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B. B. Jain & R. B. Patel

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Dependence of mesomorphism on molecular rigidity of nonlinear and linear isomeric and nonisomeric chalconyl esters

B. B. Jain and R. B. Patel

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

ABSTRACT

The novel thermotropic liquid crystalline (LC) materials have been synthesized and studied through chalconyl ester nonlinear (meta homologous series $RO-C_6H_4-CH=CH-COO-C_6H_4-$ CO-CH=CH-C₆H₄-OC₁₄H₂₉ (n) with a view to understand the effect of molecular structure on LC behavior with reference to determine the difference between linear and nonlinear shaped molecules. Novel series consisted of thirteen members, whose nematogenic enantiotropic mesomorphism commences from C_6 homologue and continued up to C₁₈ homologue with the absence of smectogenic character. C₁ to C₅ homologues are nonliquid crystals as determined through an optical polarising microscope (POM) equipped with a heating stage. Transition temperatures of LC homologues alternate and are lower than the corresponding n-alkoxy cinnamic acids. Cr-N/I and N-I transition curve behaved in normal manner. N-I transition curve exhibited odd-even effect in a phase diagram. Textures of nematic phase are threaded or Schlieren or determined by miscibility method and from observing mesophase on the top of the POM. Thermal stability for nematic is 92.5 and mesophase length minimum to maximum is 19.0 to 32.0°C at the C_{10/18} and C₆ homologue. Group efficiency order on the basis of thermal stability is $\frac{-\text{COO}}{\text{linear}} > \frac{-\text{CH}=\text{CH}-\text{COO}}{\text{linear}} > \frac{-\text{CH}=\text{CH}-\text{COO}}{\text{Nonlinear}}$ Thus, novel series is nematogenic and low melting type. Analytical and spectral data confirmed the molecular structures of homologues.

KEYWORDS

Nematic; smectic; mesomorphism; enantiotropy; liquid crystals

Introduction

Increasing utility of LC materials [1] in the benefits of mankind [2–10] especially with –COO- or –CH=CH–COO- central bridges in combination with –N=N-, -CO–CH=CH–or –CH–CH–CO– chalconyl groups because an ester group may improve the quality of agricultural products and chalconyl group may reduce the consumption of insecticides or pesticides including manufacture of LC devices. Therefore present investigation is planned with a view to understand and establish the effects of molecular structure [11–15] on LC properties with reference to central bridges –CH=CH–COO– in linear and nonlinear isomeric vinyl carboxy central bridge with common chalconyl group as central group. The novel investigation will include synthesis, Characterization by elemental analysis, IR and H¹NMR spectra, thermal study by hot stage polarizing microscopy, LC texture



determination, mass spectra including qualitative approach to thermodynamic quantity enthalpy and entropy instead of DSC scan or DSC scan. Then, evaluated thermometric data will be compared with structurally similar analogous series. Evaluated thermal data will be interpreted and discussed in terms of molecular rigidity and flexibility [16–19]. Number of ester homologous series are reported till the date.[20–30].

Experimental

Synthesis

Trans 4-n-alkoxy cinnamic acids (A) were prepared by modified method [31,32]. α -3-Hydroxy benzoyl β -4/tetradecyloxy ethylene (B) was prepared by usual established method [33]. Esters were synthesized by a literature method [34]. Thus, the Chalconyl—ester homologue derivatives were decomposed filtered, washed with solution of sodium bicarbonate and sodium hydroxide followed by water dried and purified till constant transition temperatures obtained using an optical polarising microscope equipped with a heating stage. Alkyl halides, 4- hydroxybenzaldehyde, 3-hydroxy acetophenone, dicyclohexylcarbodimide, Dimethyl amino pyridine, DCM, Ethanol, Acetone required for synthesis were used as received except solvents which were dried and distilled prior to use. The synthetic route to a series is mentioned below in Scheme 1.

Reaction scheme

Characterization

Selected members of the novel homologous series were characterized by Elemental Analysis, infrared spectroscopy, 1H NMR spectra. IR spectra were recorded by Perkin-Elmer spectrum GX, 1H NMR spectra were recorded on Bruker using CDCl $_3$ as solvent. Microanalysis was performed on a Perkin-Elmer PE2400 CHN analyzer. Transition temperature and LC properties(Textures) were determined using an optical polarizing microscopy equipped with a heating stage. Textures of nematic phase determined by miscibility method. Thermodynamic quantity enthalpy (ΔH) and entropy (ΔH) are qualitatively discussed instead of DSC scan.

Analytical Data

IR Spectra in cm⁻¹ for Hexyloxy & Tetradecyloxy Derivatives:

Hexyloxy: 962 (Trans CH=CH), 723 Polymethylene ($-CH_2$ -)n of $-OC_6H_{13}$, 835 (-C-H-def. m di-substituted), 792 Polymethylene ($-CH_2$ -) of $-OC_{14}H_{29}$, 912 (-C-H- def. hydrocarbon), 1112(-C-O-) Str, 1161,1246 and 1390 (-C-O str in $-(CH_2)$ n chain), 1467 (-C-H- def. in CH₂), 1510 (-C=C-)str, 1604,1622 (-C=O and group),1720, 1776 (-COO- group) 2848 and 2915 (-C-H str in CH₃).

Tetradecyloxy: 958(Trans CH=CH), 723 Polymethylene ($-CH_2-$)n of $-OC_{14}H_{29}$, 833 (-C-H- def. m di-substituted), 792 Polymethylene ($-CH_2-$) of $-OC_{14}H_{29}$, 910 (-C-H- def. hydrocarbon), 1112 (-C-O-) Str, 1161,1246 and 1388 (-C-O str in $-(CH_2)$ n chain), 1465 (-C-H- def. in CH_2), 1510 (-C=C-) str, 1600,1662 (-C=O and group), 1777(-COO- group) 2848 and 2918 (-C-H str in CH_3).

Scheme 1. Synthetic route to the series.

¹HNMR spectra in CDCl₃ in δ ppm for Hexyloxy & Tetradecyloxy Derivative:

Hexyloxy: $0.85(t, -CH_3 \text{ of } -C_6H_{13}), 1.1-1.5 \text{ (m, n-poly methylene groups of-}OC_6H_{13}),$ 1.74 (m, n-poly methylene groups of -OC₁₄H₂₉), 3.9(s,-OCH₂-CH₂-ofOC₁₄H₂₉),4.11 $(s, -OCH_2-CH_2-of OC_6H_{13})$ 6.41 (d, -CH=CH-) 6.8-7.5 (s, -CO-CH=CH), 7.8 $(s, p-CH_2-CH_2-of OC_6H_{13})$ disubstituted phenyl ring).

Tetradecyloxy: 0.82 (t,-CH₃ of -C₁₄H₂₉), 1.2-1.5 (m, n-poly methylene groups of- $OC_{14}H_{29}$),1.73 (m, n-poly methylene groups of $-OC_{14}H_{29}$),3.3-3.7 (s,-OCH₂-CH₂-of $OC_{14}H_{29}$), 4.13 (s,-OCH₂-CH₂-of $OC_{14}H_{29}$), 6.5-6.8 (d,-CH=CH-) 7.1-7.5 (s,-CO-CH=CH), 7.83 (s, p-disubstituted phenyl ring).

 α -3-(4'-n-alkoxy cinnamoyloxy) benzoyl- β -4"-tetradecyloxyphenyl ethylenes

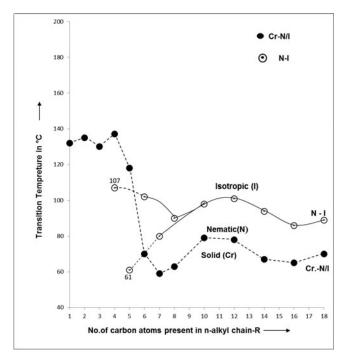


Figure 1. Phase behaviors of series.

Results and discussion

Novel nonlinear series of chalconyl derivatives synthesized by condensation of trans 4-nalkoxy cinnamic acids and α -3-Hydroxy benzoyl β -4' -n- tetradecyloxy phenyl ethylene (m.p. : 72°C and yield : 66.54%). Series consist of thirteen (C_1 to C_{18}) homologues of which C_1 to C_5 homologues are nonliquidcrystals and C₆ to C₁₈ homologues are enantiotropically nematogenic without exhibition of smectic property. Transition and melting temperatures were determined through an optical polarising microscopy equipped with a heating stage and were plotted versus the number of carbon atoms present in n-alkyl chain R of left -OR group. Transition curves, Cr-N/I and N-I were drawn by linking like or related transition points as depicted in a phase diagram (Figure 1) showing phase behavior of series. Cr-N/I transition curve follow zigzag path of rising and falling with overall descending tendency and behaved in normal manner. N-I transition curve initially descended from C₆ to C₈ through C_7 homologue and then ascended to C_{12} homologue through C_{10} and finally descended up to last C_{18} homologue with exhibition of odd-even effect. Thus, it behaves in normal manner. Odd-even effect is observed between C₆ to nearby C₉ homologues and then it disappears from and beyond C₉ homologue for higher homologues of longer n-alkyl chain 'R' of -OR group. N-I transition curve of odd members occupy lower position then even members. Textures of nematic phase are threaded or Schlieren. Analytical, spectral and thermometric data supported the molecular structures of homologues. LC properties from homologue to homologue in the same series varied keeping right terminal and geometrical shape unchanged throughout the series, except number of changing number of methylene unit or units. The lowering of transition temperatures of novel final chalconyl ester compounds as compared to corresponding dimeric cinnamic acids is due to the breaking of hydrogen bonding between acid molecules through esterification process. Alternation of transition temperatures is attributed to the odd and even number of carbon atoms present in n-alkyl chain 'R' of -OR group. The exhibition of only nematic property by the present series is attributed the suitable magnitudes (neither more nor less) of anisotropic forces of intermolecular end to end attractive cohesion and closeness as a consequence of favorable molecular rigidity and flexibility to induce a molecular arrangement in statistically parallel orientational order in floating condition under the influence of exposed thermal vibrations. However combined effect of intermolecular cohesion emerged from two opposing effects of decreasing and increasing intermolecular attractions by molecular polarity and polarizability respectively for widening molecular width which play an important role in facilitation and predomination of effective intermolecular cohesion which is sufficient and suitable to induce nematic mesophase formation but the magnitudes of the same is insufficient and unsuitable to induce smectogenic character as depending upon magnitudes of dispersion forces and dipole-dipole interactions. The nonmesomorphicity of C₁ to C₅ homologues is attributed to the low magnitudes of dispersion forces and low magnitudes of dipole-dipole interaction as well as electronic interactions which induces high crystallizing tendency and abruptly breaking crystal lattices which sharply transform into isotropic liquid without passing through an intermediate physical state of existence called as LC state. However on cooling the same from and below isotropic temperature neither the nematic or smectic phase from nonmesomorphs (C_1 to C_5) nor the smectic phase from enantiotropic nematogenic homologues observed in irreversible manner. Thus, monotropic transitions either for nonmesomorphs (C_1 to C_5) or nematogenic homologues (C_6 to C_{18}) are not observed for present novel series. The disappearance of odd-even effect from and beyond nearby C₉ homologues for N-I transition curve is attributed to the coiling or bending, or flexing or coupling of longer n-alkyl chain of -OR and $-OC_{14}H_{29}$ (n) end groups with the major axis of core structure of a molecule. N-I transition curve for odd and even homologues are extrapolated [35,36,37,38] to C₅ and C₄ homologues respectively to highlight odd-even effect and to predict their latent ability to exhibit mesophase formation at 61°C and 107°C respectively, but, due to their high crystallizing tendency, it is not realizable in actual practice. The changing behaviors of LC properties from homologue to homologue in the same series is attributed to the changing number of methylene unit or units which causes variations in the magnitudes of total molecular flexibility keeping molecular rigidity unaltered because rest of the molecular part including tailed end remains unchanged throughout the novel present series. Some LC properties evaluated from thermotropic data of presently investigated novel series-1 and compared with other structurally similar analogous series X [39] and Y [40] as mentioned below in following Figure 2.

Figure 2. Structurally similar analogous series.

Table 1. Flmental Anal	vsis for hentyloxy	decyloxy dodecyloxy	, and hexadecyloxy derivatives.
Table I. Lillicital / lila	y 313 IOI IICPLYIONY	, accylony, abaccylony	,, and nexadecyloxy acrivatives.

		%Elements found		%ElementsTheoretical	
Sr. No.	Molecular formula	С	Н	С	Н
1	C ₄₅ H ₆₀ O ₅	81.87	9.02	81.81	9.09
2	$C_{48}^{43}H_{66}^{60}O_{5}^{3}$	82.01	9.43	82.05	9.40
3	$C_{50}^{\uparrow 0}H_{70}^{0}O_{5}^{3}$	82.23	9.62	82.19	9.58
4	$C_{54}^{30}H_{78}^{70}O_{5}^{3}$	82.48	9.89	82.44	9.92

Table 2. Texture of Nematic Phase of C_7 , C_{10} , C_{14} , C_{16} by miscibility method.

Sr. No.	Homologue	Texture
1	C ₇	Threaded
2	C ₁₀	Threaded
3	C ₁₄	Schieren
4	C ₁₆	Threaded

In the above Figure 2, the homologous series-1 of present investigation and a series-X, selected for comparison are isomeric to each other but due to their linearity difference, their magnitudes of combined effects of molecular rigidity and flexibility acquire difference by positional difference of bonding –CH=CH–COO– central group to middle phenyl ring. Homologous series 1 and X differs from series –Y with respect to a central bridges vinyl carboxy and carboxy groups linking first and middle phenyl rings. Therefore magnitudes of molecular rigidity and flexibility differs from homologue to homologue in the same series and the magnitudes of only rigidity undergo variations but molecular flexibility remains unaltered for the same homologue from series to series. Thus, variations in mesomorphic properties like thermal stability, commencement of mesophase, mesophase lengths etc will attend and depend upon the magnitudes of changing features of homologous series 1,X and Y under comparative study. Following table 4 represents some thermometric data for the series under comparison.

From table 4,

- Homologous series 1, X and Y are nematogenic only with absence of smectic property.
- Thermal stability of series 1, X and Y are in increasing order.
- The nematogenic mesophase commences in equal manner for isomeric series 1 and X (C_6) but it commences late by one more (C_7) homologue in case of series-Y.

Table 3. Transition temperature of homologous series.

Sr. no			Transition temperatures in $^\circ$ C	-
	R = n-alkyl group	Smectic	Nematic	Isotropic
1	C1	_	_	132.0
2	C2	_	_	135.0
3	C3	_	_	130.0
4	C4	_	_	137.0
5	C5	_	_	118.0
6	C6	_	70.0	102.0
7	C7	_	59.0	80.0
8	C8	_	63.0	90.0
9	C10	_	79.0	98.0
10	C12	_	78.0	101.0
11	C14	_	67.0	94.0
12	C16	_	65.0	86.0
13	C18	_	70.0	89.0

Table 4. Thermal stability in °C.

Series	Series-1 (nonlinear) -CH=CH-COO-	Series-X (Linear isomeric) -CH=CH-COO-	Series-Y (Linear) -COO-
Sm-I or Sm-N Commencement of Smectic phase	_	_	_
N-I Commencement of Nematic phase	92.5 (C ₆ -C ₁₈) C ₆	99.75 (C ₆ -C ₁₈) C ₆	123.0 (C ₇ -C ₁₈) C ₇
Total mesophase lengths from t ₁ °c to t ₂ °c	19.0 to 32.0 C_{10}/C_{18} C_{6}	19.0 to 34.0 C ₆ C ₈	13.0 to 34.0 $C_7 C_8 / C_{10} / C_{12}$

• Total upper mesophase lengths are in increasing order of temperature but lower mesophase lengths are in decreasing order from isomeric series to nonisomeic series.

The exhibition of only nematic property with absence of smectic property is obviously related to the molecular arrangement in floating condition under exposed thermal vibrations as required for emergence of nematic phase which facilitated in all the three series under comparison. But, their thermal resistivity extremely against exposed thermal vibrations vary from homologue to homologue in the same series and for the same homologue from series to series due to changing magnitudes of thermodynamical quantity enthalpy (ΔH), representing energy stored in a molecule as a consequence of combined effect of molecular rigidity and flexibility. The intermolecular cohesion and closeness are affected by intermolecular distance by end to end attractions and molecular polarizablity which are operated more in nonlinear series depending upon predominancy of the intermolecular forces of dispersion and magnitudes of dipole-dipole interactions. The net effect of the suitable magnitudes of dispersive forces in an isomeric linear series-X are stronger than nonlinear series-1 which lowers the transition temperatures of series-1 as compared to linear analogous series-X. Therefore thermal stability of series-1 calculated on the basis of N-I transition temperatures acquire magnitudes of thermal stability. The highest thermal stability of linear series-Y, is due to presence of -COOcentral group in place of -CH=CH-COO- central bridge which more effectively contribute to molecular rigidity and flexibility due to decrease in length by the absence of -CH=CHunit and increased linearity as well as increase suitable magnitudes of intermolecular end to end attractions and closeness. Thus, transition temperatures and hence the thermal stability for nematic is the highest. Thus, increasing order of nematic thermal stability and the upper mesophase lengths are maintained. The commencement of mesophase (nematic) is depended upon the extent of molecular noncoplanarity. The data of commencement of mesophase for isomeric series linear and nonlinear 1 and X suggests the equitable magnitudes (C_6) of early commencement of mesophase which depends on the shapes of molecules irrespective of linear or nonlinear. However, the molecules of linear series-Y, commences its mesophase formation, a little bit later from C₇ homologue due to absence of -CH=CH- unit as compared to series 1 and X. Thus, the presence or absence of -CH=CH- unit disturb or facilitate suitable or unsuitable magnitudes of the extent of molecular coplanarity, thermal stability, the degree of mesomorphism etc.

Conclusions

 A homologous chalconyl ester series of present investigation is nematogenic with absence of smectic property and low degree of mesomorphism as well as of low ordered melting type.



- Nonlinearity of a molecule lowers the transition temperature as compared to an isomeric linear molecule.
- The group efficiency order derived for nematic in combination with -CO-CH=CH-central bridge on the basis of (i) thermal stability (ii) early commencement of mesophase and (iii) total upper and lower mesophase lengths are as under.
 - (i) Nematic

linear nonlinear

(ii) Nematic

nonlinear linear linear

(iii) Total (nematic) Mesophase length

linear linear nonlinear

nonlinear linear linear

- Mesomorphism phenomena is very sensitive and susceptible to molecular structure as a consequence of molecular rigidity and/or molecular flexibility.
- Chalconyl esters of low transition temperature may be useful for LC devices to be operated at room temperature or desired temperature.
- Ester group of present novel investigation may be useful for the agricultural production for the growth of quality and healthy fruits and flowers, potato and potato like material as well as chalconyl group being biologically active and antibacterial and antifungal may reduce the consumption of pesticides and insecticides.
- Present investigation supports previous conclusions.

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