematic phase of C_5, C_8, C_{10}, C_{18} by miscibility method.

Sr. No.	Homologue	Texture
1	C_5	Schlieren
2	C_8	Threaded
3	C_{10}	Threaded
4	C_{18}	Schieren

Table 3. Transition temperatures in°C.

		Transition Temperatures in°C		
Compound No.	Homologue(n-alkyl chain)	Smectic	Nematic	Isotropic
1	C_1	—	—	132.0
2	C_2	—	—	142.0
3	C_3	—	—	144.0
4	C_4	—	—	122.0
5	C_5	—	—	118.0
6	C_6	—	114.0	142.0
7	C_7	—	98.0	110.0
8	C_8	—	88.0	110.0
9	C_{10}	—	69.0	108.0
10	C_{12}	—	83.0	112.0
11	C_{14}	—	66.0	90.0
12	C_{16}	—	72.0	92.0
13	C_{18}	—	108.0	118.0

because molecular rigidity from series to series and homologue to homologue in the same series are constant throughout due to three phenyl rings and two central bridges. The changing magnitudes of molecular flexibility depends upon changing tail end group polarities and polarizability or the difference of group polarities of terminal, including lateral groups, atomicity of an individual tail group, and the vector sum of its bond polarities, dipolarities of phenyl ring-tail group, status of tail end group under exposed thermal vibrations, modes of vibrations depending upon the number of degrees of freedom (3n–5) or (3n–6), where n is total number of atoms of flexible specy, etc. m,p-Dimethoxy group are highly polar and polarizable (series-Y) groups, the one of them meta substituted methoxy group is laterally substituted group which induces polarizability of a molecule and the para substituted methoxy group strengthen and induces end to end attractions. Thus the suitable magnitude of anisotropic forces of cohesion and closeness commences from C_2 homologue derivative as nematogenic masophase and from C_6 homologue induces smectogenic mesophase formation in case of series-Y. The lateral methoxy group contributes to layered or sliding layered molecular arrangement under floating condition to induce smectogenic character in addition to nematogenic character. The absence of lateral substitution at ortho or meta position like series-Y do not induce suitable magnitudes of intermolecular polarizable nature of attractions except suitable magnitudes of end to end cohesion and closeness, which hinders the smectogenic mesophase formation but favours to induce only nematogenic mesophase formation identically to commence from C_6 member of the series 1 and X. The lowest value of nematic thermal stability of present novel series-1 is attributed to uncertainity arising in the status of longest n-alkyl chain ($-C_{12}H_{25}$) of tail end group bonded to phenyl ring through oxygen atom. The highest thermal stability for

Table 4. Thermal stabilities in°C.

Series →	1($-OC_{12}H_{25}$)	X(-H)	Y(-m,p- dimethoxy)
Smectic-IsotropicOrSmectic-NematicCommencement of Smectic phase	—	—	115.6(C_6-C_{14})C_6
Nematic-Isotropic commencement of nematic phase	110.25(C_6-C_{18})C_6	164.5(C_6-C_{16})C_6	156.6(C_2- C_{16})C_2
Total mesophase length Range in°C t_i to t_j c_i to c_j	10.0 to 39.0C_{18} C_{10}	07.0–45.0C_{16} C_{10}	36.0–67.0C_{16} C_{10}

nematic of series-X is attributed to the absence of tail end group (or -H) in which molecular flexibility in addition to molecular rigidity are sufficient to induce nematogenic mesophase formation relatively at higher transition temperatuters as compared to series 1 and Y. Vector sum of the bond polarities, strengthens the molecular rigidity or enthalpy (ΔH) value due to absence of tail end group (or by Hydrogen atom) in case of series-X under comparison. Thus, inter molecular cohesion and closeness vary for the same homologue from series to series with changing tail end group which consequently causes variations in molecular rigidity, flexibility, polarity and polarizibility, dispersion forces, permanent dipole moment across the long molecular axis, dipole—dipole, and electronic interactions etc. Thus, present novel chalconyl ester homologous series is partly nematogenic with low degree of mesomorphism and middle-ordered melting type.

Conclusions

- Chalconyl ester derivatives of thermotropic liquid crystals are nematogenic without exhibition of smectogenic character, with low degree of mesomorphism and low thermal stability.
- Smectic and nematic group efficiency order derived on the basis of (a) thermal stability (b) early commencement of mesophase and (c) the total mesophase length with respect to tail end group.
  (a) **Smectic**
  m, p-dimethoxy > $-H = -OC_{12}H_{25}$
  Nematic
  $-H >$ m, p dimethoxy > $-OC_{12}H_{25}$
  (b) **Smectic**
  m, p-dimethoxy > $-H = -OC_{12}H_{25}$
  Nematic
  m, p-dimethoxy > $-H = -OC_{12}H_{25}$
  (c) Total mesophase length range – (Sm + N)
  $-H >$ m, p-dimethoxy > $-OC_{12}H_{25}$
- Molecular flexibility operates mesomorphism, if molecular rigidity is constant.
- The phenomina of mesomorphism is very sensitive and susceptible to a molecular structure.
- Thermotropic chalconyl ester derivatives may be useful in the manufacture of LC devices using study of binary systems. Chalconyl ester derivatives are antibacterial, antimalarial, anticancer etc. which may be studied by biologists through lyotropic mesomorphism.
- Present investigation of novel substances has raised the reliability and credibility to the conclusions drawn earlier.

Acknowledgments

Authors are thankful to the management of K. K. Shah Jarodwala Maninagar Science College, Ahmedabad and Head of Chemistry Department Dr. R. B. Patel. Authors are also thankful to Dr. A.V. Doshi, Ex-Principal, M. V. M. Science and Homesci. College, Rajkot and Dr. Hemant R. Prajapati, Assi. Prof., C. U. Shah Science college, Ahmedabad for their whole hearted support and co-operation as and when needed during course of this investigation. Thanks are due to NFDD Centre, Saurashtra University, Rajkot for Analytical Services.

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