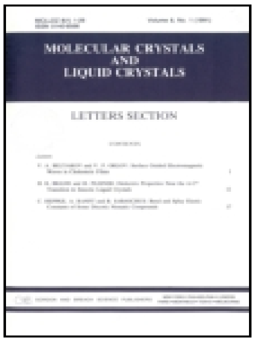

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Mesomorphism of Novel Symmetric Dimers with Changing Alkoxy Terminal End Group

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A novel homologous series of symmetric dimers has been synthesized. It consists of thirteen (C_1 to C_8, C_{10}, C_{12}, C_{14}, C_{16} and C_{18}) dimer ester derivatives from trans cinnamic acid with pyrocatechol. Mesomorphic property commences from C_7 homologue as smectic. C_1 to C_6 members of the series are nonmesomorphic. C_7 is monotropic smectic and the rest of the homologues from C_8 to C_{18} are enantiotropic smectic. The nematic mesophase is totally absent. Textures and transition temperatures of the homologues are determined by an optical polarizing microscope equipped with a heating stage. IR, ^1H NMR spectra, mass spectra, and differential scanning calorimetry of some members is included. The spectroscopic analyses support the molecular structures. Mesomorphic properties of the novel symmetric dimer series are compared with structurally similar series. The novel series is partly nonmesomorphic and predominantly smectogenic with textures of the A or C type, without the exhibition of the nematic phase.

Keywords Liquid crystal; Monotropic; Nematic; Smectic; Symmetric dimers

Introduction

The liquid crystalline (LC) state of matter was first reported in 1888 [1]. Many researchers of various disciplines in science and technology have shared their keen interest with different views, aims and objectives in order to benefit mankind. Chemists have synthesized and reported many novel LC substances [2–5] useful other research groups. The chemists have synthesized novel conventional and unconventional LC and characterized them and further studied the effects of molecular structure on LC property [6, 7] by comparing LC behaviors with other series. Vorlander described LC properties in 'V'-shaped compounds [8, 9]. Over the past 30 years, chemists have shared their increasing interest in unconventional symmetric and asymmetric LC dimers [10]. The present investigation is planned to synthesize symmetric dimers in which, two identical mesogenic units are bonded to a spacer at an angle 60 degrees through a 1,2-dihydroxy benzene (pyrocatechol) unit to construct 'V' shaped novel LC dimers to understand and establish the relations between molecular structure and liquid crystallinity on the basis of molecular rigidity and flexibility [11–15]. Thus, the present study is aimed at fundamental aspects of liquid crystals rather than directed at applications [16, 17].

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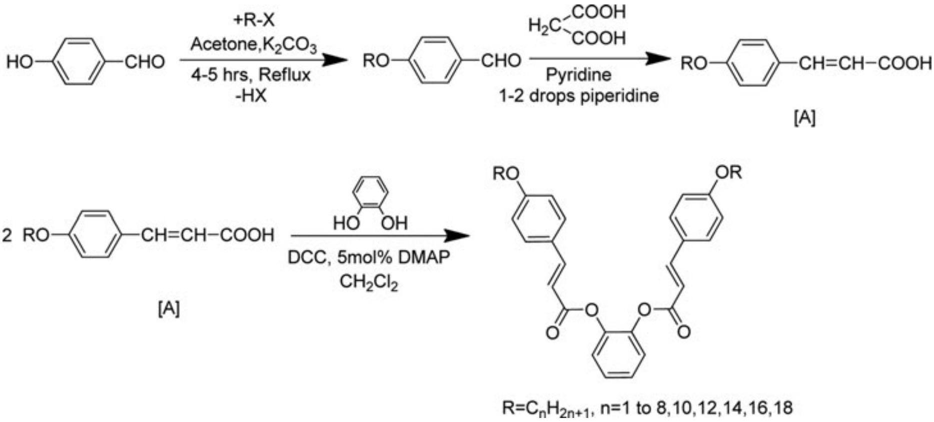


Scheme 1. Synthetic route to the series.

Experimental

Synthesis

4-Hydroxy benzaldehyde was alkylated by suitable alkylating agents R-X by refluxing for 4-5 hr with acetone and K_2CO_3 through an established method [18]. 4-*n*-Alkoxy benzaldehydes were individually condensed with malonic acid in pyridine in the presence of few drops of piperidine to obtain trans 4-*n*-alkoxy cinnamic acids. Transition temperatures of dimeric 4-*n*-alkoxy cinnamic acids matched with the reported values. Trans *n*-alkoxy cinnamic acids were individually treated with pyrocatechol (ortho dihydroxy benzene) in the presence of DCC, 5 mol% DMAP and CH_2Cl_2 in the usual manner [19]. Final products were washed with methanol under vacuum filtration, then recrystallized until constant transition temperatures were obtained. The synthetic route to the series is shown in scheme-1.

The chemicals 4-hydroxy benzaldehyde, R-X (alkyl halides), K_2CO_3, acetone, DCC, DMAP, CH_2Cl_2, pyrocatechol etc. were used as received except solvents which were dried and distilled prior to use.

Characterization

Some selected members of a series were characterized by elemental analysis (Table 1), infrared spectroscopy, ^{1}H NMR spectra, mass spectroscopy, and DSC analysis (Table 2).

Table 1. Elemental analysis for (1) Butyloxy (2) Octyloxy (3) Octadecyloxy derivatives

Sr. No.	Molecular formula	Elements% Found	Elements% Calculated
		C H O	C H O
1	$C_{32}H_{34}O_6$	74.75 6.62 18.63	74.69 6.66 18.65
2	$C_{40}H_{50}O_6$	76.71 8.02 15.27	76.65 8.04 15.31
3	$C_{60}H_{90}O_6$	79.38 10.02 10.60	79.42 10.00 10.58

Table 2. DSC data for C_{14} and C_{16} homologues at the heating rate of 5°C per minute

Homologue	Transition	Transition Temperature °C (t)	ΔH Jg^{-1}	Absolute temperature °K $T = t + 273$	$\Delta s = \Delta H/T$
C_{14}	Cr-Sm	70.57	1.77	343.57	0.00515
	Sm-I	94.21	8.78	367.21	0.02391
C_{16}	Cr-Sm	87.37	154.38	360.37	0.4284
	Sm-I	94.57	11.02	367.57	0.02998

Microanalysis was performed on a EuroEA Elemental Analyzer. IR spectra were recorded on a Shimadzu FTIR-8400 spectrometer. ^{1}H NMR spectra were recorded on Bruker Avance-III (400 MHz) spectrometer using $CDCl_3$ as a solvent. Mass spectra were recorded on Shimadzu GC-MS Model No.QP-2010. Differential scanning calorimetery (DSC) was performed by Shimadzu DSC-60 at scanning rate of 5°C min^{-1}. The liquid crystal behavior and the type of textures were determined by a miscibility method based on microscopic observations.

Analytical Data

Elemental analysis (Table 1)

Spectral Data

^{1}HNMR in ppm for Ethyloxy Derivative. 1.38–1.44 (t,6H,-CH_3 of both –OC_2H_5 group), 4.01–4.06 (m,4H,CH_3-$\underline{CH_2\text{-}O}$- of both phenyl rings containing alkoxy chain), 6.42–6.46(d,2H, -OOC-C$\underline{H}$ = CH- of both phenyl rings containing alkoxy chain), 7.59–7.63(d,2H, -OOC-CH = C$\underline{H}$- of both phenyl rings containing alkoxy chain), 7.27–7.45 (4H, middle phenyl ring), 6.82–7.84 (8H, both phenyl ring with alkoxy chain), The NMR data is consistent with the molecular structure.

^{1}HNMR in ppm for Decyloxy Derivative. 0.86–0.89 (t,3H,-CH_3 of –$OC_{10}H_{21}$ group), 1.27 (m,24H,CH_3-$\underline{\{CH_2\}_6}$- of both phenyl rings containing alkoxy chain), 1.40–1.44(p,4H,-$\underline{CH_2}$-CH_2-CH_2-O- of both phenyl rings containing alkoxy chain), 1.70–1.84 (4H,-$\underline{CH_2}$-CH_2-O- of both phenyl rings containing alkoxy chain), 4.07–4.13 (t,4H,-CH_2-O- of both phenyl rings containing alkoxy chain), 6.42–6.62(d,2H, -OOC-C$\underline{H}$ = CH- of both phenyl rings containing alkoxy chain), 7.42–7.44(d,2H, -OOC-CH = C$\underline{H}$- of both phenyl rings containing alkoxy chain), 7.25–7.29 (4H, middle phenyl ring), 6.83–7.81 (8H, both phenyl ring with alkoxy chain), The NMR data are consistent with the molecular structure.

IR in cm^{-1} for Hexyloxy Derivative. 3045(C-H str. of aromatic), 2929, 2920 &2852 (C-H str. of (-CH_2-)$_n$ group of –OC_6H_{13}), 1730 (C=O str. of carbonyl carbon of ester group), 1697–1641 (C=O str. of α,β unsaturated ketone), 1641 (C=C str. of alkene), 1573, 1535 &1510 (C=C str. of aromatic ring), 1450(C-H bending of –CH_2 alkane group), 1377 & 1342 (C-H bending of alkene), 1301, 1246, & 1224 (C-O str. of ester group), 991 &

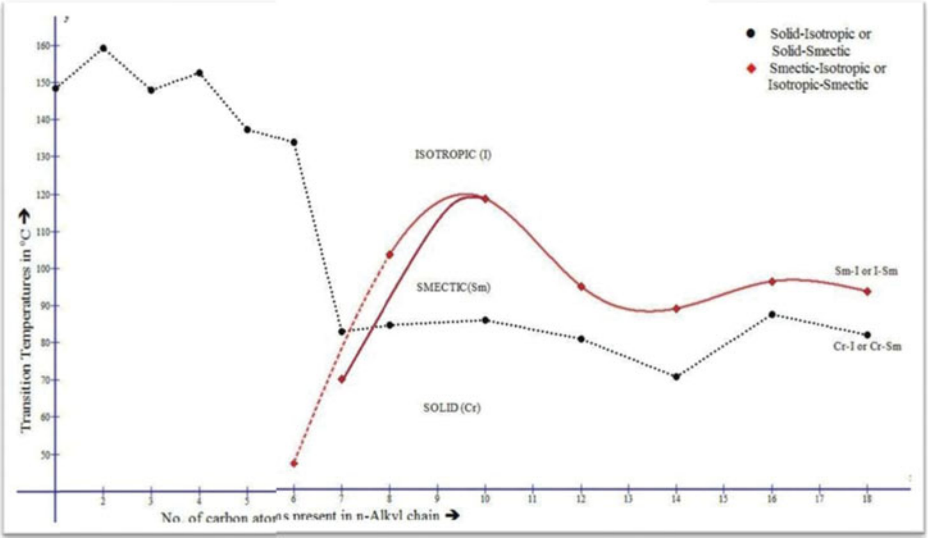


Figure 1. Phase behavior of series.

698 (C-H bending of alkene disubstituted). The IR data is consistent with the molecular structure.

IR in cm^{-1} for Dodecyloxy Derivative. 3066 (C-H str. of aromatic), 2955, 2929, & 2856 (C-H str. of (-CH_2-)$_n$ group of –$OC_{10}H_{21}$), 1739 (C=O str. of carbonyl carbon of ester group), 1676 (C=O str. of α,β unsaturated ketone), 1639 (C=C str. of alkene), 1577 & 1510 (C=C str. of aromatic ring), 1479 (C-H bending of –CH_2 alkane group), 1388 & 1332 (C-H bending of alkene), 1296, 1286, & 1282 (C-O str. of ester group), 995 & 835 (C-H bending of alkene disubstituted). The IR data are consistent with the molecular structure.

Mass Spectra of Propyloxy Derivative

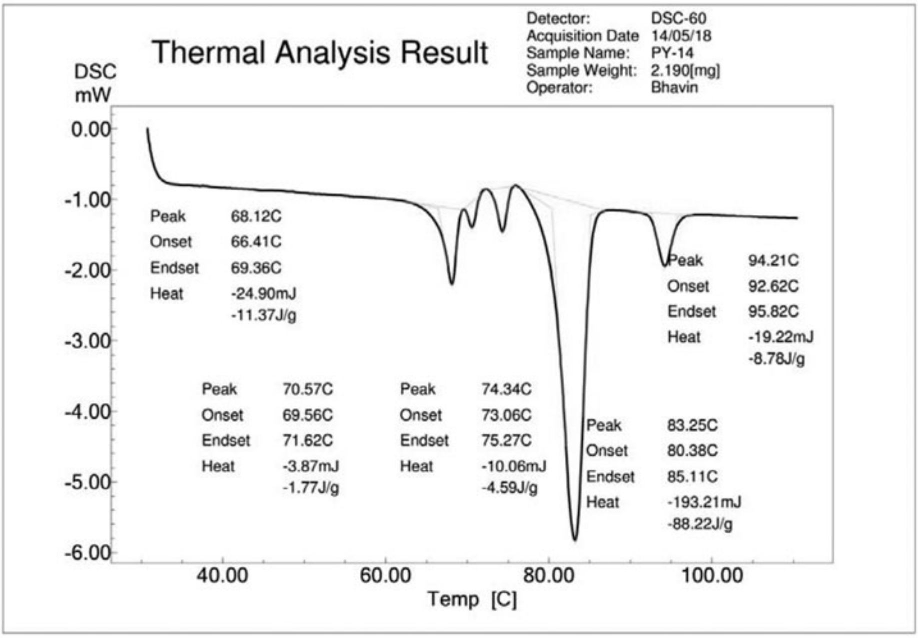
m/z (rel.int%): 486$(M)^+$, 412, 329, 287, 231, 204, 189 (base peak), 162, 147, 119, 91

Mass Spectra of Heptyloxy Derivative

m/z (rel.int%): 598$(M)^{+\cdot}$ 337, 245(base peak), 215, 189, 147, 119, 91, 57, 41

Differential Scanning Calorimetry

The transition temperatures in °C and °K as associated value of enthalpy (ΔH) changes in Jg^{-1} of C_{14} and C_{16} homologues from DSC measurements are summarized in following Table 2. C_{14} and C_{16} homologues are enantiotropically smectogenic which undergo Cr-Sm-I transition recorded during heating at the rate of 5°C per minute. DSC thermo gram of C_{16} (Fig.-2) bearing texture of smectic-C confirmed by miscibility method.



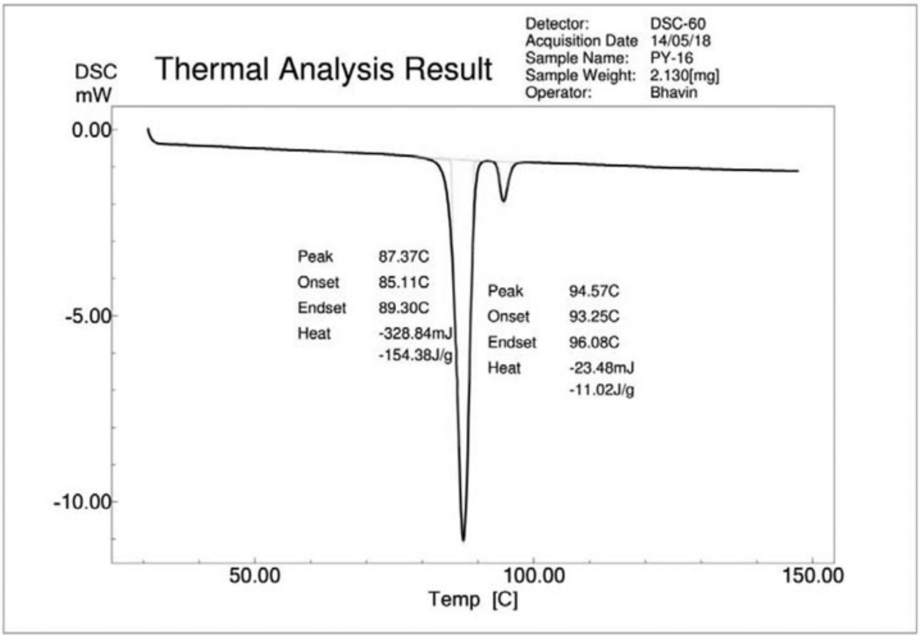


Figure 2. DSC spectra of tetradecyloxy and heptadecyloxy homologues.

Results and Discussion

Trans 4-*n*-alkoxy cinnamic acids are dimeric, but their dimerization disappears on esterification on linking with pyrocatechol and yields novel symmetric ester dimers with transition

Table 3. Transition temperatures in °C

Compound No.	*n*-alkyl chain C_nH_{2n+1} (*n*)	Sm	N	Isotropic
1	C_1	—	—	148.4
2	C_2	—	—	159.3
3	C_3	—	—	148.1
4	C_4	—	—	152.6
5	C_5	—	—	137.5
6	C_6	—	—	133.8
7	C_7	(69.8)	—	82.6
8	C_8	84.5	—	103.4
9	C_{10}	85.7	—	118.6
10	C_{12}	80.8	—	94.9
11	C_{14}	70.5	—	89.0
12	C_{16}	87.4	—	96.3
13	C_{18}	81.8	—	93.5

() indicates monotropy.

temperatures relatively lower than the corresponding trans p-*n*-alkoxy cinnamic acids. Methoxy (C_1) to hexyloxy (C_6) homologue derivatives of dimers are nonmesomorphic and the heptyloxy (C_7) to octadecyloxy (C_{18}) dimers exhibit smectogenic character without the exhibition of a nematogenic mesophase. The C_7 homologue is monotropic smectic and the rest of dimers C_8 to C_{18} are enantiotropic smectic. The transition temperatures (Table 3) as determined using an optical polarizing microscopy equipped with a heating stage were plotted against the number of carbon atoms present in the alkoxy terminal end group (Figure 1). Cr-I or Cr-Sm and Sm-I transition curves are obtained by linking like or related points showing the phase behavior of the novel dimers in a phase diagram. A Cr-I or Cr-Sm transition curve follows a zigzag path of rising and falling values with an overall descending tendency as the series is ascended. The Sm-I transition curve exhibits an odd-even effect. It initially rises for odd and even members of the series and passes through a maxima at the decyloxy (C_{10}) dimer and then descends as the series is ascended. Thus, the Cr-I or Cr-Sm and Sm-I transition curves behave in a normal established manner. The Sm-I transition curves for the odd and even members merge into each other at the decyloxy (C_{10}) dimer and then it follows as a single transition curve for the higher homologues. Mesomorphic properties of the dimers vary from homologue to homologue depending upon the variation in molecular structure. Thermal stability (average) for smectic phase is 95.95°C. Thus, the present 'V'-shaped dimer molecules of the series are smectogenic without the exhibition of any nematogenic property.

Two identical mesogenic units are bonded to a rigid spacer to form symmetric dimers of 'V'-shaped geometry. The two identical units individually containing chemical bonds have equal vector sum of bond polarities but due to the 'V' shape, the vector sum of all bonds of a molecule as a whole have some resultant value which generates the dipole moment across the long molecular axis of a molecule generating dipole–dipole interactions, dispersion forces, suitable or unsuitable magnitudes of anisotropic forces of intermolecular end to end and/or lateral attractions as consequence of the molecular rigidity and flexibility. Intermolecular anisotropic forces of cohesion are unsuitable for the C_1 to C_6 dimers which are unable to withstand exposed thermal vibrations exposed upon them. Hence, the crystal

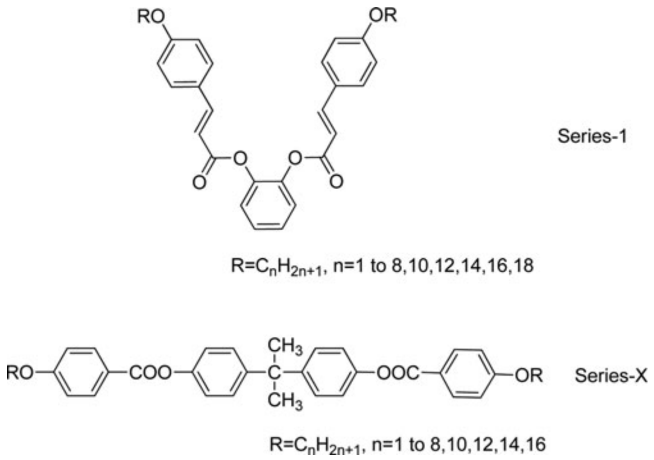


Figure 3. Structurally similar series.

lattices abruptly break and sharply melt into the isotropic state without the exhibition of a mesomorphic state. However, on increasing the length of the two terminal alkoxy chains, the combined effect of molecular rigidity and flexibility become favorable for generating lamellar packing of molecules in a sliding layered arrangement of floating layers. Hence the homologues from C_7 to C_{18} exhibit smectic mesomorphism. The absence of nematogenic character is attributed to the relatively weaker end to end attractions. The Sm-I curve for the even numbered dimers is extrapolated [20, 21, 22] to the C_6 homologue dimer to determine the latent transition temperature (LTT). However, the mesophase formation is unrealizable due to the LTT position being far below the isotropic temperature. The disappearance of odd-even effect from and beyond C_{10} member of the dimer series is attributed to the coiling or bending or flexing or coupling of longer *n*-alkyl chain with the principal axis of the core molecular structure. Variations in mesogenic properties from homologue to homologue in same dimer series is attributed to the sequentially added methylene unit in the terminal alkoxy chains, which causes variations in permanent dipole moment across the core structure of a molecule. Thus, the presently investigated novel dimer series is predominantly smectogenic and partly nonmesomorphic, whose mesomorphic phase length varies between 8.9°C to 32.9°C and is of a middle ordered melting type. Some mesomorphic properties of present symmetric dimer series-1 are compared with structurally similar known Series-X [23] as shown in Fig. 3.

The symmetric dimer series-1 of the present investigation and a dimer series-X as chosen for comparison are identical with respect to the terminal alkoxy flexible chains. Therefore molecular flexibility contribution due to this similarity may be considered as equivalent. However, the molecular part linking two RO-C_6H_4- parts differ from each other with respect to molecular rigidity and flexibility as well as their shapes and size or with respect to suitable magnitudes of anisotropic forces of intermolecular cohesive energy and closeness due to their varying permanent dipole moment across the long molecular axis, dipole–dipole interactions, electronic interaction etc. for the same homologue from series to series under comparative study. Thus, the phenomena of mesomorphism, the type and the degree of mesomorphism depended upon changing molecular parts

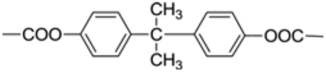
-CH = CH-COO-C_6H_4-OOC-CH = CH- and  $-COO-C_6H_4-\underset{CH_3}{\overset{CH_3}{C}}-C_6H_4-OOC-$ respectively

Table 4. Average thermal stabilities in °C

Dimer series →	1	X
Smectic–Isotropic or Smectic–Nematic	95.95°C (C_8-C_{18})	129.0°C (C_6-C_{10})
Commencement of Smectic phase	C_7	C_6
Nematic–Isotropic	–	147.7°C (C_6-C_{16})
Commencement of Smectic phase	–	C_6
Total mesophase length	8.9–32.9°C (only smectic)	18–56°C Sm:12–34°C/N: 16–32 °C

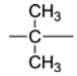
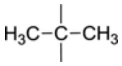
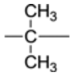
for dimer series-1 and X. The differing mesogenic molecular part of series-1 contains an aromatic phenyl ring linked to two vinyl carboxylate (-CH = CH-COO-) units. Whereas, the differing mesogenic part of series-X contain two methyl groups bonded to carbon (or n-propane) and two aromatic carboxylate (-C_6H_4-COO-) units bonded to remaining two valences of a carbon atom (Bisphenol-A). Thus, changing molecular part of series-1 contain only one phenyl ring and conjugated -CH = CH- two units which contributes molecular rigidity; whereas in case of series-X, two phenyl rings bonded to aliphatic  $-\overset{CH_3}{\underset{CH_3}{C}}-$ unit, which contributes partly to molecular rigidity and partly to molecular flexibility. Thus, suitable magnitudes of anisotropic forces of intermolecular end to end and lateral cohesive forces differs for the same homologue from series-1 to series-X and differ from homologue to homologue in the same series. Table 4 represents some mesomorphic properties like thermal stabilities of mesophase or mesophases, commencement of mesophase, mesophase length, trend of transition temperatures etc. as mentioned below.

Table 4 indicates that,

- Presently investigated series-1 is smectogenic without exhibition of nematic property, whereas homologous series-X is partly smectogenic and partly nematogenic.
- Average thermal stability for smectic of series-1 is lower (95.95°C) than series-X (129.0°C).
- Average thermal stability for nematic of a series-X is 147.7°C, whereas present series-1 does not exhibit nematogenic property i.e. nematic mesophase formation is not stabilized till the last member of a series-1, irrespective of the presence of vinyl carboxylate units, which has preferred tendency to form nematic phase.
- Smectogenic mesophase commences from C_7 member of a series-1, whereas smectic and nematic mesophase formation commences from C_6 member of a series-X.
- Smectogenic mesophase length stabilities and ranges minimum of 8.9°C to maximum of 32.9°C; whereas the same smectogenic mesophase length ranges between 12 °C and 34 °C.
- Isotropic transition temperatures of ‘V’ shaped nonlinear dimer series-1 ranges between 82.6°C to 159.3°C; whereas it ranges between 108°C and 226°C for entire series, but of course, it ranges between 108°C and 160°C for smectogenic dimers only or Sm-N transition temperatures ranges between 92°C and 150°C.

Each homologue of a homologous series-1 contain overall three phenyl rings bonded through two -CH = CH-COO- central bridges with common and same *n*-alkoxy terminal end groups for the same homologue. But the molecules of dimer series-X contain four phenyl rings bonded through –COO- central bridges via  $H_3C-\overset{|}{\underset{|}{C}}-CH_3$ with identical *n*-alkoxy terminal end groups. Therefore dimers of series-1 are less aromatic but contain conjugation in central bridges with two -CH = CH- units. Dimers of series-X are more aromatic, but contain relatively shorter central bridges -COO- via aliphatic  $-\overset{CH_3}{\underset{CH_3}{C}}-$ unit. Therefore suitable magnitudes of anisotropic forces of intermolecular cohesion depending on molecular shape and of almost equal molecular length for the same homologue from series to series induces relatively stronger end to end attraction and molecular polarizibility in addition to attractions needed for lamellar packing of molecules in crystal lattices of series-X. Thus, the molecules of series-1 exhibited and stabilized smectic mesophase only with low smectic thermal stability and late commencement of smectic phase from and beyond C_7 homologue. Whereas, dimer series-X exhibited and stabilized smectic and nematic phase with a little bit higher thermal stability and early commencement of smectic phase from C_6 homologue. The depression of transition temperatures of series-1 reflects the low resistivity toward exposed thermal vibrations as compared to corresponding homologue resistivity series-X. Hence, the variations in mesomorphic behavior of the same homologue from series to series depend upon differing molecular part of a substance.

Conclusions

- Presently investigated 'V'-shaped dimer series is smectogenic only.
- Molecular rigidity and flexibility of the dimers depend upon the rigidity offered by the spacer unit linking two identical mesogenic units of a symmetric dimer.
- Two arms of a 'V'-shaped dimers with their two arms at an angle sixty degree are generally exhibit smectic polymesomorphism or smectic and nematic mesophase formation. This statement is very well supported by present investigation and thus, it raises the credibility to the conclusions drawn earlier.
- Presently investigated novel ester series of thirteen dimers (C_1 to C_{18}) are smectogenic and nonmesomorphic from C_1 to C_6, whose transition curves behaved in normal manner in a phase diagram with exhibition of an odd-even effect for Sm-I transition curve.
- Thus, 'a phenomenon of mesomorphism is very sensitive and susceptible to molecular structure.'

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