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P. K. Rakhasia, Avani R. Ranchchh & U. C. Bhoya

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# Molecular flexibility dependence on mesogenic behaviors of isomeric and nonisomeric series

P. K. Rakhasia, Avani R. Ranchchh, and U. C. Bhoya

Department of Chemistry, Saurashtra University, Rajkot, Gujarat, India

#### ABSTRACT

A novel homologous series containing vinyl ester and azomethane central bridges and n-alkoxy as well as 3",4"-dimethyl groups as flexible terminal/lateral groups viz.  $RO-C_6H_4-CH = CH-COO-C_6H_4-N=CH-C_6H_3-CH-C_6H$ (CH<sub>2</sub>)<sub>2</sub> have been synthesized and studied with a view to establishing the relation between molecular structure and thermotropic liquid crystal (LC) properties with reference to molecular flexibility within the series. The series consists of twelve homologues (C<sub>1</sub> to C<sub>16</sub>). C<sub>6</sub> and C<sub>7</sub> homologues are smectogenic plus nematogenic and C<sub>8</sub> to C<sub>16</sub> homologues are only smectogenic, and the rest of the homologues (C1 to C<sub>5</sub>) are nonmesomorphic. Transition temperatures and the textures of the homologues were determined using an optical polarizing microscope equipped with a heating stage (POM). The textures of a nematic phase are threaded or Schlieren and that of a smectic phase are of the A or C type. Analytical, thermal and spectral data support the molecular structures. Smectic and nematic thermal stabilities are 116.85°C and 147.5°C, respectively. Whose Sm–N/I and N-I mesophase lengths are varied between 15°C to 21°C and 25°C to 62°C, respectively. The novel compounds are compared with structurally-similar series.

#### **KEYWORDS**

Enantiotropy; mesomorphism; nematic; smectic

# Introduction

A physically distinct state of a matter between the two well-known states of crystalline solid and isotropic liquid is called the liquid crystalline (LC) state [1] and it has proved very important [2–5] in thermotropic and lyotropic environments. A wide range of scientific, technological and industrial researchers are working on the unique state of matter of liquid crystals with different, but complementary aims and objectives. One of the very important and basic aims and objectives is the study of the effects of molecular structure on LC properties, which is the focus of the present investigation [6–10]. The aim of study was to synthesis novel LC substances through a homologous series containing three phenyl rings, two central bridges, –CH = CH–COO– and –N=CH– with flexible terminal and lateral groups. Then to characterize these novel materials through thermal, analytical and spectral studies, and evaluate and discuss the thermometric data in terms of molecular rigidity and flexibility [11–14] in comparison with known structurally-similar materials. Several ester homologues series with –N=N–, –CH=N–, –CO–CH = CH– or –CH = CH–CO– groups have been reported to date [15–21].

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CONTACT U. C. Bhoya Sdrucbhoya@gmail.com Department of Chemistry, Saurashtra University, Rajkot 360005, Gujarat, India



Where, R=C<sub>n</sub>H<sub>2n+1</sub>, n=1 to 8, 10, 12, 14, and 16

Scheme 1. Synthetic route to the novel series.

# Experimental

# **Synthesis**

4-n-Alkoxybenzaldehydes were synthesized by refluxing 4-hydroxybenzaldehyde (1 equiv.) with corresponding n-alkyl bromides (1 equiv.) in the presence of potassium carbonate (1 equiv.) and acetone as a solvent [22]. The resulting 4-n-alkoxybenzaldehydes were reacted with malonic acid (1.2 equiv.) in the presence of 1–2 drops piperidine as catalyst and pyridine as solvent to yield corresponding trans-4-n-alkoxy cinnamic acids (A) [23]. 4-Hydroxy amino benzene 3', 4'-dimethyl benzal(B) was prepared by an established method [24] M.P.170–172°C, Yield- 76.5%. Coupling of compound A and B was carried out by a Steglich esterification to yield 4-(4'-n-alkoxy cinnamoyloxy) amino benzene 3", 4"-dimethyl benzal [25].

The synthetic route to the novel homologous series of Schiff's base cinnmoyl esters is shown in Scheme 1.

# Characterization

Some members of the novel series were characterized by elemental analysis (Table 1), infrared spectroscopy, <sup>1</sup>H NMR spectra and mass spectroscopy. Microanalysis was performed on

		Elements %Found			Elements %Calculated		
Sr. No.	Molecular formula	С	Н	0	С	Н	0
1	C <sub>27</sub> H <sub>27</sub> NO <sub>3</sub>	78.49	6.55	11.57	78.42	6.58	11.61
2	$C_{30}^{-1}H_{33}^{-1}NO_{3}^{-1}$	79.15	7.27	10.51	79.09	7.30	10.54
3	C <sub>36</sub> H <sub>45</sub> NO <sub>3</sub>	80.18	8.36	8.85	80.11	8.40	8.89

Table 1. Elemental analysis for (1) Propyloxy (2) Octyloxy (3) Dodecyloxy derivatives.

EuroEA Elemental Analyzer. IR spectra were recorded on Shimadzu FTIR Model -IRAffinity-1S (MIRacle 10), <sup>1</sup>H NMR spectra were recorded on Bruker spectrometer using CDCl<sub>3</sub> as a solvent and mass spectra were recorded on a Shimadzu GC-MS Model No.QP-2010. The liquid crystal behavior and the type of textures were determined by a miscibility method on microscopic observations.

# **Analytical data**

#### Spectral data

#### 1H NMR in ppm for ethyloxy derivative

1.35–1.38 (t,3H,–CH<sub>3</sub> of  $-OC_2H_5$  group), 3.98–4.03 (m,2H,CH<sub>3</sub>-<u>CH<sub>2</sub>-</u>O), 7.44–7.48 (d,1H, –C<u>H</u>=CH–COO–), 6.41–6.45 (d,1H, –CH=C<u>H</u>–COO–), 8.27 (s,1H, –C<u>H</u>=N–), 3.26 (s, 6H, two –CH<sub>3</sub> groups attached to benzene), 6.84–6.88 & 7.87–7.90 (4H, phenyl ring containing ethoxy chain), 7.46–7.48 & 7.76–7.80 (4H, middle phenyl ring), 7.02–7.80 (3H, phenyl ring containing two methyl ring). The NMR data are reliable with the molecular structure.

#### 1H NMR in ppm for pentyloxy derivative

0.85–0.88 (t,3H,–CH<sub>3</sub> of  $-OC_5H_{11}$  group), 1.29–1.32 (m,2H,CH<sub>3</sub>-<u>CH</u><sub>2</sub>–(CH<sub>2</sub>)<sub>3</sub>–O–)1.37–1.39 (p,2H,-<u>CH<sub>2</sub>–</u>CH<sub>2</sub>–CH<sub>2</sub>–O–), 1.70–1.75 (p,2H,-<u>CH<sub>2</sub>–</u>CH<sub>2</sub>–O–), 3.91–3.94 (t,2H,–CH<sub>2</sub>–O–), 7.45–7.47 (d,1H, –C<u>H</u>=CH–COO–), 6.40–6.44 (d,1H, –CH=C<u>H</u>–COO–), 8.27 (s,1H, –C<u>H</u>=N–), 2.19–2.21, (s, 6H, two –CH<sub>3</sub> groups attached to benzene), 7.02–7.04 & 7.75–7.78 (4H, phenyl ring containing ethoxy chain), 7.45–7.47 & 7.75–7.79 (4H, middle phenyl ring), 6.88–7.89 (3H, phenyl ring containing two methyl ring). The NMR data are reliable with the molecular structure.

#### *IR* in cm<sup>-1</sup> for butyloxy derivative

3020 (C-H str. of alkene disubstituted), 2924 & 2866 (C-H str. of  $(-CH_2-)_n$  group of  $-OC_4H_9$ ), 1726 (C=O str. of carbonyl carbon of ester group), 1629 (C=C str. of alkene), 1598 & 1508 (C=C str. of aromatic ring), 1309 & 1265 (C-H bending of alkene), 1203 (C-O str. of ether linkage), 1132 (C-O str. of ester group), 999, 970 & 931 (C-H bending of alkene). The IR data are consistent with the molecular structure.

#### *IR* in cm<sup>-1</sup> for octyloxy derivative

3037 (C–H str. of alkene disubstituted), 2920 & 2850 (C–H str. of  $(-CH_2-)_n$  group of  $-OC_8H_{17}$ ), 1720 (C=O str. of carbonyl carbon of ester group), 1629 (C=C str. of alkene), 1600, 1571 & 1506 (C=C str. of aromatic ring), 1394 & 1319 (C–H bending of alkene), 1246 (C–O str. of ether linkage), 1118 (C–O str. of ester group), 991, 968 & 923 (C–H bending of alkene). The IR data are consistent with the molecular structure.

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Table 2.	Transition	temperatures	in°C
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Compound No.	n-alkyl chain C <sub>n</sub> H <sub>2n+1</sub> (n)	Sm	Ν	lsotropic
1	1	_	_	123.0
2	2	_	_	140.0
3	3	_	_	183.0
4	4	_	_	166.0
5	5	_	_	155.0
6	6	(71.0)	92.0	117.0
7	7	(101.0)	116.0	178.0
8	8	81.0	_	116.0
9	10	72.0	_	128.0
10	12	68.0	_	130.0
11	14	77.0	—	128.0
12	16	82.0	—	108.0

Sm = Smectic, N = Nematic, () indicate monotropy

# Mass spectra of methyloxy derivative

*m*/*z* (rel.int%): 385 (M)<sup>+</sup>, 224, 180, 161, 133, 103, 77

#### Mass spectra of heptyloxy derivative

*m*/*z* (rel.int%): 469(M)<sup>+</sup>, 343, 260, 245, 224, 181, 147, 119, 91, 65

### **Result and discussion**

The homologues from  $C_1$  to  $C_5$  members of the novel series are nonmesomorphic. However, nematogenic and smectogenic mesomorphism commences from the  $C_6$  homologue, but only smectogenic mesomorphism is exhibited by the  $C_8$  to  $C_{16}$  homologues in an enantiotropic manner. Transition and mating temperatures as determined by POM (Table 2) were plotted to give Cr -Sm/I, Sm - N/I, N-I transition curves, showing phase behaviors of the series in a phase diagram (Fig. 1). The Sm-I transition curve exhibits an odd-even effect, which appears up to the C<sub>12</sub> homologue. The N-I curves for odd and even members were extrapolated up to the  $C_{12}$  homologue to show the odd-even effect for the N-I transition curve. The transition curve for odd members occupies the upper position as compared to transition curve for even membered homologues. The Sm-N or Sm-I transition curve initially rises, passes from maxima at  $C_{12}$  homologue and then descends in the usual established manner. The imaginary N-I transition curve showing the odd-even effect is shown by an extrapolation method [26-29]. The Cr-M or Cr-I transition curve adopts a zigzag path of rising and falling values with an overall descending manner of values. Thus transition curves of a phase diagram behaved in expected normal manner. Odd-even effect disappears from and beyond C<sub>12</sub> homologue. The mesogenic property varies from homologue to homologue in the present series.

The lowering of transition temperatures of novel homologues as compared to corresponding dimeric n-alkoxy acids are attributed to the breaking of hydrogen bonding by esterification process. The lack of smectic or/and nematic mesophases either monotropically or enatiotropically by the  $C_1$  to  $C_5$  homologues is attributed to their high crystallizing tendency which arises from low magnitudes of dispersion forces and low magnitudes of dipole-dipole interaction. The mesophase formation by the  $C_6$  to  $C_{16}$  homologues is attributed to the disalignment of molecules, perpendicular or at an angle less than ninety degree to the floaling surface, depending upon thermal resistivity causes by suitable magnitudes of intermolecular dipole–dipole interaction and dispersion forces which induced by favorable end to end attractions or / and lateral attractions of suitable magnitudes as a consequence of their molecular rigidity and



Figure 1. Phase behavior of series.

flexibility, which maintains mainly sliding layered molecular arrangement for all the mesogenic homologues as well as maintain statistically parallel orientational order of molecules under floated condition for C<sub>6</sub> and C<sub>7</sub> homologues. Thus, C<sub>6</sub> to C<sub>16</sub> mesogenic homologues showed smectogenic character, whereas C<sub>6</sub> and C<sub>7</sub> homologues shows appearance of smectogenic and nematogenic mesophase formation, of different textures and of different range of temperatures. All molecules of each mesogenic homologues individually, randomly orient in all possible directions from and beyond isotropic temperature, which on cooling the same from and below isotropic temperature reappears smectic or/and nematicmesophase formation in reversible manner for a temperature range which was appeared on heating. Thus,  $C_6$  to C<sub>16</sub> homologues are enantiotropically mesogenic. Odd-even effect observed for a novel series is due to the sequential addition of methylene unit or units at n-alkyl chain "R" bonded to first phenyl ring through oxygen atom. Decreasing and normal tendency of transition curves observed is attributed to the shortening but suitable molecular length or their permanent dipolemoment across the molecules of which longer n-alkyl chain "R" of -OR group coil or bend or flex or couple to lie with major axis of core structure in expected manner and causes disappearance of -odd-even effect from and beyond C<sub>12</sub> homologue. The variations in mesogenic behavior, properties, degree of mesomorphic from homologue to homologue in present novel series are attributed to the changing number of carbon atom or atoms in n-alkyl chain "R" of left n-alkoxy, -OR group, keeping the rest of the molecular part unaltered of fixed molecular part unaltered of fixed molecular rigidity and changing total molecular flexibility due to left -OR group and fixed flexibility, polarity and polarizability of 3", 4"-dimethyl terminal and lateral groups at the other end. Some mesogenic behaviors and properties of presently investigated novel series 1 are compared with structurally similar analogous series X [30] and Y [31] as mentioned below in Fig. 2.

Homologous series 1 and X are identical with respect to three phenyl ring and two central bridges contributing to the total molecular rigidity and left n-alkoxy -OR terminal contributing partly to the total molecular rigidity for the same homologue from series to series. However they differ with respect to 3",4" dimethyl and  $-OCH_2-CH_3$  terminal and/or lateral



Figure 2. Structurally similar series.

groups for the same homologue from series to series, contributing partly to the total flexibility. Homologous series 1 and Y are isomeric to each other, but their molecular rigidity may differ by very small or of negligible magnitudes due to -N=CH- as well as -CH=N- inversion of central bridge linking middle and third phenyl rings for the same homologues affecting molecular rigidity. The molecular flexibility and the molecular polarizability also differ from each other (1 and Y) due to positional difference of 3'', 4''-dimethyl and 2'', 5''-dimethyl group for the same homologue from series to series. Therefore, the variations in mesogenic properties and the degree of mesomorphism among the series 1, X and Y depend upon the differing features and their magnitudes for the same homologue from series to series some thermotropic LC properties for series 1, X and Y in comparative manner, as under.

Form Table 3, it is clear that,

- Homologous series 1, X and Y are smectogenic in addition to nematogenic.
- Mesomorphism of smectic type commences from C<sub>6</sub> homologue of the series 1 and Y but it commences late from C<sub>12</sub> homologue in case of series X.
- Nematogenic mesomorphism commences from  $C_6$ ,  $C_1$  and  $C_2$  homologue of the series 1, X and Y respectively.
- Thermal stability for smectic and nematic of present series 1 is more than an isomeric series Y, but less than series X.

Series $\rightarrow$	Series 1 (3", 4"-dimethyl)	Series X (–OCH <sub>2</sub> –CH <sub>3</sub> )	Series Y (2", 5"-dimethyl)
Sm—N or Sm—I Commencement of smectic phase N—I Commencement of nematic phase Total mesophase length in°C	$\begin{array}{c} 116.85\\ (C_6 - C_{16})\\ C_6\\ 147.5\\ (C_6 - C_7)\\ C_6\\ 26.0 \text{ to } 77.0\\ C_{16}C_7\end{array}$	$\begin{array}{c} 122.5\\ (C_{12} - C_{18})\\ C_{12} \\ 270.7\\ (C_{1} - C_{18})\\ C_{1}\\ 111.0 \text{ to } 194.0\\ C_{18}C_{7}\end{array}$	$ \begin{array}{c} 100.16\\(C_8 - C_{18})\\C_6\\110.83\\(C_5 - C_{10})\\C_2\\4.0 \text{ to } 41.0\\C_{18}C_{12}\end{array} $

Table 3. Relative thermal stabilities in°C.

• Upper and lower degree of mesomorphism of present novel series 1 is lower than a series X and more than a series Y.

The suitable magnitudes of anisotropic force of dispersion vary with the linearity or nonlinearity performing to the geometrical shape of molecules. Aromaticity and central bridges are identically of series 1, X and Y are same. The molecules of series X are more linear than series 1 and X without lateral substitution. Therefore, intermolecular distance parameter predominated against polarizability faster, affecting the magnitudes of the forces of cohesion for series X, Y and 1. Thus, presence of lateral one -CH<sub>3</sub> group series 1 or two -CH<sub>3</sub> as lateral group affects the possibility and suitability of magnitudes of intermolecular end to end and/or lateral cohesion which causes more or less magnitudes of facilitating and maintaining either only statistically parallel orientational order of molecules at the cost of maintaining sliding layered molecular arrangement or facilitating statistically parallel orientational order of molecular arrangement in addition to sliding layered molecular arrangement like series 1, X and Y of different temperature ranges for the same homologue from series to series; to induce either only smectic or only nematic or both together one after another mesophases or in general mesophases. The molecules of different moieties and of different geometrical shape and size are formed from their atoms of different elements by breaking of odd bonds and forming of new bonds in standard state or at a particular temperature. The change of energy involved in all this process as heat of reaction and heat of formation ( $\Delta H$ ) from atoms of elements in standard state, which differs from homologue to homologue in the same series and for the same homologue from series to series. Therefore, the resistivity towards exposed thermal vibrations for the same homologue from series to series of mesogenic homologue vary in transition temperature, mesophase lengths due to extant of molecular noncoplanarity and thermal stabilities for smectic and nematic. Thus smectic and nematic thermal stabilities early or late commencement of mesophase as well as upper and lower mesophase lengths are in decreasing order as series X > series 1 > series Y. Hence, magnitudes of molecular rigidity and flexibility as depended upon thermodynamic quantities and molecular structure play an important role in mesogenic tendency of individual molecule of same series or of structurally similar analogous isomeric or non isomeric series.

# Conclusions

- Ester homologous series of azomethan central bridge is predominantly smectogenic and partly nematogenic and of middle-ordered meeting type with considerable mesophase lengths.
- The group efficiency order derived for smectic and nematic based on (i) thermal stability (ii) early commencement of mesophase (iii) mesophase lengths are as under.

I. Smectic:  $4''-OCH_2-CH_3 > 3'',4''-dimethyl > 2'',5''-dimethyl$ Nematic:  $4''-OCH_2 - CH_3 > 3'',4''-dimethyl > 2'',5''-dimethyl$ II. Smectic:  $3'',4''-dimethyl = 2'',5''-dimethyl > 4'' - OCH_2-CH_3$ Nematic:  $4''-OCH_2-CH_3 > 2'',5''-dimethyl > 3'',4''-dimethyl$ III. Total mesophase lengths: Upper and lower:  $4''-OCH_2-CH_3 > 3'',4''-dimethyl > 2'',5''-dimethyl$ 

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- Inversion of central group -N=CH- to -CH=N- in isomeric series can cause variations in mesogenic tendency.
- Study of binary systems of present investigation can lower transition temperatures nearby 50°C
- Mesogenictendancy of a substance is very sensitive and susceptible to the molecular structure of a substance.
- Present investigation raises and supported the conclusion drawn earlier.

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