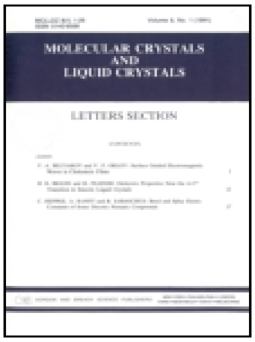

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A Comparative Study of Mesomorphism in Symmetric and Nonsymmetric Dimers

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A homologous series consisting of 12 homologue symmetrical dimers, viz., bis(4-n-alkoxy benzoyloxy) 2,2-diphenyl propanes was synthesized, studied, and evaluated to establish the relation between molecular structure and mesomorphic properties on comparative basis with nonsymmectric dimers. The methoxy to pentyloxy homologue dimers, the novel symmetric series, are nonmesogenic. Mesomorphism commences from the hexyloxy homologue as smectic and nematic. The hexyloxy to decyloxy dimers possess smectogenic character with nematogenic character and the dodecyloxy to hexadecyloxy homologue dimers exhibit nematogenic character without the exhibition of smectogenic character. All the mesogenic homologues are enantiotropic mesomorphs. The smectic and nematic thermal stability of series is 129.0°C and 147.7°C, respectively, and mesomorphic phase length varies between 18°C for the hexadecyloxy homologue and 56°C for the heptyloxy homologue.

Keywords Liquid crystal; mesomorphs; nematic; smectic; symmetrical dimers

Introduction

The molecules of mesogenic materials are geometrically constructed by linking two identical or unidentical units with a flexible spacer, generally a polymethylene unit [1, 2]. Dimers consisting of two identical units are known as symmetric dimmers [3, 4]. The magnitudes of molecular rigidity and flexibility [5–8] depend upon the number of phenyl rings, i.e., molecular aromaticity, length, polarity of flexible spacers, molecular polarizability, dispersion forces, etc., which [9, 10] contribute to the suitable magnitudes of anisotropic forces of end to end as well as lateral to terminal intermolecular closeness and attractions required to induce thermotropic mesomorphism [11], degree of mesomorphism, and the type of mesomorphism. The present investigation is planned with a view to establishing and understanding the relation between molecular structure and mesomorphic tendency of a substance [12]. The present novel investigation involves the synthesis of a novel homologous series of symmetric dimers in which two identical units, viz., $RO{-}C_6H_4{-}COO{-}C_6H_4{-}$ are bonded to the middle carbon of propane ($R = C_nH_{2n+1}$). The novel dimers are to be characterized by elemental analysis, infrared, ^{1}H NMR, and mass spectroscopy, and for mesomorphism through an optical polarizing microscope equipped with a heating stage.

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Experimental

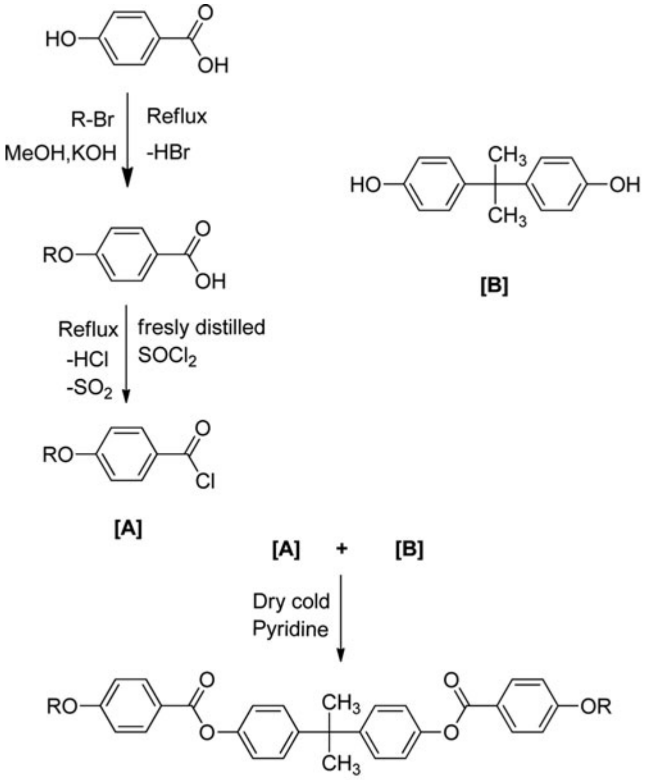
Synthesis

4-n-Alkoxy benzoic acids were prepared by reacting 4-hydroxy benzoic acid and suitable alkylating agent (R-X) in methanol in the presence of KOH by a modified method of Dave and Vora [13]. The dimeric 4-n-alkoxy benzoic acids are individually converted to 4-n-alkoxy acid chlorides by refluxing in an excess of thionyl chloride [14, 15] and then treated with bis(4-hydroxy) 2,2-diphenyl propane (m.p. 158°C) [16] in ice cooled pyridine [17]. Next day, the dimer formed was decomposed, filtered, washed, dried, and purified until constant transition temperatures were obtained.

The chemicals required for synthesis are 4-hydroxy benzoic acid, methanol, KOH, thionyl chloride, bis(4-hydroxy) 2,2-diphenyl propane, pyridine, and ethanol, and they were used as received except solvents, which were dried, purified, and distilled prior to use. The synthetic route to the series is shown in Scheme 1.

Characterization

The constitution of all the synthesized compounds has been characterized by elemental analysis (Table 1), FT-IR and ^{1}H NMR spectroscopy were further supported



Bis(4-n-Alkoxy benzoyloxy)-2,2-diphenyl propanes

Where, R= C_nH_{2n+1} and n = 1 to 8 and 10,12,14,16

Scheme 1. Synthetic route to the novel series.

Table 1. Elemental analysis for (1) propyloxy, (2) hexayloxy, and (3) decyloxy derivatives

		Elements % found			Elements % calculated		
Sr. no.	Molecular formula	C	H	O	C	H	O
1	$C_{35}H_{36}O_6$	76.11	6.60	17.29	76.06	6.57	17.37
2	$C_{41}H_{48}O_6$	77.41	7.57	15.02	77.33	7.60	15.07
3	$C_{49}H_{64}O_6$	78.63	8.56	12.81	78.57	8.61	12.82

by mass spectroscopy. Purity of all the compounds has been checked on thin layer chromatography. Elemental analyses were performed on EuroEA Elemental Analyzer (Perkin-Elmer PE 2400 CHN Analyzer, Waltham, Massachusetts, U.S.A.). IR spectra were recorded on Shimadzu FTIR-8400 (SHIMATZU FTIR-8400 Spectrophotometer, Nishinokyou Kuwabara-cho, Nakagyo-Ku Kyoto, Japan), ^{1}H NMR spectra were recorded on BRUKER Avance-III (400 MHz) spectrometer (Bruker Spectrometer, Billerica, Massachusetts, U.S.A.) using $CDCl_3$ as a solvent and mass spectra were recorded on Shimadzu GC-MS Model No. QP-2010 (SHIMATZU GC-MS Model No. QP-2010, Nishinokyou Kuwabara-cho, Nakagyo-Ku, Kyoto, Japan). The liquid crystal behavior and the type of textures were determined by a miscibility method on microscopic observations.

Analytical Data

Elemental analysis (see Table 1).

Spectral Data

^{1}H NMR in ppm for Heptyloxy Derivative. 0.88 (t, 6H, $-CH_3$), 1.29–1.50 (m, 16H, $-(CH_2)_4-$), 1.70 (m, 4H, $-CH_2CH_2O-$), 1.71 (s, 6H, $CH_3-C(Ph)_2-CH_3$), 4.03 (t, 4H, $-OCH_2$), 6.99–7.12 (dd, 8H, Ar—H), 7.29–8.01 (d, 8H, Ar—H). The NMR data confirm the molecular structure.

^{1}H NMR in ppm for Tetradecyloxy Derivative. 0.87 (t, 6H, $-CH_3$), 1.25–1.48 (m, 44H, $-(CH_2)_{11}-$), 1.75 (m, 4H, $-CH_2CH_2O-$), 1.73 (s, 6H, $CH_3-C(Ph)_2-CH_3$), 4.05 (t, 4H, $-OCH_2$), 7.10–7.40 (dd, 12H, Ar—H), 8.10 (d, 4H, Ar—H). The NMR data confirm the molecular structure.

IR in cm^{-1} for Butyloxy Derivative. 3075 (C—H Str. of aromatic ring), 2839, 2986 (C—H Str. of polyethylene group), 1728 (C=O Str. of ester group), 1508, 1606 (C=C Str. of aromatic ring), 1419 (aliphatic C—H bend), 1066, 1170, 1271 (C—O Str. of ester group), 842 (para substituted phenyl ring). The IR data are consistent with the molecular structure.

IR in cm^{-1} for Octyloxy Derivative. 2852, 2928 (C—H Str. of polyethylene group), 1732 (C=O Str. of ester group), 1512, 1604 (C=C Str. of aromatic ring), 1429 (aliphatic C—H bend), 1062, 1165, 1257 (C—O Str. of ester group), 844 (para substituted phenyl ring). The IR data are consistent with the molecular structure.

Mass Spectra of Pentyloxy Derivative. *m/z* (rel. int%): 608 $(M)^+$, 524, 418, 191, 121.

Mass Spectra of Ethyloxy Derivative. *m/z* (rel. int%): 524 $(M)^+$, 149, 137, 121.

Results and Discussion

4-n-Alkoxy benzoic acids are dimeric and on linking it with bis(4-hydroxy) 2,2-diphenyl propane through corresponding acid chloride yields novel symmetrical dimeric homologues, which exhibit mesomorphic behavior from the hexyloxy dimer. Transition temperatures (Table 2) were determined by an optical polarizing microscope equipped with a heating stage. Nematogenic mesophase formation occurs from the hexyloxy to the hexadecyloxy homologues; but the hexyloxy, heptyloxy, octyloxy and decyloxy homologues exhibit enantiotropically smectogenic mesophase formation in addition to the nematogenic mesophase. The shorter homologues of methoxy to pentyloxy homologue failed to exhibit mesomorphism even in the monotropic condition. A phase diagram (Fig. 1) showing mesogenic phase behavior is plotted for the number of carbon atoms present in the n-alkyl chain of left and right n-alkoxy terminal end groups versus transition temperatures. Transition curves, viz., solid-isotropic/mesomorphic, smectic-nematic, and nematic-isotropic, are obtained by joining like or related points. The solid-isotropic/mesomorphic transition curve follows majorly a zigzag path of rising and falling values as the series is ascended and behaves in a normal manner. The smectic-nematic transition curve initially rises and then descends after passing through maxima at the octyloxy dimer; and behaves in a normal established manner up to the decyloxy dimer and then, the curve ceases to appear for higher homologue dimers. The nematic–isotropic transition curve is descended up to the decyloxy homologue and behaves in a normal manner, but it deviates from normal behavior for the higher dodecyloxy, tetradecyloxy, and hexadecyloxy derivatives. The smectic–nematic and nematic–isotropic transition curves show an odd–even effect and an alternation of the transition temperatures. The odd–even effect diminishes for higher homologues. The transition curves showing odd–even effects are extrapolated [18–20] to the lower odd (fifth) member of the series to magnify the odd–even effect and to determine the smectic–nematic transition temperature of fifth member of the series. The smectic–nematic transition curve is also extrapolated for the dodecyloxy dimer to predict its latent transition temperature (L. T. T.)

Table 2. Transition temperatures in °C of series 1

Compound no.	R = *n*-alkyl group C_nH_{2n+1}	Transition temperature in °C		
		Sm	Nm	isotropic
1	1	–	–	226.0
2	2	–	–	176.0
3	3	–	–	140.0
4	4	–	–	115.0
5	5	–	–	130.0
6	6	106.0	128.0	160.0
7	7	114.0	146.0	170.0
8	8	116.0	150.0	166.0
9	10	80.0	92.0	108.0
10	12	–	126.0	150.0
11	14	–	101.0	120.0
12	16	–	142.0	160.0

Sm: Smectic, Nm: Nematic.

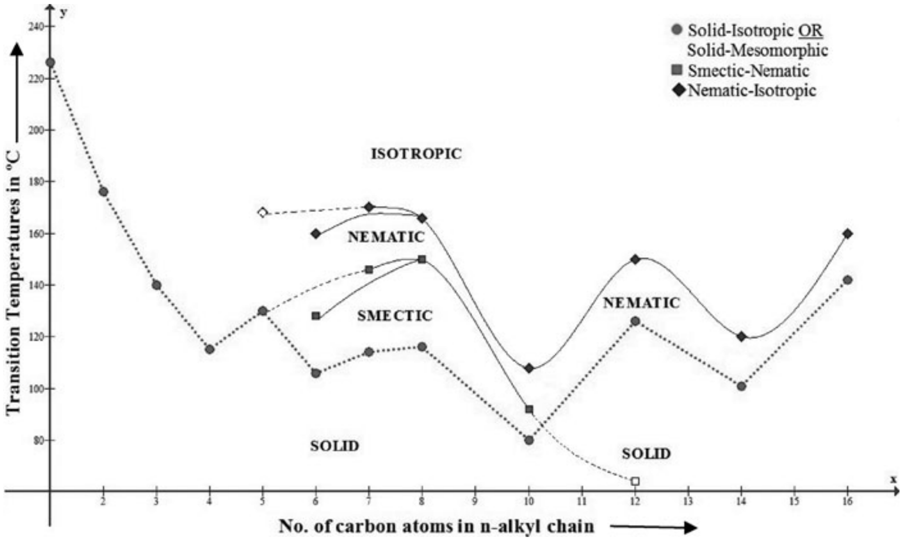


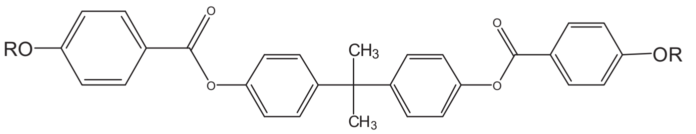
Figure 1. Phase behavior of series.

for smectic, which is far below (64.0°C) the solid-nematic transition temperature (126.0°C). The extrapolation of the transition curves is done by following the trend of the corresponding transition curve [21]. Mesogenic properties from homologue to homologue in the present series show variations due to the combined effect of molecular rigidity and flexibility. Thus, the present novel series is predominantly nematogenic and partly smectogenic with high melting type and wide degree of mesomorphism. The smectogenic mesophase length varies between 12°C and a maximum 34°C at the octyloxy homologue and the nematogenic mesophase length varies from a minimum of 18°C at the hexadecyloxy homologue to a maximum of 32°C at the hexyloxy homologue. Thus, overall the mesogenic phase length varies between 18°C at the hexadecyloxy dimer and 56°C at the heptyloxy derivative. The smectic–nematic and nematic–isotropic thermal stabilities are 129.0°C and 147.7°C, respectively.

Dimerization of 4-n-alkoxy benzoic acids disappears by breaking of hydrogen bonding between two molecules by the process of esterification through corresponding acid chlorides, yielding symmetric dimer as final product using the symmetrical phenolic component of propane. The absence of mesogenic properties in first five homologues is attributed to their high crystallizing tendencies, which arise from their inability to resist thermal vibrations due to inadequate quantum of mesophase thermal stabilization energy (enthalpy ΔH) as a consequence of unfavorable magnitudes of molecular rigidity and flexibility. The exhibition of mesomorphism by the hexyloxy to hexadecyloxy dimers is attributed to the suitable magnitudes of anisotropic forces of intermolecular closeness and end to end and/or lateral attractions as a consequence of favorable magnitudes of molecular rigidity and flexibility to maintain the disalignment of molecules at an angle less than 90° within definite range of temperature on a floating surface. Thus, molecules of hexyloxy to decyloxy dimers occupy a sliding layered ordered arrangement of molecules in floating condition within a definite temperature range due to the presence of lamellar packing of molecules in their original crystal lattices to form a smectic phase. Then the same four dimers show a statistically parallel orientational order of molecules in a floating condition causing the appearance of the nematic mesophase. However, the dodecyloxy, tetradecyloxy, and hexadecyloxy homologues due to the absence of lamellar packing of molecules in their crystal lattices fail to exhibit smectogenic character in a floating condition, but maintain the statistically parallel orientational order of molecules showing the exhibition of only nematogenic character.

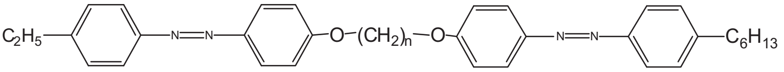
The changing mesomorphic behavior from homologue to homologue in the presently investigated novel series is attributed to the sequentially and progressively added methylene unit in the terminal chains. The odd–even effect for the smectic–nematic and nematic–isotropic transition curves appears for a very small range of the corresponding curve and from the sixth and seventh members of the series merges into the main trend of the corresponding curves at the octyloxy derivative. The odd–even effect disappears from and beyond the octyloxy homologue, because the longer chain may coil, bend, flex, or couple with the major axis of the core. The extrapolation of the smectic–nematic and nematic–isotropic transition curves for the pentyloxy homologue predicts a nematic–isotropic and solid-nematic transition temperature of 170°C and 130°C, respectively. The mesogenic properties of present series 1 are compared with structurally similar homologue series X [22] as shown in Fig. 2.

The presently investigated novel dimer series 1 and structurally similar homologous series X of dimers chosen for comparison are identical with respect to four phenyl rings, which are contributing toward molecular linearity and rigidity. However, they differ with respect to their symmetry character and the groups, linking the phenyl rings. The novel series 1 is symmetric, while series X is nonsymmetric. The linking groups of each section of the dimers are different with two —COO— groups present in series 1 and two —N=N— groups in series X. The central linking groups of $—C(CH_3)_2$- and $—O—(CH_2)_n—O—$ for series 1 and series X, respectively, are also different. Thus, the molecular changes obviously cause variations in mesogenic properties and the degree of mesomorphism for series 1 and X. The above variations between series 1 and X generate variations in combine effects of molecular rigidity and flexibility and the suitable magnitudes of anisotropic forces of intermolecular closeness and end to end as well as lateral attractions due to varying molecular polarity and polarizability, types of symmetry, dispersion forces, polarity of –COO— and —N=N— group, location and number of methylene units in n-alkyl chain at the central place or at the end of a molecule including effects due to unlike $—C_6H_{13}$ and $—C_2H_5$ end groups, etc.



$R= C_nH_{2n+1}$ Where, n=1to 8, 10, 12, 14, 16

Series-1



Series-X

Where, n = 4,5,6,7,8,9,10,11,12

Figure 2. Structurally similar isomeric/nonisomeric series.

- Symmetric homologous series 1 is smectogenic in addition to nematogenic (C_6—C_{10}) and only nematogenic for C_{12}, C_{14}, and C_{16} homologues. At the same time, series X is smectogenic in addition to nematogenic (C_4—C_9) and only nematogenic for C_{10}, C_{11}, and C_{12}.
- Odd–even effect is exhibited by transition curves of the phase diagram for smectic–nematic and nematic–isotropic phase transformation by series 1 and X. However, odd–even effect is relatively more pronounced for series X, than series 1.
- Addition of methylene unit at the n-alkyl chain of the middle central group and terminal end group does not lengthen molecular length in equal magnitude and affects unequally in thermal stability and transition temperatures behavior.
- Smectic and nematic thermal stabilities of series 1 are relatively lower than a series X, facilitating mesophase stabilization and group efficiency order.
- Smectic in addition to nematic mesophase commences from sixth (C_6) member of a series 1; whereas it commences earlier by two homologues, i.e., from fourth (C_4) member of a series -X. Following Table 3 represents the average thermal stability values for smectic and nematic mesophases.
- Total mesomorphic phase length for series 1 varies between 18°C and 56°C and that of a series X, wonderfully varies between 18°C and 58°C.

Both the series (1 and X) under comparison are long and linear with differing features and possess different magnitudes of anisotropic forces of intermolecular end to end and lateral attractions as a consequence of their individually differing molecular rigidity and flexibility. Both series show mesomorphic behavior as smectic and nematic type. The thermal stability of presently investigated dimer series 1 is lower than that of series X for the smectic and the nematic mesophases. The oxygen atom of the central carboxy group in the molecule of series 1 will bump into the non-bonded sides of the adjacent hydrogens of the aromatic ring, which will cause considerable strain on the molecule. Consequently, a twist around C—O bond will occur forcing the phenyl ring out of the plane of a molecule. Thus, the coplanarity of the molecules is reduced to some extent making them thick. The central bridge —N=N— certainly endows the molecules with coplanarity in trans position. On account of these differences, the mesophase thermal stabilities (smectic and nematic) of series 1 are in general way relatively lower than the corresponding mesophase (smectic and nematic) thermal stabilities of a homologous series X. Moreover, early or late commencement of smectic and nematic mesophase depends upon the extent of noncoplanarity caused by a molecule. Thus, difference in molecular coplanarity causes commencement of smectic and nematic mesophase earlier by two homologues (C_4) in case of series X and a little bit late, from sixth (C_6) homologue in series 1. The addition of methylene unit simultaneously to the

Table 3. Average thermal stability in °C

Series→	[1]	[X]
Smectic—nematic	129.0	139.5
Commencement of	[C_6—C_{10}]	[C_4—C_9]
smectic phase	C_6	C_4
Nematic—isotropic	147.7	152.7
Commencement of	[C_6—C_{16}]	[C_4—C_{12}]
nematic phase	C_6	C_4

terminal group of series 1 and to the middle central group $-(CH_2)_n-$ of series X does not increase the molecular length in equal magnitudes, because n-alkyl chain in terminal group of series 1 is bonded toward one side of a phenyl ring through oxygen atom, while n-alkyl chain in middle central bridge is bonded toward both end side of the two phenyl rings through two oxygen atoms. Therefore, n-alkyl chain bonded as terminal end group enjoys relatively more flexibility in series 1 as compared to less flexibility enjoyed by n-alkyl chain at the middle central position in case of series X. Thus, n-alkyl chain as terminal end group can coil, bend, flex, or couple to lie with major axis of the core structure; but as middle central group cannot. Thus, unequal increase in molecular length by adding methylene unit causes variations in the molecular rigidity and flexibility. Hence, such variations are reflected in the mesogenic properties between two same homologues of different series; or series to series. Odd–even effect is more pronounced in series X as compared to series 1, because mesomorphism commences earlier from fourth (C_4) homologue in series X while it commences late from sixth (C_6) homologue in series 1. Individual smectic and nematic mesophase length differs for series 1 and X, but total mesophase length (smectic + nematic) varies between 18°C and 56°C as well as 18°C and 58°C for series 1 and series X, respectively.

Conclusions

- A symmetric novel homologous series of dimers of propane derivatives with four phenyl rings and symmetrically bonded two n-alkoxy terminal end groups as well as two carboxy central groups synthesized and its mesomorphic properties are compared with an unsymmetrical azoester homologous series bearing $-C_2H_5$ and $-C_6H_{13}$ terminal end groups and $-O-(CH_2)_n-O-$ middle central group linking second and third two phenyl rings out of four.
- The group efficiency order derived on the basis of (i) thermal stabilities and (ii) early commencement of mesophase for smectic and nematic are as under

(i) Smectic: unsymmetrical series X > symmetrical series 1
Nematic: unsymmetrical series X > symmetrical series 1
(ii) Smectic: unsymmetrical series X > symmetrical series 1
Nematic: unsymmetrical series X > symmetrical series 1

- Therefore, group efficiency order of,
- Middle central group of n-alkyl chain > terminal end group of n-alkyl chain
  OR

$$-O-(CH)_n-O- > C_nH_{2n+1}-O-$$

- The individual smectic and nematic mesophase lengths differ between symmetric and unsymmetric homologous dimer series but total mesophase length range remains very closer. That is, addition of methylene unit to n-alkyl chain of either terminal end group or to the middle central group of a dimer homologous series induces total mesophase length range (Sm+Nm) almost equivalent irrespective of symmetry (symmetric or nonsymmetric) of a dimer series, but individual mesophase length range for smectic and/or nematic may alter.
- Molecular rigidity and flexibility are sensitive and susceptible to molecular structure of a substance.

- Suitable magnitudes of anisotropic forces of intermolecular attraction as a consequence of favorable molecular rigidity and flexibility can induce mesomorphism.
- The novel series of dimers is predominantly nematogenic and partly smectogenic.

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